RECALCITRANT CARBON AND NITROGEN IN AGRICULTURE SOILS WITH RESIDUE ACCUMULATION AND FERTILIZATION UNDER TROPICAL CONDITIONS

ABSTRACT: Soil organic matter has a strong relation to total organic carbon, and about 85% of organic carbon consists of humic substances (HS), classified as humin (HU), humic (HA) and fulvic acids (FA), and denominated as recalcitrant carbon in soil. HS are formed by complex, heterogeneous and polydisperse molecules, which have significant influence on the soil physical and chemical characteristics. The study evaluates the hypothesis that agricultural soils treated with organic residues may present higher carbon stocks as presented in forest soils. The aim of this study was to evaluate alterations in recalcitrant carbon and nitrogen stocks in Oxisol (Forest - FL, unfertilized Brachiaria - UB, and fertilized Brachiaria - FB) and Cambisol (Forest - FC, Coastcross - CC, sugarcane - CA, and silage corn - SM) at surface (0.0 - 0.1) and subsurface (0.1 - 0.2 m), in the Zona da Mata in Minas Gerais state, Brazil. Result shows that fertilization, low soil disturbance and residual removal promoted increase of C and N content in HS, being close to native forests. Both, carbon and nitrogen recalcitrant, presented reduction with soil depth. HU ranged from 10.5 to 16.7 g kg$^{-1}$ and presented the highest concentration compared to FA and HA. In Oxisol, FL and FB presented the highest SH concentration and demonstrate the positive effect of fertilization on carbon stocks improving soil quality in well-managed and productive areas. While, In Cambisol, FC and CC presented higher carbon and nitrogen in HS, mainly for HU and HA fractions, and SM showed the lowest concentrations in all fractions. Our results suggest that soil managements with lower soil disturbance and residual removal promotes increasing of carbon and nitrogen in recalcitrant fraction, with concentration close to native forests. Pasture should be fertilized to improve recalcitrant carbon and nitrogen stocks, avoiding process of degradation in tropical soil. It is an important outcome due to high levels of degraded areas in Brazil caused by inadequate use of soil mainly with pasture.

KEYWORDS: Brachiaria pasture. Humin. Fulvic acid. Humic acid. Organic matter

INTRODUCTION

Soil organic matter (SOM) is considered a C reservoir in the terrestrial environment with concentration three times higher than the atmospheric (SMITH, 2012). SOM improves soil structure, fertility, and crop yields (PANETTIERI et al., 2017), as well as SOM affects soil density and porosity (ALMEIDA et al., 2014) and nutrient cycling and availability (COLLETO et al., 2015).

SOM play role on soil preservation, mitigating erosion, improving soil water infiltration and retention and stabilizing soil structure (STEVenson, 1994). Therefore, management of SOM stock is the key to effective ecological intensification and sustainability (TSIAFOULI et al., 2015), promoting higher soil organism abundance and diversity (GARRATT et al., 2018).

The capacity of SOM stocks is intrinsic to the ecosystem and depends on several factors, such as vegetation structure and composition, rainfall regime, soil moisture, temperature and nutrient contents (STOCKMANN et al., 2013; SÁNCHEz–GONZÁLEZ et al., 2017), as well as soil texture and mineralogy (ZINN et al., 2011).

SOM has positive and strong correlation to total organic carbon (TOC) (Blair et al., 1995; Stevenson, 1994), and about 85% of TOC consists of humic substances (HS), classified as humin and humic and fulvic acids (STEVenson, 1994) and denominated as recalcitrant carbon in soil. HS are formed by complex, heterogeneous and polydisperse molecules, which have significant influence on the
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soil physical and chemical characteristics (SILVA et al., 2014).

HS stock can be used to understand evolution and degradation processes in soil, as well as support the conservation management system (CUNHA et al., 2001). Studies on these aspects should be encouraged to support mitigate global climate-change (SOLLINS et al., 2017) and identify where human factors are significantly contributing to carbon losses into the atmosphere (AKPA et al., 2016), as a soil sustainable indicator (SALTON et al., 2011).

