

# For how long does the quality and quantity of residues in the soil affect the carbon compartments and CO<sub>2</sub>-C emissions?

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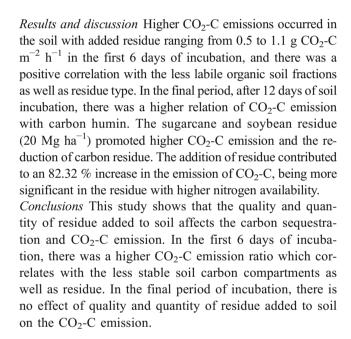
#### **Abstract**

*Purpose* The mineralization/immobilization of nutrients from the crop residues is correlated with the quality of the plant material and carbon compartments in the recalcitrant and labile soil fractions. The objective of this study was to correlate the quality and quantity of crop residues incubated in the soil with carbon compartments and CO<sub>2</sub>-C emission, using multivariate analysis.

Materials and methods The experiment was conducted in factorial 4+2+5 with three replicates, referring to three types of residues (control, sugarcane, Brachiaria, and soybean), and two contributions of the crop residues in constant rate, CR (10 Mg ha<sup>-1</sup> residue), and agronomic rate, AR (20, 8, and 5 Mg ha<sup>-1</sup> residue, respectively, for sugarcane, soybean, and Brachiaria), evaluated five times (1, 3, 6, 12, and 48 days after incubation). At each time, we determined the CO<sub>2</sub>-C emission, nitrogen and organic carbon in the soil, and the residues. In addition, the microbial biomass and water-soluble, labile, and humic substance carbons fractionated into fulvic acids, humic acids and humin were quantified.

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**Keywords** Multivariate analysis · Residue · Soil carbon pools · Soil respiration

### 1 Introduction

The no-till system has been a conservative management system used since 1970 in Brazil (Embrapa 2010), one of the leaders in the use of this system, which occupies more than half of its area planted with annual crops (Mapa 2014). Studies have shown significant ecological, economic, environmental, and social benefits of this system in maintaining crop residue (stubble, pointers, and green leaves) on the ground (Heinz et al. 2011).



The residues added to the soil surface help to increase the organic matter (SOM), an important source of soil organic carbon (TOC-S), and that, when decomposed/mineralized, releases large amounts of nutrients (Balota et al. 2014) and  $\rm CO_2$  via microbial activity (Gassen and Gassen 1996). With the addition of residue, C sequestration in the soil also occurs (Bordonal et al. 2013), mitigating the concentration of greenhouse gases (GHG) (Alvaro-Fuentes and Paustian 2011), mainly carbon dioxide ( $\rm CO_2$ ) (Glaser et al. 2001; Lehmann et al. 2011).

The main crops used in no-till systems are grasses or legumes, among them sugarcane (Saccharum officin arum), soybean (Glycine max), and Brachiaria (Brachiaria decumbens). These materials differ in chemical composition, specifically regarding free sugars, fructans, starch, pectin, hemicellulose, cellulose (Gunnarsson et al. 2008), and lignin (Trinsoutrot et al 2000; Wang et al. 2004), besides the N content and their C/N ratio (Trinsoutrot et al. 2000; Moritsuka et al. 2004; Six et al. 2006; Dorodnikov et al. 2011), leading to different decomposition times. The material quality is one of the most important parameters in nutrient dynamics in agricultural soils (Lal 2004), besides the amounts added. The real benefits of the no-till system to increase and sequester C have recently been questioned (Baker et al. 2007) since it will depend on the quality or quantity of the residue added to soil.

Thus, the aim of this study was to correlate, using multivariate analysis, the influence of time and the quality and quantity of different types of residues incubated on carbon sequestration and CO<sub>2</sub>-C emission from the soil.

### 2 Materials and methods

## 2.1 Soil sampling and analysis

The experiment was conducted at the Pedology Laboratory, Federal University of Uberlândia, Brazil. A factorial 4+2+5 was established with three replicates, referring to three types of residues (control, sugarcane, *Brachiaria*, and soybean), and two contributions of the crop residues at constant rate, CR (10 Mg ha<sup>-1</sup> residue), and agronomic rate, AR (20, 8, and 5 Mg ha<sup>-1</sup> residue, respectively, for sugarcane, soybean, and *Brachiaria*), in five different times (1, 3, 6, 12, and 48 days after incubation). In addition, we analyzed the control treatment (soil without residue input).

The residue contributions added to soil have the objective of assessing the quality of the plant material at a CR of 10 Mg ha<sup>-1</sup>. The objective of the AR is to evaluate the effect of the quantity with the average contribution of sugarcane, soybean, and *Brachiaria* residue to the soil. For this, Eq. 1 was used. In this equation, the sum of the agronomic rate (AR) of

sugarcane (20 Mg ha<sup>-1</sup>), soybean (8 Mg ha<sup>-1</sup>), and *Brachiaria* (5 Mg ha<sup>-1</sup>) is subtracted from the number of residues (3) and then divided by the number of residues (3).

$$CR = AR_{sugarcane + soybean + brachiaria} - N/N$$
 (1)

The soil was collected in June 2013 in the 0.0–20.0 cm layer from an area cultivated with sugarcane under a Dystrophic Red-Yellow Latosol, loamy texture located between the cities of Uberlândia and Uberaba in the Triângulo Mineiro (latitude 19°13′00.22″S and longitude 48°08′24.80″ W). In the laboratory, the soil was air-dried, sieved (<2 mm) to obtain fine air-dried soil (FADS), and moistened to 60 % of its water holding capacity (WHC) for the experiment setup.

