

Lipid abundance and composition of a humic Oxisol as a function of land use

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ABSTRACT: Soil organic matter of a humic Oxisol has been characterized by analyzing litter and soil lipid composition under vegetal covers (coffee, pasture and fragment of native forest). Soil and litter samples were collected from each site at two depths (0-10 and 60-100 cm). Lipid composition was determined by gas chromatography-mass spectrometry (CG-MS). In the solvent extracts of the soil lipids, even-numbered n-alkanoic acids in the range of C₁₆-C₃₂ indicate a soil organic matter input from higher plant waxes. The major compound classes in both the soil and the litter included saturated n-alkanoic acids, with average values in the total spectrum of 58 and 53%, respectively. Clear differences in lipid composition were found between the topsoil and subsurface. Long chain alkylic compounds (alkanoic acids and alkanes) accumulated preferably at depths of 60-100 cm. On the surface, the soil from natural vegetation had a total lipid value higher than those cultivated soils, and showed a predominance of unsaturated alkanic acids at 60-100 cm. Preservation of alkylic moieties with depth was observed under forest and pasture, indicating that these systems of land use are prone to lipid accumulation. Lipids, especially the long-chain (> C₂₀) ones, may be important with regard to C stabilization and humification processes in subsoil of humic Oxisols, highlighting the importance of lipids compounds in the bulk structure of organic matter these soils.

Key words: soil and litter lipid, soil management, tropical soil, grass, coffee

Abundância e composição de lipídios em Latossolo Húmico em função do uso do solo

RESUMO: A matéria orgânica de um Latossolo Húmico foi caracterizada por meio da análise da composição lipídica do solo e da serapilheira sob coberturas vegetais (café, pastagem e fragmento de mata nativa). Em cada área foram coletadas amostras de solo em duas profundidades (0-10 e 60-100 cm), assim como amostras da serapilheira. A composição lipídica foi determinada por cromatografia gasosa-espectroscopia de massa. No extrato lipídico do solo a ocorrência de ácidos alcanóicos com número par de carbonos (C₁₆-C₃₂) indicou presença de cera de plantas superiores na composição da matéria orgânica do solo. Tanto no solo quanto na serapilheira a principal classe de compostos incluiu n-alcanóicos saturados, com valores médios de 58 e 53% do total do cromatograma, respectivamente. Clara diferença foi encontrada entre os extratos lipídicos obtidos na superfície e subsuperfície do solo. Compostos alquílicos de cadeia longa (ácidos alcanóicos e alcanos) acumularam-se preferencialmente entre 60-100 cm. Superficialmente, o solo sob vegetação natural apresentou maiores teores de lipídios totais que os solos cultivados, mostrando também predominância de ácidos alcanóicos insaturados entre 60-100 cm. Nas áreas sob mata e pastagem houve, em profundidade, preservação de alcanos de cadeia curta; evidenciando maior conservação dos mesmos nestes sistemas de uso da terra. Os lipídios, especialmente os de cadeia longa (>20), podem ser muito importantes nos processos de estabilização e humificação do C em camadas subsuperficiais de Latossolos Húmicos, ressaltando a importância dos compostos lipídicos na composição da matéria orgânica destes solos.

Palavras-chave: lipídios do solo e serapilheira, manejo do solo, solo tropical, pastagem, cafeicultura

Introduction

The influence of soil management systems on the contents and quality of soil organic matter (SOM) of subtropical Brazilian soil and, consequently, on the properties and the ability of the soil to promote carbon sta-

bilization (sequestration) is a research subject of major interest. The general pattern of land use in the Zona da Mata Mineira region in the state of Minas Gerais, Brazil, is coffee (*Coffea arabica*) cropping and pastures of molasses grass (*Melinis minutiflora*) (Resende et al., 2002). Most of these cultivated areas are supported by

humic Oxisols because of their great friability, permeability and water retention capacity (Ker, 1999). Furthermore, these clay reddish-orange soils are acidic and characterized by the presence of a darker A horizon with humic fraction enrichment and an average C stock of 30.2 kg m⁻² (Andrade et al., 2004). The type of land use can affect the C stock in these soils, as well as the composition and quality of SOM added. Among the SOM fractions, lipids participate in the biogeochemical soil processes, as well as in the relationship between plants and their ecological environments (Quénéa et al., 2004; Jansen et al., 2006; Wiesenberg et al., 2006; Wiesenberg et al., 2008).