According to Panettieri et al. (2017), changes on SOM dynamics under different land uses are still poor understood. Thus, incubation research and field trials are needed to compare the old and new SOM understanding (SMITH et al., 2018). New studies will help to develop alternative management practices to support the expected intensification of food production (PICCOLLO et al., 2018).

To test hypotheses that area in use of forest presents higher carbon stocks and is similar to agriculture use with residues accumulation on soil and fertilization, the aim of this study was to evaluate alterations in total and recalcitrant carbon and nitrogen stocks in Oxisol and Cambisol under different uses and managements in the Zona da Mata.

MATERIAL AND METHODS

The study was carried out in areas belong to EMBRAPA Dairy, located in Coronel Pacheco, Minas Gerais, Brazil (21°14’S latitude; 43°15’W longitude; 435 m altitude). The climate is classified as Aw according to Koppen, with an average annual temperature and rainfall of 21°C and 1581 mm, respectively.

The experiment tested three soil uses (Forest - FL, unfertilized Brachiaria - UB and fertilized Brachiaria - FB) in a Oxisol, and four soil uses (Forest - FC, Coastcross - CC, Sugarcane - SC and silage corn - SM) in alluvial Cambisol. Both soils, were tested at two layers (0.0 - 0.1 m and 0.1 - 0.2 m), using four replicates.

In Oxisol, pasture was cultivated with Brachiaria sp. since 2003, and fertilized with nitrogen (N), phosphorus (P) and potassium (K) using a dose of 250 kg ha\(^{-1}\) of 20-05-20 every year, in fertilized treatment. This area was deforested in 1950 and angola grass (\(Panicum\) numidianum) was initially planted, succeeded in 1986 by elephant grass (\(Pennisetum\) purpureum) until 2011 (Table 1).

### Table 1. In Oxisol, management and use history with Forest (FL), unfertilized brachiaria pasture (UB) and fertilized brachiaria pasture (FB) located in Coronel Pacheco, Brazil.

| Soil use | Soil use and management |
|----------|-------------------------|
| FL       | Secondary Atlantic Forest |
| UB       | 1950: Deforestation 1950 – 1985: Angola grass 1986 – 2011: Brachiaria pasture |
| FB       | 1950: Deforestation 1950 – 1988: Angola grass 1989 – 2002: Elephant grass 2003 – 2011: Brachiaria pasture |

In Cambisol, area with Coastcross grass (\(Cynodon\) dactylon L.) was implemented in 1992, fertilized with 10 kg ha\(^{-1}\) of 20-20-20 (N, P, and K). In 2002, this rate was changed to 1 Mg ha\(^{-1}\) of formulation 05-20-20, with the addition of 50 kg ha\(^{-1}\) of the N top dressed in summer. Liming was performed at the time of planting with 3 Mg ha\(^{-1}\) of limestone and following planting with 1 Mg ha\(^{-1}\) every two years. By 1979, this area had become mostly Angola grass without fertilizer management, which was succeeded in 1980 until 1982 with Oat (\(Avena\) strigosa) intercropped with ryegrass (\(Lolium\) multiflorum) under irrigated pasture management in winter.

Silage corn area has been established since 1986, before that angola grass without fertilization was then established until 1985, vegetable crop species up to 1973, and clearing performed in 1935. At sugarcane area (\(Saccharum\) offinarum L.) implementation was in 1990, which received fertilizer only up to the fifth year at a dose of 300 kg ha\(^{-1}\) of 8-30-16 with producing around 70 Mg ha\(^{-1}\) in the first four years and decreasing to 40 Mg ha\(^{-1}\) due to the lack of proper management. In the history of...
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The area after deforestation in 1935, then Angola grass establishment until 1989 without a specific management. From 1983 to 1991 there was the implantation of summer grain corn (Table 2).