For characterization, a soil fraction was utilized for the determination of chemical and physical properties. The soil texture was measured using the pipette method. Potassium (K<sup>+</sup>), calcium (Ca<sup>2+</sup>), magnesium (Mg<sup>2+</sup>), pH (soluble in H<sub>2</sub>O), and available phosphorus (P) were measured using the methodologies described by Embrapa (1997). The total nitrogen content of the soil (N-S) and residue (N-St) were determined according to the Kjeldahl method using the methodologies of Embrapa (1997) and total organic carbon (TOC-St) and residue (TOC-St) by the method suggested by Yeomans and Bremner (1988) (Table 1).

The value of lignin (%) was obtained in Costa et al. (2013), Santos et al (2004), and Reddy and Yang (2009), respectively, for sugarcane, *Brachiaria*, and soybean residues. The cellulose value (%) was obtained in Almeida et al. (2009), Gonçalves et al (2007), and Reddy and Yang (2009) and crude protein value (%) in Pereira et al (2000), Santos et al (2004), and Maheri-Sis et al (2011), respectively, for sugarcane, *Brachiaria*, and soybean residues.

During the time of the residue sampling (sugarcane, *Brachiaria decumbens*, and soybean), no distinction was made between stem and leaves in order to obtain a homogeneous sample, and the moisture retention of the material was not considered. In the laboratory, the samples were cut to an average size of 2 cm<sup>2</sup> and placed in an oven at 60 °C for 48 h for subsequent analyses and incubation.

## 2.2 Soil incubation

For incubation, a column of 700 g of soil, reaching a height of 13 cm, was placed in a 15-cm-height, 10.5-cm-diameter PVC (polyvinylchloride) pot and total volume of 1298.8 cm<sup>3</sup> fixed on a Styrofoam base properly insulated to prevent soil water loss.

The setup was left in an open laboratory environment at a controlled temperature of 25 °C. This temperature is appropriate for microbial activity (Stanford et al. 1973) since the



**Table 1** Chemical and physical characteristics of sugarcane, *Brachiaria*, and soybean residues and Red-Yellow Latosol used in the experiment

Characteristics <sup>a</sup>	Soil	Residue		
		SC	BR	SO
Sand (g kg <sup>-1</sup> )	630	_	_	_
Silt $(g kg^{-1})$	140	_	_	_
Clay $(g kg^{-1})$	230	_	_	_
pH (H <sub>2</sub> O)	5.60	_	_	_
$P (mg dm^{-3})$	2.47	_	_	_
$K^+ (mg dm^{-3})$	208.08	_	_	_
$\mathrm{Mg}^{2+}$ (cmol <sub>c</sub> dm <sup>-3</sup> )	0.56	_	_	_
$Ca^{2+}$ (cmol <sub>c</sub> dm <sup>-3</sup> )	2.20	_	_	_
TN (N-St or N-S) $(g kg^{-1})$	0.69	1.44	1.68	2.49
TOC (N-St or N-S) $(g kg^{-1})$	7.40	120.00	122.40	142.12
C/N	-	83.63	72.85	50.22
Lignin (%)		$25.80^{(4)}$	$12.76^{(6)}$	$14.0^{(2)}$
Cellulose (%)		$72.90^{(1)}$	29.71 <sup>(7)</sup>	44.00 <sup>(2)</sup>
Crude protein (%)		$2.50^{(5)}$	$2.80^{(6)}$	$5.10^{(3)}$

<sup>&</sup>lt;sup>a</sup> In table: *SC* sugarcane, *SO* soybean, *BR Brachiaria decumbens*; potassium (K<sup>+</sup>); calcium (Ca<sup>2+</sup>); magnesium (Mg<sup>2+</sup>); pH (H<sub>2</sub>O); phosphorus (P); total nitrogen (TN) content of the soil (N-S) and residue (N-St) and total organic carbon (TOC) content of the soil (TOC-S) and residue (TOC-St); C/N: relationship between soil organic carbon (TOC and TOC-St) and nitrogen total nitrogen content of the soil (N-S and N-St). (<sup>1)</sup> Almeida et al. 2009; (<sup>2)</sup> Reddy and Yang 2009; (<sup>3)</sup> Maheri-Sis et al. 2011; (<sup>4)</sup> Costa et al. 2013; (<sup>5)</sup> Pereira et al. 2000; (<sup>6)</sup> Santos et al. 2004; (<sup>7)</sup> Gonçalves et al. 2007

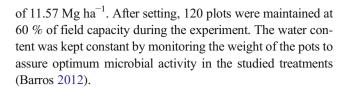
temperature and humidity of the soil contribute to the increase in soil biological activity (Almeida et al. 2009).

For incubation of the sugarcane residue in the soil, 17.31 g pot<sup>-1</sup> of residue corresponding to 20 Mg ha<sup>-1</sup> was added, equivalent to an average deposition of between 10 and 30 Mg ha<sup>-1</sup> year<sup>-1</sup> of dry matter on the soil surface in a mechanized planting system (Urquiaga et al. 1991) composed of pointers and green leaves cut to a size of 10–12 cm (Oliveira et al. 1999).