The lipids (alkanoic acids, alkanes, alkanols, ketones, wax esters, terpenoids, steroids, and phospholipids) are often described as organic compounds that are insoluble in water and soluble in organic solvents, with significant contribution in the aliphatic composition of SOM (Dinel et al., 1990; Stevenson 1994; Bull et al., 2000). In general, the accumulations of SOM rich in aliphatic compounds is linked to a high lipid input, particularly long-chain aliphatic compounds, mainly from vegetation, microbial activity and low soil pH (Bull et al., 2000).

The study of the composition of the total lipid extracts (TLEs) in both topsoil and subsoil layers may provide additional information about both compounds with value as plant-derived biomarkers of SOM sources and diagenesis products in the soil profile (Schulten and Schnitzer, 1990; Quénéa et al., 2004; Almendros et al., 2001; Jandl et al., 2007; Weisenberg et al., 2010). The soil lipid composition at the top layers in different agrosystems, may reflect the direct impact of the soil use mainly the influence of the plant litter or the entrance or preservation of organic contaminants (Weisenberg et al., 2006; Ahangar et al., 2009). Wiesenberg et al. (2010) observed changes in the soil lipid composition due to the vegetation change from green fallow (C₃ grass) to wheat (C₃ grass) cropping in a Gleyic Cambisol after four years. In turn, Zelles et al. (1995) observed differences between fatty acids in grassland and cultivated soils when monounsaturated fatty acid fraction was evaluated, showing that it is possible to evaluate changes in the land use through soil lipid composition.

Gas chromatography-mass spectrometry (GC/MS) and analytical pyrolysis are two techniques used to lipids characterization in plant and soil. Several studies have used these techniques to assess the concentrations, distribution pattern, and turnover rates of these compounds in the soil (Jandl et al., 2004; Feng et al., 2010).

As far as we know, studies on the lipid composition in Brazilian tropical soil are still rare. In this paper, we studied TLEs of these soils with two aims: i) to assess the influence of agricultural management practices (coffee and pasture) on the lipid composition of top and deep soil layers; ii) to evaluate differences in lipid composition of topsoil and subsoil in the darker A horizons.

Material and Methods

The study was done in the Atlantic Forest Biome region at "Zona da Mata Mineira", Southeast Brazil (20°28'26" S, 42°28'55" W, average altitude of 712 m a.s.l.). The typical climate of the region is subtropical with rainy summers and dry winters. The mean annual rainfall and temperature are 1300 mm and 18°C, respectively. The vegetation in the area can be classified as a Mountain Semi-deciduous Mesophytic forest (Semi-deciduous tropical forest) with emergent canopy (*Fme*) (IBGE, 1993).

The studied soil was described as a Typic Hapludox, clayey, dystrophic, and acid, with high content of exchangeable Al and organic matter. These soils are derived from migmatite rocks and are typical in the high altitude areas. For this study, three sites in the Zona da Mata Mineira with the typical pattern of land use (coffee, pasture, and forest) under the same taxonomic soil units and slope were sampled. The standard farming exploration when established at the beginning of the settling caused continuous deforestation, replacing the native tropical forest with cultures that became traditional in the region (coffee plantations and for other farming and pastures).