Table 2. In Cambisol, management and use history with Forest (FL), pasture with Coacross (CC), sugarcane (SC) and silage corn (SM) located in Coronel Pacheco, Brazil.

| Soil use | Soil use and management |
|----------|-------------------------|
| FC       | Secondary Atlantic Forest|
|          | 1935: Deforestation     |
|          | 1935 - 1979: Angola grass|
|          | 1980 - 1982: Pasture oats|
|          | 1983 - 1991: Summer grain corn|
|          | 1992 - 2011: Coastcross  |
| CC       | 1935: Deforestation     |
|          | 1935 - 1989: Angola grass|
|          | 1990 - 2011: Sugarcane cultivation|
|          | 1935: Deforestation     |
|          | 1935 - 1973: Cultivation of vegetable species|
| SC       | 1973 - 1985: Angola grass|
|          | 1986 - 2011: Silage corn |

Variables determination and Statistical analysis

Samples were collected in disturbed conditions at the surface (0.0 - 0.1 m) and subsurface layers (0.1 - 0.2 m) and forwarded to the laboratory to determine total soil organic carbon - TOC (YEOMANS; BREMNER, 1988) and total organic nitrogen - TON (TEDESCO et al., 1995). Humic substances were identified according to the technique of differential solubility, separating the fulvic acids (C-FA), humic acids (C-HA) and humin (C-HU) in accordance with International Humic Substances Society (Hayes et al., 1989; Yeomans and Bremner, 1988). After HS fractionation, we determined the amount of N (N-HS) in each of the fractions (C-HA, C-FA and C-HU) via sulfuric digestion with subsequent Kjeldahl distillation (TEDESCO et al., 1995). The variables were submitted to analysis of variance (ANOVA) by the F-Test and compared according to the Tukey-Test ($p < 0.05$).

RESULT AND DISCUSSION

TOC and TON results

TOC presented a variation between 1.2 to 3.3 %, decreasing at soil depth with higher difference at FC (± 0.8 %), following CC (± 0.7 %), Figure 1. TOC decrease with soil depth has been reported in various articles (COLLETO et al., 2015; ALMEIDA et al., 2014; SANTANA et al., 2011; PULROLNIK et al., 2009), with highest TOC in surface due to accumulation and decomposition of vegetal and animal materials (ROSCOE et al., 2006), which depending on quality and quantity of the residues to increase TOC in soil (ALMEIDA et al., 2016).

In Oxisol, FL and FB presented similar TOC stocks, varying between 2.6 and 2.7 g kg$^{-1}$ (Figure 1). This similarity is due to soil fertilization and liming (at implantation), which contributes to better vegetative development of pasture and higher production of organic material deposited on the soil. It is interesting result and shows how important is soil fertilization in pasture, mainly in areas with high level of degradation.

Poroto et al. (2009) and Santana et al. (2011) have observed similar TOC levels between pasture areas and native forest. Generally, Forest presents higher SOM and TOC levels compared to other vegetation coverage due to soil management with soil movement, promoting water erosion and increasing organic residue decomposition (JAKELAITIS et al., 2008; CUNHA et al., 2012).

In Cambisol, SM presented the lowest TOC in both layers, with variations from 1.2 to 1.4 g kg$^{-1}$ TOC (Figure 1). It clearly is a result of soil intense management with tillage, which provides an increase in the oxidation of organic matter. On the other hand, CC even being agriculture area, resulted in the highest TOC, with an increase of 49 % compared to SC at 0.0 - 0.1 m layer (Figure 1).
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Figure 1. Total organic carbon (TOC; %) and nitrogen (TON; %) in a Oxisol (Forest - FL, unfertilized Brachiari - UB and fertilized Brachiari - FB) and Cambisol (Forest - FC, Coastercross - CC, Sugarcane - SC and silage corn - SM) at the surface (0.0 - 0.1 m) and subsurface (0.1 - 0.2 m). Bars identified with distinct lowercase letters (soil uses) and uppercase (layer) differ by the Tukey test (p <0.05).