In the treatment with soybean residue, 6.92 g pot<sup>-1</sup> was added, related to the input of 8 Mg ha<sup>-1</sup>. Soybean cultivation provides low dry matter contribution to the soil surface (Embrapa—Empresa Brasileira de Pesquisa Agropecuária 2014), ranging from 4.81 to 2.81 Mg ha<sup>-1</sup>, consisting of stems, petiole, trifoliate leaves, and pods (Kurihara et al. 2013).

The input of 5 Mg ha<sup>-1</sup> of *Brachiaria* pasture residue (*Brachiaria decumbens*) was obtained with the addition of 4.32 g pot<sup>-1</sup> of dry residue. The average Brazilian covering dry matter production of *Brachiaria decumbens* is 6.0 Mg ha<sup>-1</sup> (Torres et al. 2005; Rossi et al. 2011) and can range from 4.8 to 11.17 Mg ha<sup>-1</sup> (Nunes et al. 2006).

To evaluate the quality of plant material, we added the same dose of 10 g pot<sup>-1</sup> for all types of residue related to input



# 2.3 Emission of CO<sub>2</sub>-C and soil C compartments

Evaluation of CO<sub>2</sub>-C emissions from the soil were conducted at 1, 2, 3, 4, 6, 8, 10, 13, 16, 19, 22, 25, 28, 31, 34, 37, 44, and 48 days after incubation (DAI), using a portable infrared gas analyzer system. This instrument consists of a closed chamber with an internal volume of 854.2 cm<sup>3</sup> and ground contact area of 83.7 cm<sup>2</sup> (LI 8100; Li-Cor Inc., Lincoln, NE, USA). After closing, the system quantifies the variations in the concentration of CO<sub>2</sub>-C within the chamber over time by optical absorption spectroscopy in the infrared spectral region.

On the 1st, 3rd, 6th, 12th, and 48th day after incubation, respectively, the 24 setups with all the soil sampling treatments were dismantled. They were subsequently sieved in a 2-mm mesh, homogenized, and the remaining residue separated. With the soil still damp, the measurement of soil microbial biomass carbon (C-Mic) was conducted by the method described by Vance et al. (1987) using a microwave oven for irradiation (Islam and Weil 1998).

After drying the samples, soil organic carbon (TOC-S) and residue (TOC-St) were determined using oxidation with potassium dichromate method in acid medium (Yeomans and Bremner 1988). It should be noted that for characterizing the soil and residue TOC before incubation, we used the same method.

The water-soluble carbon (C-WSC) was extracted with deionized water. For carbon oxidation, we followed the procedures recommended by Yeomans and Bremner (1988). The quantitative extraction and fractionation of humic substances (HS) in humin (C-HU), fulvic acid (C-FA), and humic acid (C-HA) were determined by differential solubility, established by the International Humic Substances Society (Swift 1996), and the labile C (C-labile) was performed with the methodology proposed by Mendonça and Matos (2005), determined by sample oxidation with 0.033 mol  $\Gamma^{-1}$  potassium permanganate and the reading of the extracts in a spectrophotometer at 565 nm.

Nitrogen from soil (N-S) and residue (N-St) was determined using the Kjeldahl method described by Black (1965). For the soybean residue treatment, it was not possible to perform the N-St analysis in the final period (48th DAI) because there was not enough plant material due to its high decomposition rate.

#### 2.4 Data processing and statistical analysis

The variability of the studied properties was previously evaluated by means of descriptive statistics by calculating the



mean, standard deviation, and minimum and maximum values.

Subsequently, the variables were subjected to multivariate exploratory analysis by hierarchical clustering and principal component methods. For the cluster analysis, a similarity matrix was constructed with the Euclidean distance and the connections of the clusters were conducted with the Ward method (Sneath and Sokal 1973). In this method, the distance between two groups is defined as the sum of squares between the two groups through all variables (Hair 2007). The Euclidean distance among accesses to the set of variables was calculated, distinguishing between the studied factors (residue types, doses, and time) and exposing the group structure contained within the data in a dendrogram.

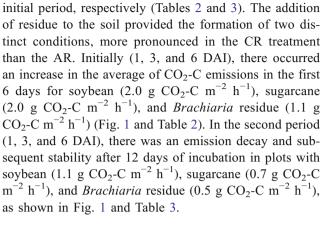
For principal component analysis (PCA), we used the variables CO<sub>2</sub>-C emission, nitrogen soil (N-S) and residue (N-St), and carbon soil (TOC-S) and residue (TOC-St), in addition to humic substance carbon (C-HS) fractionated in fulvic acids (C-FA), humic acid (C-HA), and humin (C-HU). Subsequently, we scaled up the number of variables according to their characteristics to better visualize the interaction between the variables in the coordinate axes. These new axes, the eigenvectors (new variables) called principal components (PC), are generated by linear combinations of the original variables constructed from the eigenvalues of the covariance matrix (Hair 2007; Piovesan et al. 2008). In order to obtain a simpler and more parsimonious model, we used the Kaiser criterion (1958) with eigenvectors above the unit. Analyses were conducted in the STATISTICA 7.0 software (StatSoft Inc., Tulsa, OK, USA).