The soil under coffee has been cultivated for the last 12 years and the fertilization management consists of the addition of NPK (25-10-20) fertilizer twice a year and lime every two years. Before the coffee was established, the site was under pasture (*Melinis minutiflora*) during 40 years. The pasture sites were established in the 1940s after slashing and burning the forest. However, after livestock market and low revenues in the 1960s, a great part of this area was converted to coffee plantation. In the region the major area that once was devoted to pastures is now degraded and farmers keep only the cleared areas for cattle grazing, without much management. The molasses grass (*Melinis minutiflora*) predominates in the pasture, and the generalized occurrence of this grass is indicative of low-production acid soils. For comparative purposes, soil samples under the native forest ecosystem were also sampled and studied.

Soil sampling was carried out at the beginning of the wet season in the year 2005. Due to the polychrome presented by A horizon, composite soil samples were taken from superficial (0-10 cm) and sub-superficial (60-100 cm) depths. Three plots of 100 m², each spaced 10 m apart, were sampled by boring five times each subplot with an auger. For each depth of each plot twenty sub-samples were collected and pooled. Soil samples were air dried at room temperature and sieved through a 2 mm mesh. The main physico-chemical characteristics of the soils are given in Table 1.

Litter samples were collected from the same subplots as the soil samples using a 0.25m² (0.5 × 0.5m) metallic platform; four sub-samples collected in an area of *circa* 100 m². For each plot the samples were thoroughly mixed to form a single sample, dried at 40°C in an oven, milled, weighed and analysed in triplicate. Litter (2 g) and soil (20 g) lipids were Soxhlet-extracted at approxi-

mately 70°C for 16h, using 200 mL of the mixture dichloromethane/methanol (3:1 v/v) (van Bergen et al., 1997). Total lipid content was gravimetrically estimated as percentage of dry soil. To discuss the data we used descriptive statistics, considering the standard errors of the mean. For the compound abundance we established the distribution patterns on the basis of the relative abundance of the compounds in the chromatograms.

The free lipids were methylated with trimethylsilyldiazomethane and silylated with *N,O*-bis(trimethylsilyl)-trifluoroacetamide (BSTFA) before analysis by gas chromatography-mass spectrometry (GC-MS). Separation of lipids compounds was achieved using a SE-52 fused silica capillary column (30 m × 0.32 mm i.d., film thickness 0.25 μm). The column oven temperature was programmed to increase from 40 to 100°C at 30°C min⁻¹ and then to 300°C at 6°C min⁻¹. Helium was used as carrier gas at a flow rate of 1.5 mL min⁻¹. Mass spectra were measured at 70eV ionizing energy. Individual compounds were identified by inspecting mass fragments, key single ion monitoring (SIM) for the different homologous series, by low resolution mass

spectrometry and by comparison with published mass spectra and computerized libraries (NIST and Wiley).

Results and Discussion

Lipid contents ranged from 0.72 to 1.49 g (kg soil)⁻¹ for the topsoil (0-10 cm) and from 0.61 to 1.12 g (kg soil)⁻¹ for the subsoil (60-100 cm) (Figure 1-B). The decrease in the TLE with the increase in depth (Figure 1-B) has been observed by other researchers (Ziegler and Zech, 1989; Amblès et al., 1998; Naafs et al., 2004). The extracted lipids accounted for 2.2 – 5.7% of soil C, with an average value of 3.23%. These lipid contents are consistent with the values suggested as typical by Stevenson (2004) (1.2 – 6.3%) and Diné et al. (1990) (4-8%).

The total amounts of TLE was higher in the coffee litter in comparison to pasture and forest litter (Figure 1-A). Although extractable soil lipids content was low under forest, it was higher than that found for the cultivated soils (Figure 1-B). Despite the relatively low values found for forest litter, the soil under forest has higher lipid content than cultivated soils. This result suggests the occur-

Table 1 – Average values for physico-chemical characteristics of two layers of the A horizon of the studied humic Oxisol under coffee, pasture and natural forest.

