RADIOCARBON DATING OF TOTAL SOIL ORGANIC MATTER AND HUMIN FRACTION AND ITS COMPARISON WITH ^{14}C AGES OF FOSSIL CHARCOAL

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ABSTRACT. During the last decade radiocarbon dating has been used extensively in distinct regions of Brazil to provide information about soil chronology in paleoenvironmental studies. This paper presents ^{14}C data of soil organic matter (SOM), humin fraction, and charcoal in several soil profiles under natural vegetation from different Brazil locations (north, central, and southeast regions). The main objective is to compare the obtained ^{14}C dating of total SOM with humin, the oldest fraction of SOM. In order to validate the humin ages these data are compared with the age of charcoal collected at similar depths. The ^{14}C ages obtained on charcoal were, in most of the cases, in agreement with the humin fraction considering the experimental errors, or 20% older in average. The dates obtained from total SOM showed significantly younger ages than the humin fraction indicating contamination by younger carbon. These results show the humin fraction is considered a reliable material for ^{14}C dating in soils. However, the humin fraction ages could be assumed as the minimum ages for carbon in soils.

INTRODUCTION

The radiocarbon dating of the soil organic matter (SOM) is a polemic subject, mainly due to the complexity of the soil formation. Different studies have shown the SOM is formed by different components with different ages (Campbell et al. 1967; Scharpenseel et al. 1968; Martel and Paul 1974; Goh et al. 1976; Trumbore 1996). One of the problems for ^{14}C dating of SOM is contamination by invasion of roots, infiltration of organic compositions dissolved in water, influence of microorganisms, and of the soil fauna, resulting in the rejuvenating of the obtained ages (Nowaczyk and Pazdur 1990).

One alternative material is charcoal fragments found in the soils (Soubiès 1980; Saldarriaga and West 1986; Pessenda et al. 1996, 1998; Gouveia et al. 1999a, Gouveia and Pessenda 2000). These are considered biologically inert and physically stable in relation to isotopic changes with the environment, therefore, one of the more appropriate materials for the ^{14}C dating. Even if they absorb organic compounds from another soil layers, an acid-alkaline-acid treatment is enough to remove such contamination (European Science Foundation 1985). Therefore, it is possible to have a better understanding of ages profiles in soil by analyzing fractions of SOM horizons and comparing them with fossil charcoal, naturally buried at similar depths.

This paper focuses on the comparisons of the ^{14}C dating of SOM (total soil) and humin fraction, which is probably the more stable organic compound and, theoretically, the oldest and representative of the soil age (Balesdent 1987; Balesdent and Guillet 1992; Becker-Heidmann et al. 1988). The validity of the humin dating is verified by comparisons with the ^{14}C ages of charcoal collected in similar depths in different Brazilian regions (Pará, Minas Gerais, São Paulo, and Paraná States). This study is part of a major project aiming to use carbon isotopes to reconstruct paleovegetation changes during the Late Pleistocene and Holocene in Brazil (Pessenda et al. 1996a, 1996b, 1998, 2000).

METHODS

Study Sites

Samples for the comparison between the total SOM (total soil) and the humin fraction dating were collected in Londrina (Paraná State), Jaguariúna, Piracicaba, and Botucatu (São Paulo State), Alta-

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mira (Pará State), and Humaitá (Amazonas State) (Figure 1). For the comparison between the humin and charcoal, the sampling sites were located in Salitre (Minas Gerais State), Jaguariúna, Anhembi, and Botucatu (São Paulo State). All soils were under natural vegetation (Table 1).

Table 1 Soil, vegetation types, and location of the study regions

Site (state)	Soil taxonomy (USDA)	Vegetation	Location
Londrina (PR)	Alfisol	Mesophitic Semideciduous forest	23°19′S; 51°22′W
Botucatu (SP)	Oxisol	Semideciduous forest	23°S; 48°W
Jaguariúna (SP)	Oxisol	Semideciduous forest	22°40′S; 47°1′W
Anhembi (SP)		Semideciduous forest	22°45′S; 47°58′W
Piracicaba (SP)	Oxisol	Semideciduous forest	22°41′S; 47°40′W
Salitre (MG)	Oxisol	Mesophitic Semideciduous forest	19°S, 46°46′W
Humaitá (AM)	Dystropept	Savanna/tropical forest	7°31′S, 63°2′W
Altamira (PA)	Alfisol	Tropical forest	3°30′S; 52°53′W

Figure 1 Map of Brazil showing the study sites

In Humaitá and Botucatu sites the soils were collected from two trenches separated by approximately 2 km and 1.5 km, respectively, denominated Humaitá I, Humaitá II, and Botucatu I, Botucatu II.