# 3 Results

#### 3.1 Effect of time on the soil CO<sub>2</sub>-C emission

In the soil incubation, the lowest CO<sub>2</sub>-C emission occurred in the treatment without residue addition (Fig. 1)

Fig. 1 CO<sub>2</sub>-C emission of soil with residue of soybean, *Brachiaria*, sugarcane, and treatment control with two contributions of the residue crops in agronomic rate (AR) and constant rate (CR) during 48 days of incubation



with an average from 0.1 to 0.2 g m<sup>-2</sup> h<sup>-1</sup> at the final and

## 3.2 Effects of residue quality and quantity

Evaluating the quantity (AR) and quality (CR) of residue added to the soil, it was found that the AR soybean residue dose presented the highest emission of accumulated CO<sub>2</sub>-C (48.57 g kg<sup>-1</sup>), followed by sugarcane (49.06 g kg<sup>-1</sup>) and *Brachiaria* (23.30 g kg<sup>-1</sup>), and with respective increases of 86.98, 87.11, and 72.87 % compared to the control treatment (6.32 g kg<sup>-1</sup>; Fig. 2).

The CR showed the highest emission of accumulated CO $_2$ -C with soybean residue (80.53 g kg $^{-1}$  C-CO $_2$ ), followed by *Brachiaria* (47.30 g kg $^{-1}$  C-CO $_2$ ) and sugarcane (33.21 g kg $^{-1}$  C-CO $_2$ ) and with respective increases of 92.15, 86.63, and 80.96 % compared to the control treatment (6.32 g kg $^{-1}$ ; Fig. 2).

The greater decreases in TOC-St with AR doses occurred with soybean incubation, followed by *Brachiaria* and sugarcane residue, respectively 90.74, 67.30 g, and 73.84 kg<sup>-1</sup> of carbon. At the CR, greater decreases were obtained from soybean, sugarcane, and *Brachiaria* residue, respectively 64.80, 60.00, and 45.60 g kg<sup>-1</sup> TOC-St (Fig. 3). The higher the residue dose added to the soil, under C/N ratio conditions ranging from 50.22 to 83.63 g kg<sup>-1</sup> (Table 1), the greater the increase in the CO<sub>2</sub>-C emission.

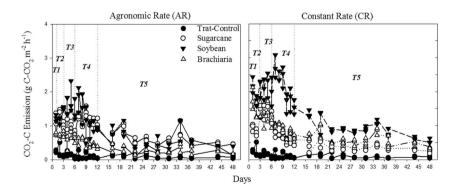




Table 2 Descriptive statistics in the initial period of variables<sup>a</sup>: humin C (C-HU), fulvic acid (C-FA), humic acid (C-HA), water-soluble carbon (C-WSC), carbon labile (C-Labile), microbial biomass carbon (C-Mic), total carbon organic soil (TOC-S), carbon organic soil residue (TOC-St), nitrogen organic soil (N-S), nitrogen organic residue (N-St), and emission of CO<sub>2</sub>-C of soil

	Mean	SD	Max	Min	Mean	SD	Max	Min
	No residu	e		Sugarcane				
C-HU	12.0	0.7	13.1	10.9	12.4	1.2	14.9	10.0
C-HA	2.1	0.2	2.6	1.9	2.2	0.4	3.1	1.6
C-FA	3.5	0.3	3.8	3.0	3.5	1.1	7.5	1.8
C-WSA	0.2	0.1	0.4	0.1	0.2	0.0	0.2	0.1
C-Labile	5.2	0.0	5.2	5.2	5.2	0.0	5.2	5.2
C-Mic	1631.8	497.1	2336.9	1018.5	2515.5	1128.4	4954.4	1094.7
TOC-S	22.4	1.5	24.8	20.1	22.7	2.1	26.2	18.4
N-S	1.3	0.2	1.8	1.1	1.6	0.5	3.4	1.3
TOC-St	_	_	_	_	93.3	30.9	133.8	45.0
N-St	_	_	_	_	8.0	1.5	11.5	3.9
CO <sub>2</sub> -C	0.2	0.5	1.9	0.8	2.0	1.3	9.8	4.6
	Brachiaria			Soybean				
C-HU	12.4	1.1	14.0	10.6	12.4	1.1	14.0	10.0
C-HA	2.3	0.3	2.8	1.9	2.3	0.5	3.3	1.2
C-FA	3.4	0.5	4.2	2.4	3.7	0.4	4.8	3.1
C-WSA	0.2	0.1	0.3	0.1	0.2	0.1	0.4	0.0
C-Labile	5.2	0.0	5.2	5.2	5.2	0.0	5.2	5.2
C-Mic	2117.7	1049.7	3900.8	681.9	2239.6	865.4	3481.7	1150.1
TOC-S	23.1	2.8	26.7	18.0	23.4	2.2	26.5	18.9
N-S	1.5	0.2	1.8	1.3	1.7	0.3	2.2	1.3
TOC-St	115.5	30.0	152.4	54	87.0	37.9	135	24.6
N-St	7.9	1.8	11.5	3.1	10.6	4.4	19.6	4.9
CO <sub>2</sub> -C	1.1	2.9	13.4	3.6	2.0	3.0	15.4	5.5