CC result was not expected is due to absence of soil tilling operations, which maintains the carbon physically protected from microbial attacks (STEVenson, 1994). Neufeldt et al. (2002), Pulrolnek et al. (2009) and Almeida et al. (2014) have reported similar results, and according to Jantalia et al. (2007), soil preparation has a negative effect on TOC stocks up to 30 cm deep in relation to the native Forest area.

TON was higher in the surface layer in all uses, with high relation to TOC accumulation (Figure 1) but was not possible to observe that relation on SM and SC (Figure 1). Probably, this uniformity at SM and SC between the layers is due to more intense and mechanized operations and the history of the cultivation (Table 2).

In Oxisol, there was higher TON at FL and FB as well observed to TON, with significant difference to UB at the surface layer (Figure 1). As well as, there was higher TON accumulation in FC and CC in Cambisol (Figure 1). The highest concentration of TOC and TON in FL and FC, was expected and is due to greater stability and less human intervention, which results in constant maintenance of plant residues and microorganisms responsible for mineralization. Generally, TON content is fall rapidly in soil cultivated with cereals, legumes and pasture due to N needs of microorganism (Almeida et al., 2016; Stevenson 1994).

Humic Substance results

HU ranged from 10.5 to 16.7 g kg\(^{-1}\) and presented the highest concentration compared to FA and HA (Table 3). This is a common outcome and has been presented by Assis et al. (2006), Rossi et al. (2011) and Martin et al. (2015). Generally, HU presents higher concentration due to strong resistance to microbial degradation and the strong association to soil mineral fraction, especially in oxide soil (Stevenson, 1994). We can also point out that HU presents higher molecular size and greater degree of stability as characteristics. Therefore, FA and HA are less stable and can be polymerized or mineralized faster by soil microbial activity (Fontana et al., 2006; Volkoff et al., 1978).

In Oxisol, FL and FB presented the highest humic substances concentration and demonstrate the positive effect of fertilization on carbon stocks improving soil quality in well-managed and productive areas. Higher HU and FA in soil also were found by Santana et al. (2011) comparing pasture with mineral fertilization and native pasture.

According to Martins et al. (2009), areas with brachiaria tends to present higher HU concentrations due to accumulation and decomposition of leaves and stems at the soil. Residues that presents high lignin content promote higher accumulation of SH in soil. HS presented higher N in surface with increases of 27 %, 20 % and 7 % compared to subsurface, for the HU, HA, and FA, respectively (Table 3).
Table 3. Carbon and nitrogen humic (HU), humic acids (HA) and fulvic acid (FA) in Cambisol (Forest - FL, pasture with Coastcross - CC, sugarcane - SC and silage corn - SM) at the surface (sur; 0.0 - 0.1 m) and subsurface layer (sub; 0.1 - 0.2 m).

| Soil Use | C-HU     | C-HA     | C-FA     |
|----------|----------|----------|----------|
| FC       | Sur      | Sub      | Sur      | Sub      | Sur      | Sub      | Mean     |
|          | 21.9aA*  | 12.3abB  | 5.6aA    | 2.5aB    | 4.5      | 3.7      | 4.1A     |
| CC       | 23.1aA   | 12.7aA   | 4.5aA    | 2.1aB    | 3.6      | 2.7      | 3.2b     |
| CA       | 23.2aA   | 9.7bB    | 3.0cA    | 2.3aA    | 3.0      | 2.7      | 2.9b     |
| SM       | 10.8bA   | 9.8bA    | 0.9dA    | 0.7bA    | 1.8      | 1.5      | 1.6c     |

| Soil Use | N-HU     | N-HA     | N-FA     |
|----------|----------|----------|----------|
| FC       | Sur      | Sub      | Sur      | Sub      | Sur      | Sub      | Mean     |
|          | 1.5bA    | 0.9aB    | 0.8      | 0.5      | 0.4      | 0.3      | 0.3b     |
| CC       | 2.1aA    | 1.1aB    | 0.7      | 0.5      | 1.1      | 1.0      | 1.1A     |
| CA       | 0.9cA    | 0.8aA    | 0.5      | 0.4      | 0.5      | 0.4      | 0.5b     |
| SM       | 0.9cA    | 0.8aA    | 0.3      | 0.3      | 0.2      | 0.2      | 0.2b     |

*Means following distinct lowercase letters in the column (soil layer) and the uppercase on the line (soil use) differ by the Tukey test (p < 0.05).

