An isotopic study of a late Quaternary loess-paleosol sequence in SW Germany

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ABSTRACT

A late Quaternary loess-paleosol sequence at Schattenhausen (SW Germany) was studied by isotopic methods ($\delta^{13}C$, $\delta^{18}O$, ^{14}C age). The stable isotopic composition of organic carbon indicates that C3 vegetation dominated the site during most of the time of formation of the loess-paleosol sequence ($\delta^{13}C$ = -26 to -20 ‰). However, there was a short period in the Late Pleniglacial with a pronounced C4 excursion of the $\delta^{13}C$ values (-16 to -19 ‰) of organic matter. Calcified root cells ($\delta^{13}C$ = -8 to -11 ‰ and $\delta^{18}O$ = ca. -6.5‰) are isotopically different from diffuse carbonate in the loess and soil matrix ($\delta^{13}C$ = -1 to -2.5 ‰; $\delta^{18}O$ = -6 to -7.5 ‰). The stable isotopic composition of carbon and oxygen of the calcified root cells in combination with their ^{14}C ages suggest that they formed, at least partially, in the Holocene.

Key words: loess-paleosol sequence, pedogenic carbonate, stable isotope, radiocarbon age, late Quaternary, SW Germany.

RESUMEN

Se estudió una secuencia cuaternaria de loess-paleosuelos en Schattenhausen (SW de Alemania) por métodos isotópicos ($\delta^{13}C$, $\delta^{18}O$, edad ^{14}C). La composición de isótopos estables de carbón orgánico indican que vegetación de tipo C3 dominó el sitio durante la mayor parte del tiempo de formación de la secuencia ($\delta^{13}C$ = -26 a -20 ‰). Sin embargo, se registró un periodo en el Pleniglacial Tardío con una oscilación pronunciada hacia C4 de los valores $\delta^{13}C$ (-16 to -19 ‰) de la materia orgánica. Las células de raíces calcificadas ($\delta^{13}C$ = -8 a -11 ‰; $\delta^{18}O$ = -6.5 ‰) son isotópicamente diferentes de los carbonatos difundidos en la matriz del loess y del suelo ($\delta^{13}C$ = -1 a -2.5 ‰; $\delta^{18}O$ = -6 a -7.5 ‰). La composición de isótopos estables de carbono y oxígeno en las células de raíces calcificadas, en combinación con sus edades de ^{14}C, sugieren que fueron formadas, al menos parcialmente, en el Holoceno.

Palabras clave: secuencias loess-paleosuelos, carbonatos pedogenéticos, isótopos estables, edad por radiocarbono, Cuaternario tardío, SW de Alemania.

INTRODUCTION

Over the last decade a number of isotopic studies on loess deposits have been conducted (Frakes and Jianzhong, 1994; Wang *et al.*, 1997, 2000; Wang and Follmer, 1998; Hatté *et al.*, 1998, 1999; Muhs *et al.*, 1999; Johnson and Willey, 2000). This research concentrated on two main lines of evidence. The first one is the stable isotopic composition of organic carbon in loess which is interpreted in terms of the proportion of local biomass using the C4 photosythetic pathway at the time of loess deposition (Wang *et al.*, 1997; Muhs *et al.*, 1999; Johnson and Willey, 2000) or specific responses of C3 vegetation to late Quaternary climate changes (Hatté *et al.*, 1998, 1999). The second line is the stable isotopic composition of carbon and/or oxygen of pedogenic (or secondary) carbonate (Frakes and Jianzhong, 1994; Wang *et al.*, 1997, 2000; Wang and Follmer, 1998).

Although these studies demonstrated a high potential of stable isotopes as paleoclimatic proxies, several problems still remain. First, the isotopic evidence obtained from loess sections is still very limited. Second, researchers often focus on only one of the carbon pools, *i.e.*, either organic matter (Hatté *et al.*, 1998, 1999; Muhs *et al.*, 1999; Johnson and Willey, 2000) or carbonate (Frakes and Jianzhong, 1994). Third, absolute dating methods either are used on a small scale, or fail. The latter problem is especially relevant for studies on secondary carbonate that can be formed much later than the assumed time of a soil formation episode, subjected to recrystallization, or both.