 $<sup>^</sup>a$  Values of C-HU, C-HA, C-FA, C-WSA, C-Labile, TOC-S, N-S, TOC-St, and N-St in g  $kg^{-1}$  . The values of C-Mic and emission, respectively, are in  $\mu g\ g^{-1}\ ha^{-1}$  and g CO2-C  $m^{-2}\ h^{-1}$ 

In soil with addition of 20 Mg ha<sup>-1</sup> (AR) of sugarcane residue, the C loss via CO<sub>2</sub>-C emission was 72.9 % from the residue decomposition. As for the doses of 8 and 5 Mg ha<sup>-1</sup>, respectively, of soybean and *Brachiaria*, CO<sub>2</sub>-C losses through the decomposition of residue are lower, respectively 53.2 and 31.5 %.

At the CR of added sugarcane residue, C-loss through the  $CO_2$ -C emission is 55.3 % from the residue decomposition. For the soybean and *Brachiaria* residue, there was a 100 % C-loss from the residue decomposition and 3.7 and 24.2 % of C from the soil, respectively (Fig. 2).

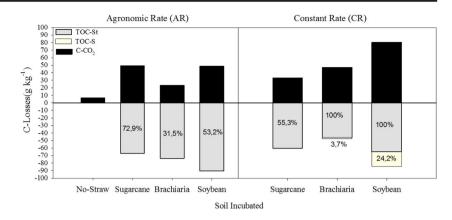
Table 3 Descriptive statistics in the final period of variables<sup>a</sup>: humin C (C-HU), fulvic acid (C-FA), humic acid (C-HA), watersoluble carbon (C-WSC), carbon Labile (C-Labile), microbial biomass carbon (C-Mic), total carbon organic soil (TOC-S), carbon organic residue (TOC-St), nitrogen organic residue (N-St), and emission of CO<sub>2</sub>-C of soil

	Mean	SD	Max	Min	Mean	SD	Max	Min
	No residue	e	'	,	Sugarcane	<del></del>	,	
C-HU	17.5	0.9	18.8	16.0	17.0	2.6	19.2	10.0
C-HA	1.1	0.4	1.8	0.6	1.0	0.3	1.3	0.6
C-FA	2.1	0.3	2.4	1.7	1.8	0.5	2.4	0.5
C-WSA	0.1	0.0	0.1	0.0	0.1	0.0	0.2	0.0
C-Labile	5.2	0.0	5.2	5.2	5.2	0.0	5.2	5.2
C-Mic	1847.1	1023.0	2988.9		2059.2	1212.4	4309.7	824.3
TOC-S	21.6	2.8	26.6	19.5	22.7	1.5	25.4	19.7
N-S	1.4	0.1	1.5	1.3	1.6	0.2	2.0	1.4
TOC-St	_	_	_	_	42.0	22.5	86.9	12.8
N-St	_	_	_	_	9.8	1.9	12.9	7.7
CO <sub>2</sub> -C	0.1	0.3	1.1	0.4	0.7	2.0	8.0	2.0
-	Brachiaria	a			Soybean			
C-HU	18.5	1.6	23.0	17.2	18.7	1.0	20.0	17.6
C-HA	1.0	0.3	1.3	0.3	1.1	0.3	1.5	0.3
C-FA	2.2	0.4	2.6	1.4	2.2	0.7	3.1	1.2
C-WSA	0.3	0.9	3.2	0.0	0.9	1.4	3.2	0.1
C-Labile	5.2	0.0	5.2	5.2	5.2	0.0	5.2	5.2
C-Mic	1276.1	359.3	1752.5	721.1	1560.5	929.6	3260.0	750.0
TOC-S	21.1	2.0	24.8	17.7	22.8	3.0	30.8	19.7
N-S	1.4	0.2	1.7	1.2	1.6	0.2	2.0	1.2
TOC-St	75.8	30.5	113.4	32.1	60.1	38.0	129.6	17.4
N-St	7.8	0.55	8.7	7.0	-	-	-	-
CO <sub>2</sub> -C	0.5	1.3	5.2	1.4	1.1	3.7	13.7	2.8

<sup>&</sup>lt;sup>a</sup> Values of C-HU, C-HA, C-FA, C-WSA, C-Labile, TOC-S, N-S, TOC-St, and N-St in g kg $^{-1}$ . The values of C-Mic and emission, respectively, are in  $\mu g$  g $^{-1}$  ha $^{-1}$  and g CO<sub>2</sub>-C m $^{-2}$  h $^{-1}$ 



Fig. 2 Carbon losses (C-Losses) of residue and soil (g kg<sup>-1</sup>), and cumulative emission of CO<sub>2</sub>-C (g kg<sup>-1</sup>) of soil with residue of soybean, *Brachiaria*, sugarcane, and treatment control in two contributions of the residue crops in agronomic rate (AR) and constant rate (CR) in 48 days after incubation



### 3.3 Multivariate analyses of variables

In the principal component analysis (PCA), there was the formation of a two-dimensional plane (Fig. 4) with three principal components (PC), PC1, PC2, and PC3, accounting for 70.02 % of the original information. The first PC1 has 36.95 % of variance, followed by CP2 with 20.67 % and CP3 with 12.40 %. This result is in agreement with the criteria established by Sneath and Sokal (1973), wherein the number of PCs used in the interpretation must be such that it explains at least 70 % of the total variance.