In Cambisol, FC and CC presented higher carbon and nitrogen in humic substances, mainly for HU and HA fractions. While, SM showed the lowest concentrations in all fractions (Table 3). According to Cunha et al. (2001), the FL presented this result because of the drastic and intrusive management with soil tillage, combined with the removal of the plant part (harvest) which contributes to the loss of soil quality by reducing the amount of C-HS.

According to Stevenson (1994) and Six et al. (2002), soil disturbance disrupts the aggregates, exposing the intra-aggregate C, making the C more available to microbial attack and reducing the amount of carbon and nitrogen in HS.

CONCLUSIONS

Soil managements with lower soil disturbance and residual removal promotes increasing of carbon and nitrogen in recalcitrant fraction, with concentration close to native forests.

Pasture should be fertilized to improve carbon and stocks, avoiding process of degradation in tropical soil. It is an important outcome due to high levels of degradation areas in Brazil caused by inadequate use of soil mainly with pasture.

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RESUMO: A matéria orgânica do solo tem uma forte relação com o carbono orgânico total, cerca de 85% do carbono orgânico é composto por substâncias húmicas (HS), classificadas como humina (HU), ácidos húmicos (HA) e fúlvicos (FA), denominadas como carbono recalcitrante no solo. As HS são formadas por moléculas complexas, heterogêneas e poli-dispersas, que exercem influência significativa nas características físicas e químicas do solo. O estudo avalia a hipótese de que solos agrícolas tratados com resíduos orgânicos podem apresentar maiores estoques de carbono como os encontrados em solos florestais. O objetivo deste trabalho foi avaliar as alterações nos estoques de carbono e nitrogênio recalcitrantes em Latossolo (Floresta - FL, Brachiaria não fertilizada – UB, e Brachiaria fertilizada - FB) e Cambissolo (Floresta - FC, coastcross - CC, cana-de-açúcar e milho para silagem - SM) na superfície (0,0 - 0,1 m) e sub-superfície (0,1 - 0,2 m), na Zona da...
Mata em Minas Gerais, Brasil. Os resultados mostram que a adubação, a baixa perturbação do solo e a remoção dos resíduos promoveram aumento do teor de C e N na HS, estando próximos às florestas nativas. Ambos, carbono e nitrogênio recalcitrantes, apresentaram redução com a profundidade do solo. A HU variou de 10,5 a 16,7 g kg\(^{-1}\) e apresentou a maior concentração em relação à FA e HA. Em Latossolo, FL e FB apresentaram a maior concentração de SH e demonstraram o efeito positivo da adubação nos estoques de carbono, melhorando a qualidade do solo em áreas bem manejadas e produtivas. Enquanto, no Cambissolo, FC e CC apresentaram maiores teores de carbono e nitrogênio nas HS, principalmente nas frações HU e HA, e SM apresentou as menores concentrações em todas as frações. Nossos resultados mostram que manejos de solo com menor perturbação e remoção de resíduos promovem o aumento de carbono e nitrogênio em frações recalcitrantes, com concentração próxima às florestas nativas. A pastagem deve ser fertilizada para melhorar o estoque de carbono e nitrogênio recalcitrante, evitando o processo de degradação do solo tropical. Este é um resultado importante devido aos altos níveis de áreas degradadas no Brasil causadas pelo uso inadequado do solo, principalmente com pastagens.

PALAVRAS-CHAVE: Pastagem de *Brachiaria*. Humina. Ácido fúlve. Ácido húmico. Matéria orgânica

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