Depth	pH (H ₂ O)	Mg ²⁺	Ca ²⁺	CEC ^a	Al ³⁺	H+Al	TOC ^c	TN ^d	BD ^e	Sand	Silt	Clay
Coffee												
0-10	5.1	5.3	21.0	124.0	13.3	124.0	3.34	0.26	0.94	330	110	560
60-100	4.8	0.3	2.3	112.4	14.0	112.4	2.77	0.15	0.85			
Pasture												
0-10	4.9	nd ^f	5.3	126.0	14.0	126.0	3.55	0.26	0.96	290	70	640
60-100	5.1	nd	1.3	110.3	7.3	110.3	3.03	0.18	0.80			
Forest												
0-10	4.8	nd	0.6	117.0	1.0	115.5	2.61	0.27	1.05	330	110	560
60-100	5.0	nd	nd	102.4	12.0	102.3	2.67	0.21	0.86			

^aCation exchange capacity (pH 7.0); ^bH+Al = potential acidity; ^cTOC = total organic carbon; ^dTN = total nitrogen; ^eBD = Bulk density; ^fno detected.

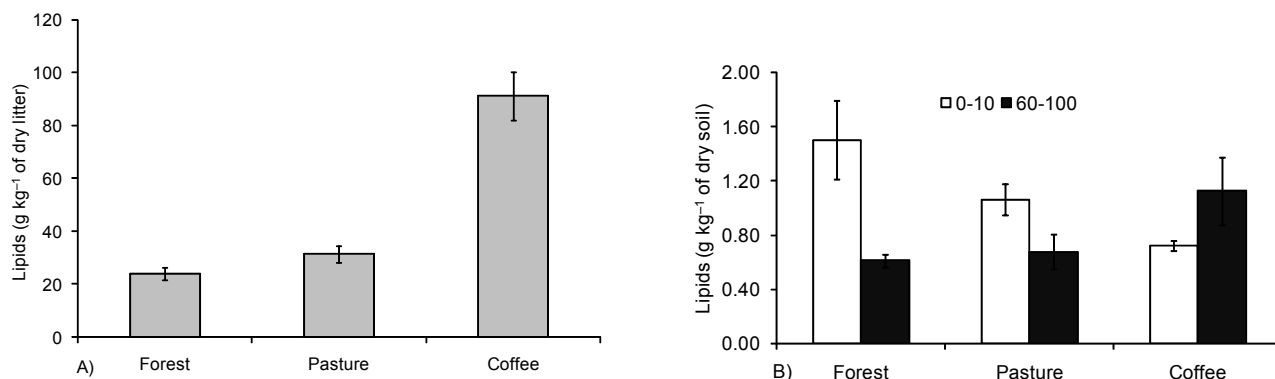


Figure 1 – Total lipid extracts from litter (A) and topsoil and subsoil (B) from humic Oxisol under coffee and pasture land use.

rence of accumulation or preservation mechanisms in the soil. It is possible that the biomass production in areas under forest, pasture and coffee, measured in 6.8, 3.2 and 2.8 Mg ha⁻¹, respectively (unpublished data), has favored the increase of lipid content in superficial soil layers in forest and pasture. Conversely, a higher lipid proportion in the deeper layers in soils under coffee may indicate that an important part of lipids from the litter is easily lixiviated along the soil profile (Nierop et al., 2005). In addition to possible lixiviation, the preservation of lipids at 60-100 cm in coffee may be favored by a low soil pH (4.8), as the acidic conditions can significantly inhibit the decomposition of lipid compounds (Dinel et al., 1990; Ziegler and Zech, 1989; Stevenson 1994).

Roots tissues (Nierop et al., 2005; Wiesenberg and Schawark, 2006; Jansen and Nierop, 2009) and aboveground biomass (Wiesenberg et al., 2010) have an important role in the soil lipid composition. Although in this study the contribution of root tissues could not be assessed, litter under forest can effectively contribute to topsoil lipid content, as compared to pasture and coffee uses. Soil lipids varied with the type of land use, more conservation systems (forest and pasture land use, respectively) presented increases in the soil lipid content. This increase in the lipid fraction will have a positive influence on the dynamics and stock of soil carbon.