The most significant attributes retained in PC1 are related to the soil CO<sub>2</sub>-C emission process. The C-HA and C-FA are related directly (same sign) with the emission of CO<sub>2</sub>-C during the initial soil incubation period (1, 3, and 6 DAI).

The C-HU attribute is inversely related to the CO<sub>2</sub>-C emission from the soil (Table 4). The C-HU had mean values ranging from 12.0 to 12.4 g kg<sup>-1</sup> C at the initial time (Table 2) and 17.0 to 18.7 g C kg<sup>-1</sup> at the end time (Table 3). In PCA, the C-HU positioned near the end of the incubation period (12 and 48 DAI) showed a close relation (Fig. 4). The mean values of the C-HU were above the averages obtained in the C-FA and C-HA at the two times (Tables 2 and 3).

At the initial time (Table 2), the C-FA obtained the higher average C amount ranging from 3.4 to 3.7 g kg<sup>-1</sup>

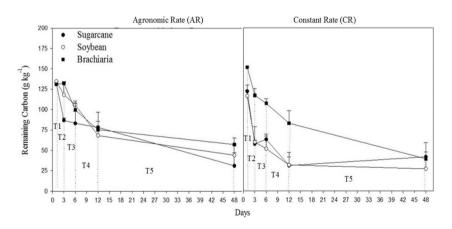
( $\pm 0.3$  to 1.1) when compared with the end time, 1.8 to 2.2 g C kg<sup>-1</sup> ( $\pm 0.3$  to 0.7) (Table 2). Decreases were also observed for the C-HA fraction, with averages from 2.1 to 2.3 g C kg<sup>-1</sup> ( $\pm 0.3$  to 0.7) at the initial time (Table 2) and 1.0 to 1.1 g C kg<sup>-1</sup> ( $\pm 0.3$  to 0.7) at the end (Table 3). These variables showed high positive correlation, very close vectors at the initial time (Fig. 4).

In PC2, the  $CO_2$ -C emission is proportionally related to the TOC-S and N contents, respectively, of the soil and residue (N-S and N-St). However, it is inversely related to the TOC-St (Table 4). The higher the amount of N in the soil and residue, as well as TOC-S, the higher the  $CO_2$ -C emission. However, the TOC-St in the residue contributes to lower the  $CO_2$ -C emissions.

The N-S was also retained in PC3, providing little information and no significant correlation with the other variables (Fig. 4 and Table 4).

The TOC-S showed averages ranging from 22.4 to 23.4 g C kg<sup>-1</sup> ( $\pm 1.5$  to 2.8) in the initial period and 21.1 to 22.8 g C kg<sup>-1</sup> ( $\pm 1.5$  to 3.0) at the end (Tables 2 and 3). Lower C-Mic levels in the soil without added residue occurred in the two times, respectively 1631.8 to 2515.1  $\mu$ g g<sup>-1</sup> ha<sup>-1</sup> and 1276.1 to 2059.2  $\mu$ g g<sup>-1</sup> ha<sup>-1</sup>. When residue was added to the soil, there occurred 18.13, 39.74, and 30.32 % reductions of C-Mic during the experiment, respectively, for the sugarcane, soybean, and *Brachiaria* residue (Tables 2 and 3).

Fig. 3 Remaining carbon from of residue of soybean, *Brachiaria*, and sugarcane in two contributions of the residue crops in agronomic rate (AR) and constant rate (CR) (divided in 5 times T1, T2, T3, T4, and T5, respectively 1, 3, 6, 12, and 48 days after incubation of soil)





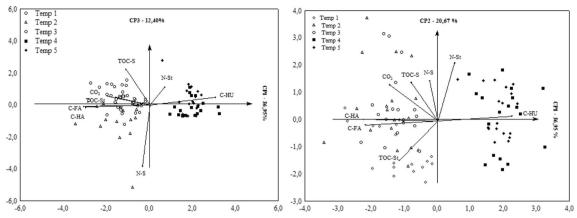


Fig. 4 Component analysis (PCA) with the variables CO<sub>2</sub>-C emission, soil organic nitrogen (N-S) and residue (N-St) and the soil organic carbon (TOC-S) and residue (TOC-St), humic substance carbon (C-HS) fractionated in fulvic acids (C-FA), humic acid (C-HA), and humin (C-HU)

The C-Mic showed a positive correlation with  $\mathrm{CO_2}$ -C emission (r=0.70). So with increase of the C-Mic in the soil, an increase of emission from soil occurred (Fig. 5). However, this variable was not used in the principal component since it did not retain a large amount of information as well as the C-labile.