These investigations were carried out in the abandoned loess pit south of Heidelberg, close to the Rhine Valley (SW Germany) by a combination of stable isotopic measurements on organic carbon ($\delta^{13}C$) and pedogenic carbonate in form of calcified root cells ($\delta^{13}C$ and $\delta^{18}O$) and ^{14}C content on several types of carbonate (calcified root cells, mollusc shells, earthworm cacite) are presented.

MATERIALS AND METHODS

The loess sequence Schattenhausen is situated between Maisbach and Nussloch south of Heidelberg in southwest Germany (49°21'N, 8°43'E). During the glacial stages, the area experienced periglacial conditions characteristic of Central Europe. Thick deposits of Quaternary calcareous loess are widespread, derived from the nearby Rhine Valley. Loess with intervening palaeosols are characteristic of the whole region, and a general pedostratigraphy has been developed by Schönhals *et al.* (1964), Semmel (1967, 1968, 1996), and Bibus (1974). Specifically, a number of recent publications are related to parts of the Neckar Valley and the Rhine-Main-region, which are close to the study site (Bibus, 1989, 1996; Frechen, 1999; Zöller and Löscher, 1999). The study site of Schattenhausen has been described in detail in Terhorst *et al.* (2001). Loess sediments belonging to the Penultimate Glacial Period form the base of the studied

sequence. These eolian sediments are overlain by a well developed Eemian Bt horizon, which has been truncated in its upper parts. On its top, an interstadial pedocomplex occurs in the form of a chernozem-like soil, which is characteristic for the Early Pleniglacial Interstadials in Middle Europe. The pedocomplex is covered by the B horizon of an Arctic Brown soil belonging to the Middle Pleniglacial part of the sequence. It is overlain by loess deposits of the Upper Pleniglacial with intercalated horizons of weak Tundra Gleys.

For laboratory analyses, samples from 19 units of the sequence were taken. $\delta^{13}C$-values were determined for each unit. $\delta^{13}C$ and $\delta^{18}O$ on secondary carbonate (calcified root cells) and diffuse carbonate in loess/soil matrix were determined for units 3, 7, 9 and 12. $\delta^{13}C$ and $\delta^{18}O$ of earthworm calcite were determined for unit 9. For the analysis of $\delta^{13}C$ in soil organic carbon, samples were previously rinsed (removal of detritus, rootlets, etc.), dried, and pretreated with 3% HCl to remove carbonate. Samples for the determination of $\delta^{13}C$ and $\delta^{18}O$ in carbonates were rinsed to isolate secondary carbonate accumulations (mostly calcified root cells), dried, and pretreated overnight with 2.5% NaOCl to remove organic carbon. The stable carbon and oxigen isotope ratios were detrmined by mass spectrometry and are reported as $\delta = (R_{sample}/R_{PDB} -1) \times 1000$, where R is $^{13}C/^{12}C$ or $^{18}O/^{16}O$ and PDB is the Pee Dee belemnite standard.

Five carbonate samples were dated by the radiocarbon method: shell fragments (unit 7), earthworm calcite (unit 7) and 3 samples of calcified root cells (2 from unit 3 and 1 from unit 7). Samples of shell fragments and earthworm calcite from unit 7 selected for ^{14}C dating were thoroughly brushed out from matrix with control under binocular microscope without rinsing to avoid ^{14}C contamination. Samples of calcified root cells from units 3 and 7 were prepared for radiocarbon measurements in the same way. To test the preservation of carbonate in the process of rinsing (both for stable isotopes and for ^{14}C), a half of the sample of root cell carbonate from unit 3 was rinsed and dried prior to ^{14}C dating. The ^{14}C measurements (AMS) were performed at the R.J. Van de Graaf Laboratory (University of Utrecht). The ^{14}C data are presented as uncalibrated and normalized to $\delta^{13}C = -25‰$ (1σ-probability).