Overall, saturated alkanolic acids were the most abundant class of compounds in the lipid soil fraction, with a dominance of long-chain over short chain compounds (Figure 2), as also found by several authors (Bull et al., 1998; Chefetz et al., 2002; Jandl et al., 2004; Quéneá et al., 2004). Alkanolic acids in soils may originate from plants and/or insects, oxidation of long chain alcohols,

and leaching down the soil profile (Nierop et al., 2005).

A homologous series of saturated alkanolic acids from C₈ (octanoic acid) to C₃₀ (triacontanoic acid) with a predominance of even numbered C-chain lengths, was observed both in the litter and soil extracts. Similar values were previously reported by Jandl et al. (2005) for Ap horizon of a Phaeozem with long-term cropping of rye and maize. The most abundant alkanolic acids in the soil samples, except the forest soil, were predominated by the ubiquitous C₁₆ and even long chain alkanolic acids (C₂₄-C₃₀). The chromatograms of the litter extracts showed maxima at C₁₆ and C₁₈. Their presence in the upper soil layer (Figure 2) is indicative of a direct contribution from the above vegetation (litter) to the soil lipid composition (Gleixner et al., 2001). In general, plant-derived signatures dominate our samples. In particular, unsaturated alkanolic acids (C_{18:1} and C_{18:2}) were most abundant in the litter and soil samples under forest (0-10 cm). The presence of an unsaturated C_{18:1} in the forest topsoil reflect the contribution of relatively fresh material, and indicates low to moderate level of lipid degradation due to the more rapid degradation of the unsaturated compounds in soil (Moucawi et al., 1981; Weisenberg et al., 2010). These alkanolic acids can also be selectively preserved in the soil with little or no alteration (Almendros et al., 1996; Nierop et al., 2005) and preferentially preservation in the clay fractions (Quéneá et al., 2004). In contrast with the litter lipid composition, the soil alkanolic acids present a bimodal distribution in the range of C₁₈-C₃₀, which is more evident in the deeper layers (60-100 cm). Thus, compared to layers 0-10 cm and 60-100 cm, a larger amount of long chain fatty acids (C₂₄-C₃₀) occurred in 60-100 cm.