#### 4 Discussion

# 4.1 Effect of residue incubation time on the CO<sub>2</sub>-C emission

The low CO<sub>2</sub>-C emission in the treatment without residue is due to the absence of organic substrate and decreases in microbial activity. Similar results were found by Babujia et al. 2010. The emission of the CO<sub>2</sub>-C results from the decay of the soil C, mainly the C-WSC, C-HA, C-FA, TOC-S, and C-HU

**Table 4** Correlation coefficient of the main components (CP1, CP2, and CP3), carbon of humin (C-HU), fulvic acid (C-FA), humic acid (C-HA), total organic carbon and nitrogen organic (TOC-S/N-S) and residue carbon (TOC-St), nitrogen (N-St), and emission of CO<sub>2</sub>-C

Attribute	CP1 <sup>a</sup> (36.95 %)	CP2 <sup>a</sup> (20.67 %)	CP3 <sup>a</sup> (12.40 %)
C-HU	0.88	0.06	0.10
C-HA	-0.87	0.02	-0.02
C-FA	-0.85	-0.05	-0.03
TOC-S	-0.31	0.50	0.46
N-S	-0.09	0.53	-0.79
TOC-St	-0.45	-0.53	0.10
N-St	0.20	0.77	0.23
CO <sub>2</sub> -C	-0.56	0.50	0.11

Correlations in italic (>0.50 in absolute value) were regarded as highly significant in the interpretation of the main significant

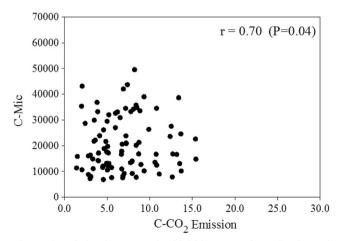
<sup>&</sup>lt;sup>a</sup> These values represent the variance total in component 1 (CP1), 2 (CP2), and 3 (CP3)



fractions (Badía et al. 2013; Knicker et al. 2013). The emission of soil CO<sub>2</sub>-C in the absence of substrate is inversely related to the stored organic C and the potential loss of soil C (Cookson et al. 1998).

The low TOC-S variation is due to its colloidal and physical stability considering the addition of all fractions with a high degree of affinity with the soil particles (Stevenson 1994). In the clay fraction, the TOC-S is stabilized primarily by its intimate association with soil minerals, which protects against biological degradation (Kaiser et al. 2002), implying greater TOC stability and consequently less significant changes.

The peaks in the CO<sub>2</sub>-C emissions from the soil with residue were for 6 days of incubation followed by an exponential decrease until the end of the experiment with 48 days of incubation. The quality and quantity of residue affects the increase of CO<sub>2</sub>-C in the first 6 days. Depending on the type of plant material, such increases can be reduced to hours



**Fig. 5** Correlation between microbial biomass carbon (C-Mic) and cumulative emission of CO<sub>2</sub>-C (g C-CO<sub>2</sub> m<sup>-2</sup> h<sup>-1</sup>) of soil with residue of soybean, *Brachiaria*, sugarcane, and treatment control in two contributions of the residue crops in agronomic rate (AR) and constant rate (CR) in 48 days after incubation

(Franzluebbers et al. 1994; Wang et al. 2004), considered as an initial rapid phase (Duong et al. 2009).

Soil  $\mathrm{CO_2}$ -C emission peaks after 3 days of incubation followed by an exponential decrease until the end of the experiment with 56 days of incubation were obtained by Guilou et al. (2011) working with  $\mathrm{CO_2}$ -C emission rates in Luvisol with surface addition of wheat residue. Cayuela et al. (2009), evaluating cotton wool incubated on the surface of a Regosol for 25 days, also showed that initial peak in the emission of  $\mathrm{CO_2}$ -C, as did Almeida et al. (2014) incubating sugarcane residue in Latosol for 79 days.

These initial increases in the CO<sub>2</sub>-C emission are related to the addition of 5 to 20 Mg ha<sup>-1</sup> of the plant material and microbial biomass activity (C-Mic), with higher expression in the treatments with soybean, *Brachiaria*, and sugarcane residue, respectively, of 27.13, 22.94, and 3.80 % during the first period (Tables 2 and 3). Under these conditions, the CO<sub>2</sub>-C emission in the soil is the result of microbial respiration (C-Mic) with greater activity during the decomposition of organic matter (Luo et al. 2006), the C-Mic considered as a fraction of easy and rapid decomposition (Six et al. 1999; Duong et al. 2009), and with high correlation with the CO<sub>2</sub>-C emissions (Cheng and Coleman 1990).

The high soil  $CO_2$ -C emission contributes to the reduction of sugars, while the lignin percentage concomitantly increases (Aneja et al. 2006) the decomposition of starch and proteins as well as some carbohydrates that start at a lower rate (Gunnarsson et al. 2008), among them the C and N present in the compounds of fructans. Another important aspect to note is that, in general, with the addition of the residue,  $CO_2$ -C emissions show a monotonic decrease over time, some more pronounced than others, which is characteristic of a behavior induced by the carbon availability of the organic matter in the labile fraction added in the beginning of the experiment (t=0) (Scala et al. 2008).

The greatest increase in soil CO<sub>2</sub>-C emission with the addition of the residue causes high metabolic activity of the microorganisms which are able to degrade not only the C of the residues but also of the soil (Badía et al. 2013). This result is explicit in PC1 with inverse correlations at the final time of the CO<sub>2</sub>-C (r=-0.56) with the C-HU (r=0.88) (Table 4), the most recalcitrant fraction of the humic substances (Duong et al. 2009; Zavalloni et al. 2011). Thus, as described by Fontaine et al. (2007), with the higher respiration, there is a decrease of the more stable SOM fractions. This is known as the priming effect because with the high soil respiration and the addition of plant residues, the breakdown of a few more resistant organic matter fractions is stimulated (Kuzyakov et al. 2000). However, residues with a high C/N ratio and lignin quantity contribute to increases in the amount of the C-HU fraction (Martens 2000) but require a longer time (decades).