RESULTS AND DISCUSSION

$\delta^{13}C$ values of organic matter

Isotopic composition of soil organic matter is determined by that of the vegetation *in situ* (Goh *et al.*, 1976; Dzurec *et al.*, 1985; Desjardins *et al.*, 1994; Nordt *et al.*, 1994; Fredlund and Tieszen, 1997). For this reason, $^{13}C/^{12}C$ ratios of organic matter in loess sediments have been used as paleoenvironmental indicators (Wang *et al.*, 1997; Hatté *et al.*, 1998, 1999; Muhs *et al.*, 1999; Johnson and Willey,

Table 1. $\delta^{13}C$ of soil organic carbon, $\delta^{13}C$ and $\delta^{18}O$ values and ^{14}C age of carbonate from the loess-paleosol sequence at Schattenhausen.

Unit	Depth (m)	Stratigraphic position	Characteristic of unit	Sample	$\delta^{13}C_{org}$ (‰)	$\delta^{13}C_{carb}$ (‰)	$\delta^{18}O_{carb}$ (‰)	^{14}C age (yr BP)	Laboratory number of ^{14}C analysis
1	0.2	Würm	Loess	Matrix	-25.37				
2	0.3	Würm	Loess	Matrix	-23.33				
3	0.6	Würm	Loess	Matrix	-24.16	-2.39	-7.48		
	0.6	Würm	Loess	Calcified root cells[1]		-8.23	-6.32	8,830±50	UtC-10624[3]
	0.6	Würm	Loess	Calcified root cells[1]				9,024±45	UtC-10625[4]
4	1.5	Würm	Loess	Matrix	-24.61				
5	1.7	Würm	Tundra gley	Matrix	-16.72				
6	2.6	Würm	Tundra gley	Matrix	-18.21				
7	3.0	Würm	Tundra gley	Matrix	-19.86	-1.01	-6.66		
	3.0	Würm	Tundra gley	Calcified root cells[1]		-8.70	-6.25	6,117±45	UtC-10627
	3.0	Würm	Tundra gley	Calcified root cells[2]		-9.61	-6.28		
	3.0	Würm	Tundra gley	Shell fragments		-7.84	-2.49	23,990±160	UtC-10626
	3.0	Würm	Tundra gley	Earthworm calcite		-12.9		24,510±190	UtC-10628
8	3.9	Würm	Loess	Matrix	-23.17				
9	4.2	Würm	Tundra gley	Matrix	-24.23	-1.08	-5.86		
	4.2	Würm	Tundra gley	Calcified root cells[1]		-10.24	-6.43		
	4.2	Würm	Tundra gley	Calcified root cells[2]		-10.72	-6.66		
	4.2	Würm	Tundra gley	Earthworm calcite		-12.81	-4.98		
10	4.5	Würm	Arctic brown soil	Matrix	-24.73				
11	4.9	Würm	Degraded chernozem	Matrix	-24.04				
12	6.0	Riss	Loess	Matrix	-24.53	-2.67	-7.47		
	6.0	Riss	Loess	Calcified root cells[1]		-9.03	-6.08		
	6.0	Riss	Loess	Calcified root cells[2]		-9.08	-6.18		
13	6.5	Riss	Loess	Matrix	-24.43				
14	7.9	Riss	Loess	Matrix	-24.01				
15	8.2	Riss	Brown soil (weakly dev.)	Matrix	-23.96				
16	8.5	Riss	Loess	Matrix	-24.78				
17	9.0	Riss	Loess	Matrix	-24.62				
18	9.3	Riss	'Wet' soil	Matrix	-24.91				
19	9.6	Riss	'Wet' soil	Matrix	-25.69				

[1] Relatively dense isolated fragments, mostly 0.5–1 mm in diameter and 3–15 mm in lenght.

[2] Porous calcified tissues of irregular form and mostly 5–30 mm in lenght.

[3] Brushed out.

[4] Rinsed.

2000). The $\delta^{13}C$ values of organic matter in the loess-paleosol sequence fall within the range from -25.69 to -16.72 (Table 1, Figure 1). Although most of the values demonstrate a C3 origin of organic matter, there are several layers with $^{13}C/^{12}C$ values suggesting that organic carbon is derived from a mainly C4 vegetation (units 5–7). It is important to note that all ^{13}C measurements on middle European loess-paleosol sequences found only organic carbon of C3 origin until now (Hatté *et al.*, 1998, 1999). One of such sequences (Nussloch) is situated less than 1 km to the west from the studied profile. The presence of C4-carbon enriched layers in the Upper

Pleniglacial deposits at Schattenhausen suggests that the vegetation cover of the late Pleistocene in the eastern Rhine Valley region had a complex structure. At least in places, plant communities could have been periodically dominated by C4 species.