Sampling and Analytical Aspects

Soil samples were collected from excavations of 100 cm×200 cm×250 cm depth. Sampling involved the collection up to 10 kg of material at 10 cm intervals. Samples were dried at 60 °C to constant weight and sieved. Any remaining plant debris was removed by flotation in HCl 0.01M. The total soil was then redried to constant weight and ^{14}C analyses were carried out at the Radiocarbon Laboratory of the Centre for Nuclear Energy in Agriculture (Brazil), using the benzene method and liquid scintillation counting (Pessenda and Camargo 1991).

The humin fraction was extracted from the total soil using acid-alkaline-acid treatment (Pessenda et al. 1996a). ^{14}C analyses were carried out at the Isotrace Laboratory of the University of Toronto (Canada) employing the AMS technique.

Charcoal was collected by hand-picking from soil samples and submitted to the conventional acid-alkaline-acid treatment (Pessenda and Camargo 1991). ^{14}C analyses were carried out at the Radiocarbon Laboratory of the Centre for Nuclear Energy in Agriculture (Brazil) and the small samples were analyzed at the Isotrace Laboratory of the University of Toronto (Canada). The results are representative of the mean age of the charcoal fragments in the layers of 10 cm depth.

RESULTS AND DISCUSSION

Total Soil and Humin Fraction

The results of ^{14}C dating of the total soil and humin fraction of soils collected in six locations in different Brazilian areas are listed in Table 2. The ^{14}C age profiles for the SOM and humin fraction show the expected trend of older ages with depth (Figure 2). One exception is the age inversion in the total soil at 140–150 cm depth in Altamira. In general, the SOM ages are significantly younger than the ^{14}C ages obtained on humin samples. This pattern has also been reported in other studies (Balesdent 1987; Balesdent and Guillet 1992; Becker-Heidmann et al. 1988). The Altamira soil showed the larger age difference among the soils analyzed in this study. Regarding individual profiles, the higher age differences are observed in the shallow layers reaching values up to 209% in the Londrina soil (Table 2). This can be related to the higher concentration of carbon content (specially fulvic and humic acids) present in the soil surface, compared with the deeper parts of the profile, that contributes significantly in the rejuvenating process of the total SOM. Some soils (Botucatu, Londrina, Altamira, and Humaitá) show a trend of decreasing ages difference with depth (Figure 2) suggesting the shallow layers are more affected by the input of younger carbon than the deeper layers. The influence of younger carbon could be also related to the type of vegetation. In Humaitá I, the humin in the 90–100 cm layer was 67% older compared to the SOM. In Humaitá II, this difference was approximately 114%. Considering the distance between these profiles (2 km), this pattern can be probably related to the fact that the decomposition of biomass is significantly higher under forest (trench II) than in the area of savanna-forest transition (trench I). Therefore, a higher input of recent organic matter by means of the biological activity and percolation was incorporated to the SOM, contaminating the profile and,consequently, turning it younger.

Summarizing, in all cases the humin ages were significantly older than the total soil samples in the same depths. Probably this is related to the presence of the fulvic and humic acids, compounds of the total soil that rejuvenates the SOM. During the chemical treatment, these acids are removed and consequently the residual humin fraction becomes older.

Table 2 ^{14}C dating of the samples of total soil and humin in relation to soil depth. Values between brackets were estimated for interpolation and used for comparison effect.