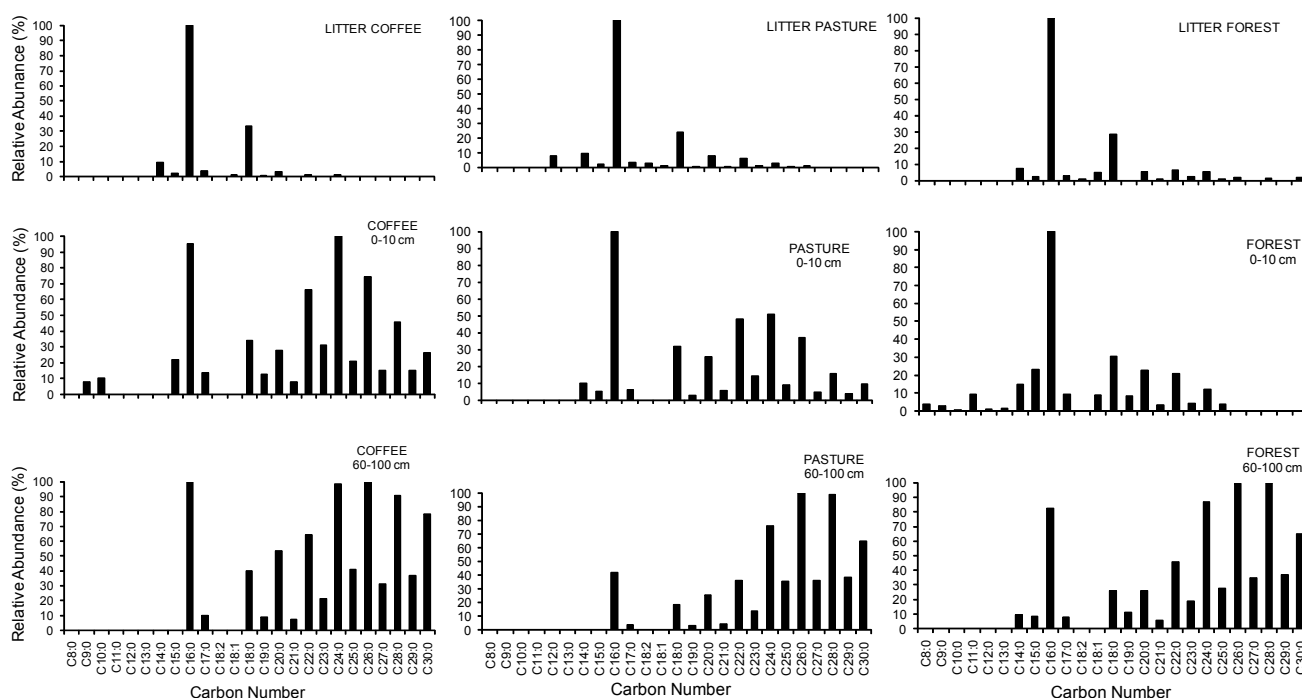


Figure 2 – Relative distributions of alkanolic acids extracted from litter and soils analysed, normalized to the most abundant compound.

Short chain (C_{15} and C_{17}) fatty acids were observed both in the litter and the surface soil layers (0-10 cm). The occurrence of short chain alkanolic acids in the 60-100 cm depth under forest suggests higher microbial activity under this land use system, the presence of these compounds being associated with biomarkers of bacterial origin (Vestal and White, 1989; Jansen et al., 2006; Jandl et al., 2007). The litter from *Coffea arabica* was characterized by higher proportions (66.36%) of saturated alkanolic acids relative to the unsaturated compounds (11.42%). The lower ratio of long-chain versus short-chain alkanolic acids with depth is explained by the selective degradation of short-chain homologues (Tan, 2003; Jandl et al., 2005). The increase in this ratio reflects differential contributions of higher plant acids (Quénéa et al., 2004). Our results demonstrate that the effects of land use in the composition of fatty acids are most noticeable in topsoil in the deeper layers. Pedogenic processes and the interaction of the lipid fraction with the mineral phase are also important in order to study the decomposition of soil organic matter along the soil profile.

The aliphatic hydrocarbons are mainly n-alkanes in the C_{14} - C_{35} range with a bimodal distribution (Figure 3). The long chain compounds in the C_{23} - C_{35} range exhibit an odd-over-even C number. This pattern distribution was previously found in soils (Amblès et al., 1989; Bull et al., 1998). The predominance of the odd-carbon-numbered long-chain n-alkanes indicates a typical contribution from higher plant waxes; the short-chain C-compounds are commonly used as bacterial biomarkers (Moucawi et al., 1981; Amblès et al., 1989; Tan, 2003).

Several authors have frequently used the alkanolic acids $C_{15:0}$ and $C_{17:0}$ as biomarkers for soil bacteria (Vestal and White, 1989; Tunlid and White, 1992; Zelles et al., 1995). In both depths of 0-10 cm and 60-100 cm, there is greater bacterial activity in the forest soil profile, as compared to tillage systems (Figure 2). This suggests that there may be differences in the microbial community composition with soil use changes.