The C-HU had the highest concentration among the soil HS. This result was also found by Conteh et al. (1998), Canellas et al. (2003), Assis et al. (2006), and Rossi et al. (2011) due to increased stability caused by the strong interaction with the soil mineral fraction (Stevenson 1994).

In the PC1, the CO<sub>2</sub>-C emission (r=-0.56) was positively correlated with C-HA (r=-0.87) and C-FA (r=-0.85) fractions (Table 4). Besides, the C-FA fractions have the characteristic highest mobility, solubility (Andreux and Becerra 1975), and mineralization rate among the HS fractions (Fontana et al. 2006).

Franzluebbers et al. (1994) and Wang et al. (2004) also found the decrease and subsequent stability of CO<sub>2</sub>-C emissions after 12 days of incubation in all treatments with added residue. These results are due to the high concentration of recalcitrant compounds, such as lignin and cellulose and other macromolecules (Wang et al. 2004), considered a slower phase during which the most recalcitrant fraction is decomposed (Duong et al. 2009).

# 4.2 Effect of quantity and quality of the residue added to the soil on CO<sub>2</sub>-C emission

The increase in soil CO<sub>2</sub>-C emission is closely related to the quality and quantity of residue added to soil. With the addition of CO<sub>2</sub>-C, a concomitant decrease in the TOC-St occurs (Stevenson 1994). Higher reductions of TOC-St in the 20 Mg ha<sup>-1</sup> dose of sugarcane residue, followed by doses of 8 and 5 Mg ha<sup>-1</sup> of soybean and *Brachiaria*, are due to increased availability of substrate since even with the higher C/N ratio (83.6 g N kg<sup>-1</sup>) and lignin (25.8 %), besides the lower N amount (1.4 g N kg<sup>-1</sup>) and crude protein (2.50 %), the sugarcane residue showed a higher amount of easily degradable material (Table 1).

Adding large amounts of residue with a high C/N ratio causes an increase in the amount of C in the organic matter, compared with the addition of soybean residue with a lower C/N (Mazzilli et al. 2014) and high quantity of crude protein (Table 1).

The quality of plant material contributed to the increased CO<sub>2</sub>-C emission in the soil from the decomposition, mainly of residue. This loss of soil C was more pronounced with the addition of soybean residue because of a higher concentration of labile materials or materials readily metabolizable by microbial activity (Cunha et al. 2011). This is because with the decomposition and mineralization of soybean residue, there is N release into soil and to microorganisms (Siczek and Lipiec 2011), increasing the biological activity due to the essential nature of N for the development of microorganisms, associated to the synthesis of compounds such as amino acids and enzymes (Stevenson 1994). This increases the concentrations of free sugars, starches, and pectins compared to grasses (Gunnarsson et al. 2008).



With high soil N availability and unavailability of plant material, microorganisms act by decomposing the C present in the soil and residue, promoting higher CO<sub>2</sub>-C emissions, since the CO<sub>2</sub>-C has a high correlation with the TOC-S (Panosso et al. 2011), and about 80 % of the plant material C is released to the atmosphere in the form of CO<sub>2</sub>-C and with an increase of 20 % in the soil in different fractions and decomposition stages (Brady and Weil 2008).

Our results suggest that the quality of the residue is very important for the increase in CO<sub>2</sub>-C emission. This condition is preponderant for the time and intensity of the microbial activity in the soil, with consequent higher CO<sub>2</sub>-C emission and C loss from the soil and added residue. Residue added in soil with C/N ratio under 20 decomposes rapidly, while materials with higher C/N ratios decompose slowly and may immobilize N (Sanchez et al. 1989).

The decomposition can be evaluated with other methods like N concentration and C/N, cellulose, and lignin ratios as suggested by Berg (1986). Those methods can be better because the nutrient release rate during the crop residue decomposition, especially the C/N ratio, depends on the characteristics of the species of plant (Giacomini et al. 2003).

The 18.17 % N-St increase from sugarcane added to the soil should be immobilized. The high C/N ratio of sugarcane, ranging from 90 to 120, promotes N immobilization in the residue in the absence of nitrogen fertilization (Meier et al. 2006). N immobilization in the sugarcane residue was also found by Almeida et al. (2014).

In the treatment with *Brachiaria* residue, there occurred consistency in the amount of TN (Tables 2 and 3) even with a high C/N ratio (Table 1). Possibly, the constancy in TN content in *Brachiaria* residue (Maciel et al. 2003) may be due to allelopathic effects (Neal 1969) associated to soil microorganisms that can act in inhibiting available nutrients (Olmsted and Rice 1970).

#### **5 Conclusions**

The quality and quantity of residue added to soil influences the carbon sequestration and C-CO<sub>2</sub> emission. In the first 6 days of incubation, there was a higher C-CO<sub>2</sub> emission ratio with the less stable soil carbon compartments (C-HA, C-FA, N-S) as well as residue (TOC-St, N-St). In the final period, there is no difference between quality and quantity of residue added to soil.

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