Sites	Depth (cm)	^{14}C (BP)		Difference (%)[a]
		Total soil	Humin	
Londrina[b]	40–50	820 ± 60	2530 ± 70	209
	90–100	2390 ± 60	3090 ± 70	29
	170–180	9340 ± 120	10,800 ± 110	16
Jaguariúna[c]	80–90	3760 ± 80	4430 ± 90	18
	150–160	4720 ± 80	5820 ± 70	23
	200–210	5970 ± 80	7490 ± 350	25
Piracicaba [b]	70–80	2680 ± 70	3440 ± 120	28
	90–100	3030 ± 70	4260 ± 110	41
	110–120	3260 ± 70	4400 ± 120	35
Botucatu I[c]	60–70	2690 ± 60	3930 ± 80	46
	120–130	4500 ± 70	5110 ± 60	14
	180–190	5750 ± 70	6490 ± 120	13
Altamira [b]	40–50	1440 ± 70	(3970)	176
	50–60	(1870)	4570 ± 80	144
	70–80	2790 ± 80	(5950)	113
	90–100	3640 ± 90	(7270)	100
	100–110	(4230)	8100 ± 110	91
	110–120	4800 ± 80	(8590)	79
	130–140	(5650)	9810 ± 140	74
	140–150	4390 ± 90	—	—
Humaitá I[d]	90–100	3570 ± 130	5960 ± 260	67
Humaitá II[d]	90–100	2360 ± 60	5040 ± 530	114
	180–190	6130 ± 90	8170 ± 430	33

[a] Relative to the total soil
[b] Pessenda et al. 1996b
[c] Gouveia et al. 1999b
[d] Pessenda et al. 1998

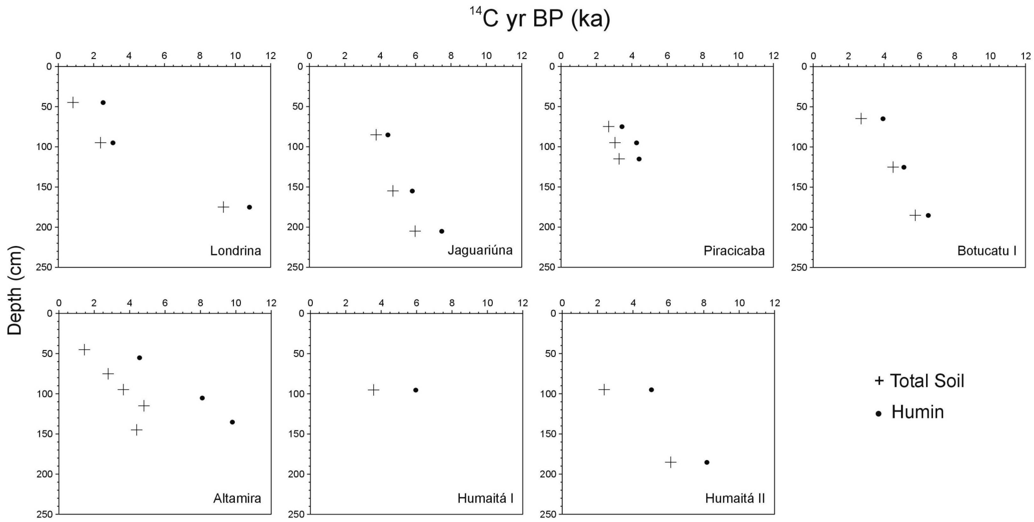

Figure 2 Comparison between total soil and humin ages

Humin Fraction and Charcoal

In order to evaluate and constrain the humin ages, these results were compared with ^{14}C ages obtained in charcoal fragments collected at the same depths. This comparison is presented in the Table 3 and Figure 3.

Table 3 ^{14}C ages of the humin and charcoal in relation to soil depth in five study sites

		^{14}C BP		
Sites	Depth (cm)	Humin	Charcoal	Difference (%)[a]
Salitre[b]	0–10	240 ± 70	160 ± 75	Concordant
	90–100	3700 ± 80	3810 ± 80	Concordant
	150–160	5550 ± 80	5930 ± 100	7
	190–200	6940 ± 80	8790 ± 100	27
Jaguariúna[c]	70–80	4770 ± 70	4800 ± 110	Concordant
	110–120	4840 ± 220	4550 ± 70	Concordant
	150–160	5820 ± 70	6330 ± 70	9
	200–210	7490 ± 350	9120 ± 90	22
Anhembi	40–50	2500 ± 60	2520 ± 60	Concordant
	60–70	2440 ± 60	2700 ± 60	11
Botucatu I	60–70	3930 ± 80	3040 ± 180	–23 (exception)
	120–130	5110 ± 60	5500 ± 70	8
	180–190	6490 ± 120	6080 ± 300	Concordant
Botucatu II	50–60	2490 ± 100	3080 ± 70	24
	90–100	3880 ± 50	4630 ± 80	19
	120–130	5010 ± 50	5660 ± 270	13
	180–190	6460 ± 70	4150 ± 450	–36