The differences in the n-fatty acids and n-alkanes distributions in relation to soil uses (mainly at 0-10 cm) (Figures 2 and 3) indicates that variations in soil lipid composition is associated with vegetation type. The extract of forest soil samples was richer in n-alkanoic acids than in cultivated soil samples (Table 2), showing that the relative distributions of n-alkanoic acids was influenced by land use. We found a strong odd-chain signal in the relative distributions of n-alkanes from vegetation (C_{15} - C_{35}). In extractable lipids from forest and coffee litter, C_{29} and C_{31} were predominant, while C_{31} and C_{33} compounds prevailed in the pasture litter. The C_{29} , C_{31} and C_{33} distribution along the soil profile evidence a clear vegetation (litter) contribution concerning n-alkanes. In the soil under pasture, a higher contribution of the homologue C_{31} is observed, whereas in the soil under coffee the C_{29} is more abundant. The dominance of C_{31} n-alkanes observed (at 0-10 cm) can be indicative of an important contribution by (grass) roots (Naafs et al., 2004). However, in this study, it is possible that n-alkanes compounds at 60-100 cm depth in soil under pastures do not originate directly from present grass vegetation, but from previous (40-50 years ago) forest cover. Likewise, the proportion of C_{31} n-alkanes in the area under coffee culture

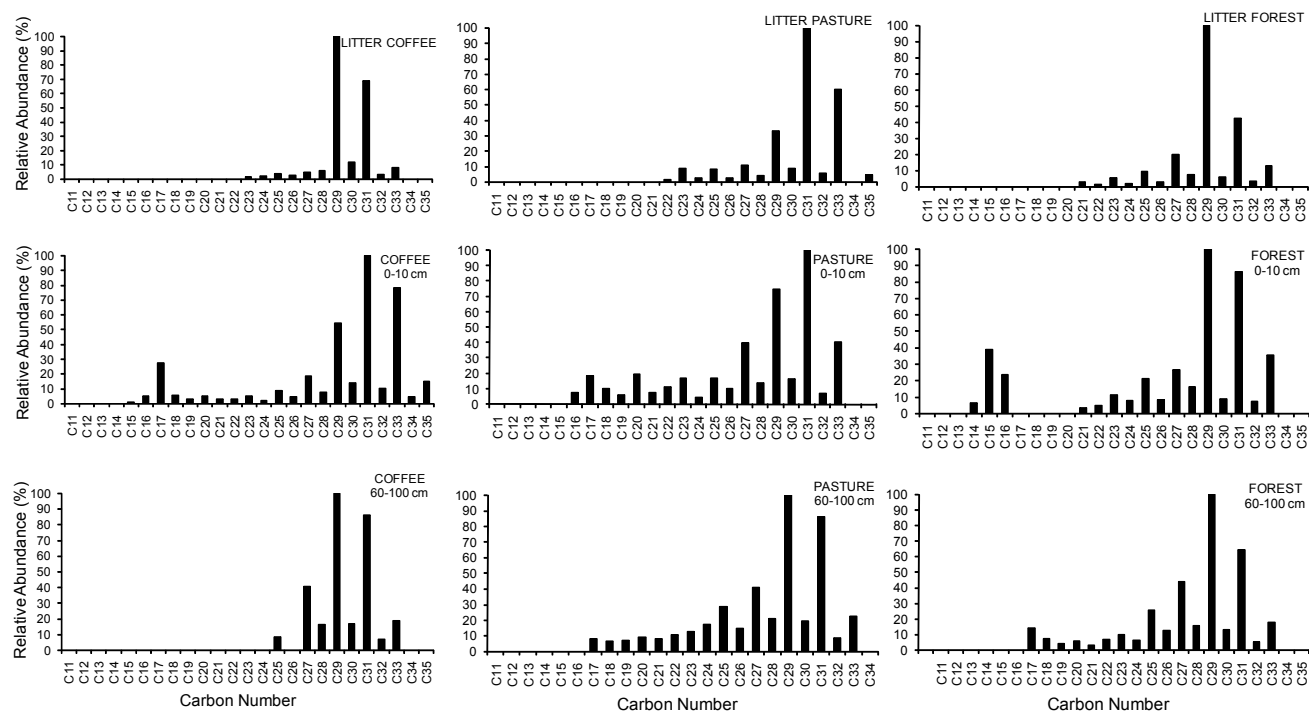


Figure 3 – Relative distributions of n-alkanes extracted from litter and soils analysed, normalized to the most abundant compound.

Table 2 – Semi-quantitative analysis of main compounds of soil and litter lipids extractable of humic Oxisol under coffee, pasture and natural forest. (Percentage of total peak area of the chromatogram and the ratios values).

Compounds	Coffee			Pasture			Forest		
	Litter	0-10 ^a	60-100 ^a	Litter	0-10 ^a	60-100 ^a	Litter	0-10	60-100
Saturated n-alkanoic acids	66.36	64.20	51.45	59.31	49.14	56.43	33.45	65.63	63.91
Unsaturated alkanolic acids	11.42	1.85	1.51	29.74	8.45	0.71	15.50	15.65	4.48
<i>n</i> -Alkanols	2.99	4.21	9.26	0.97	6.22	4.23	2.53	2.51	5.65
<i>n</i> -Alkanes	3.74	3.27	14.07	3.03	2.88	15.04	31.61	2.31	7.17
<i>n</i> -Aldehydes	3.36	1.09	1.58	-	2.80	0.88	0.40	0.06	3.27
<i>n</i> -Ketones	1.94	-	3.44	1.87	0.21	1.66	0.39	0.47	2.64
Other Compounds	10.19	27.24	18.69	5.08	30.30	21.05	16.12	13.37	12.88
Ratios Values									
<i>n</i> -Alkanolic C ₂₀₊ / C ₂₀₋	0.04	1.20	C ₂₀₊ / nd	0.07	0.10	3.40	0.12	0.37	2.30
<i>n</i> -Alkanes C ₂₀₊ / C ₂₀₋	C ₂₀₊ / nd ^b	7.20	C ₂₀₊ / nd	C ₂₀₊ / nd	4.10	10.76	C ₂₀₊ / nd	1.90	10.00
<i>n</i> -Ketones/ <i>n</i> -Alkanes	0.51	-	0.24	0.62	0.07	0.11	0.01	0.20	0.37

^aDepth in cm; ^bno detected.

shows preservation this compound, probably originated from the grassy vegetation that covered the area 12 years before its use for coffee culture. In the most conservative systems like pasture and forest, shorter chain *n*-alkanes are the better preserved at greater depth (Figure 3).

The silylated lipid fractions revealed the presence of *n*-alkanols in the samples from litter and soil, and their accumulation in depth (Table 2). These *n*-alkanols are present in epicuticular waxes and commonly observed in soil lipids (Van Bergen et al., 1997; Quénéa et al., 2004). In the case of soil under pasture, *n*-alkanols were preferentially preserved at soil surface in comparison to soil under coffee and forest. While the lipid composition of litter on pasture has lower *n*-alkanols content, it is possible that the root system contributes to the addition of these lipids in the topsoil.

The increase in free *n*-ketones at 60-100 cm may indicate changes of these compounds down the soil profile. The *n*-ketones in the soil may be a result of oxidation products of *n*-alkanes (Quénéa et al., 2004) and/or of direct input from plant material (Bai et al., 2006), but a mechanism responsible for the genesis of these compounds *in situ* in the soil has not yet been elucidated. Nevertheless, Jansen and Nierop (2009) observed that in the volcanic ash soils longer chain *n*-ketones are the primary degradation product of the corresponding *n*-alkanes. Our results showed a concomitant increase of *n*-alkanes and *n*-ketones at a depth of 60-100 cm for coffee and forest (Table 2), thus may be possible that most of the ketone compounds in these systems derive from the oxidation of alkanes.

Conclusions

The differences in lipid content and composition between the forest, pasture and coffee culture can be as-

cribed to differences in input of fresh organic materials. The lipid composition of soil and litter can be used as biomarkers in assessing changes in land use, as well as the occurrence of C stabilization mechanisms involving non humified alkylic material (waxes) in the dark soil layers of humic Oxisols at 60 cm or more in the soil profile.

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