[a] Relative to the humin
[b] Pessenda et al. 1996a
[c] Gouveia et al. 1999b; Gouveia and Pessenda 2000

In general, the charcoal ages were similar and/or older than the humin ages, except for two sets of data that show younger ages for the charcoal (Table 3). The ages profiles obtained in the Salitre and Jaguariúna soils show the same pattern (Figure 3). Samples covering the first meter show similar ages for the charcoal and the humin. Then, below 150 cm the charcoal is up to 27% older than the humin. Another distinct pattern is observed in the Botucatu soil (II), that show older ages for the charcoal compared to the humin fraction excepting the deeper soil horizon that show significantly younger ages for the charcoal (Table 3). Since the sample was representative of a very small fragment (as can be observed by the high analytical error/uncertainty of ± 450), this charcoal have probably been transported from shallow layers by the soil fauna. The other Botucatu age profile shows slightly younger age for the charcoal in the shallow soil horizon and similar ages for both dated materials in the deeper soil horizons. These results show the complexities of carbon cycling in soils and it implications for the evaluation of the most suitable material for ^{14}C dating of soil carbon. However, in the absence of charcoal the humin fraction is probably the best material for ^{14}C dating but these dates should be considered as minimum ages of the SOM.

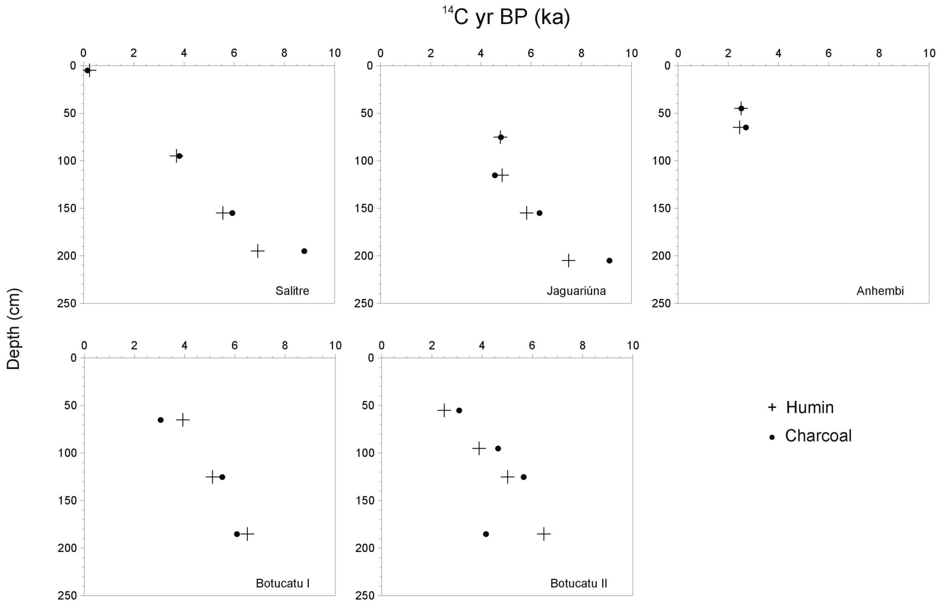

Figure 3 Comparison between the humin and charcoal ages

CONCLUSIONS

This study, performed in eight soils representative of a vast region of Brazil, clearly showed that SOM is not an appropriate material for ^{14}C dating. ^{14}C dates obtained on SOM are significantly younger than dates obtained in the humin fraction. This study also showed that the humin fraction is the more reliable material for ^{14}C dating in soil devoid of charcoal. In general, there was a good agreement of the ages between the humin fraction and buried charcoal in all the studied soils. Some soil horizons show the charcoal ages were around 20% older than the humin. A few exceptions show the opposite trend. Therefore, the ^{14}C dates obtained in the humin fraction should be considered as the minimum age of the SOM. It is recommended to date both the humin fraction and charcoal in soil studies dealing with the reconstruction of past vegetation in ecosystems composed of forest and savanna vegetation communities.

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