In the present-day world, the C4 photosynthetic pathway is largely characteristic of warm to moderate and arid to semi-arid climates and is not found in periglacial environments (Teeri and Stowe, 1976; Bird *et al.*, 1994; Lloyd and Farquhar, 1994; Cerling *et al.*, 1998; Sage, 2001). However, it is supposed that C4 species could have had a

competitive advantage over C3 species even at low temperatures as a result of low CO_2 levels during the Last Glacial Maximum (Cerling *et al.*, 1998). The composition of stable carbon isotopes in the loess-paleosol sequence at Schattenhausen as well as some other recent data on North American loess deposits (Johnson and Willey, 2000) seems to support this hypothesis. Further isotopic investigations are needed to understand the real abundance of traces of C4 vegetation in loess sediments.

$\delta^{13}C$ and $\delta^{18}O$ values of calcified root cells

Pedogenic carbonate forms mostly in carbon isotopic equilibrium with CO_2 of soil air, whereas the stable oxygen isotopic composition of pedogenic carbonate is determined mainly by the $^{18}O/^{16}O$ ratio of soil water (Cerling, 1984, 1991; Cerling *et al.*, 1989). In several works, the $\delta^{13}C$ and $\delta^{18}O$ values of pedogenic carbonate were applied as paleoenvironmental indicators to loess sediments with buried soils (Frakes and Jianzhong, 1994; Wang *et al.*, 1997, 2000). The $\delta^{13}C$ values of all samples of pedogenic carbonate in this study indicates that carbonate formed in equilibrium with a C3-derived carbon dioxide (Table 1, Figure 1). As pointed out below, ^{14}C data demonstrate that there has been no recrystallization of carbonate in the process of rinsing. A combination of the $\delta^{13}C$ and $\delta^{18}O$ values (Table 1, Figure 1) indicates that carbonate crystallized under moderate, mild climatic conditions rather than in periglacial environments (Cerling, 1984).

For comparison, the stable carbon and oxygen isotopic composition of diffuse carbonate in the matrix of loess and in soil layers was determined. For most of the samples, the isotopic composition of pedogenic carbonate is markedly different from that of diffuse carbonate (Table 1, Figure 1),

indicating different origins for both carbonate forms. Since the diffuse form has no distinct morphological features (in contrast to pedogenic one considered above), it may be constituted by a cryptocrystalline mixture of pedogenic as well as lithogenic carbonate minerals. Its relatively high $\delta^{13}C$ values and low $\delta^{18}O$ values can be explained either by formation under cold climatic conditions (Cerling, 1984) or by a substantial admixture of lithogenic calcite. Earthworm calcite has stable isotopic values differing from both those of calcified root cells and those of diffuse carbonate (Table 1, Figure 1).

^{14}C-dating of carbonate samples

Shell fragments and earthworm calcite show late Pleistocene ^{14}C ages, whereas dates on the calcified root cells fall into the Holocene (Table 1, Figure 1). There is no significant difference between two radiocarbon ages of calcified root cells from unit 3 (Table 1, Figure 1), which implies that no carbonate recrystallization occurs in the course of rinsing.

Shells of terrestrial molluscs can yield radiocarbon ages which are up to 2,000 years too old (Evin *et al.*, 1980). Even if such an effect took place during the sedimentation of the loess of the profile at Schattenhausen, the ^{14}C age of shell fragments correlates well with the stratigraphical position of the sample (Upper Pleniglacial) (Table 1, Figure 1). This age indicates also that the sample experienced no intensive diagenetic ^{14}C contamination. The radiocarbon age of earthworm calcite is in very good agreement with the age of shell carbonate (Table 1, Figure 1), and confirms the absence of substantial radiocarbon contamination in the given horizon of the profile.

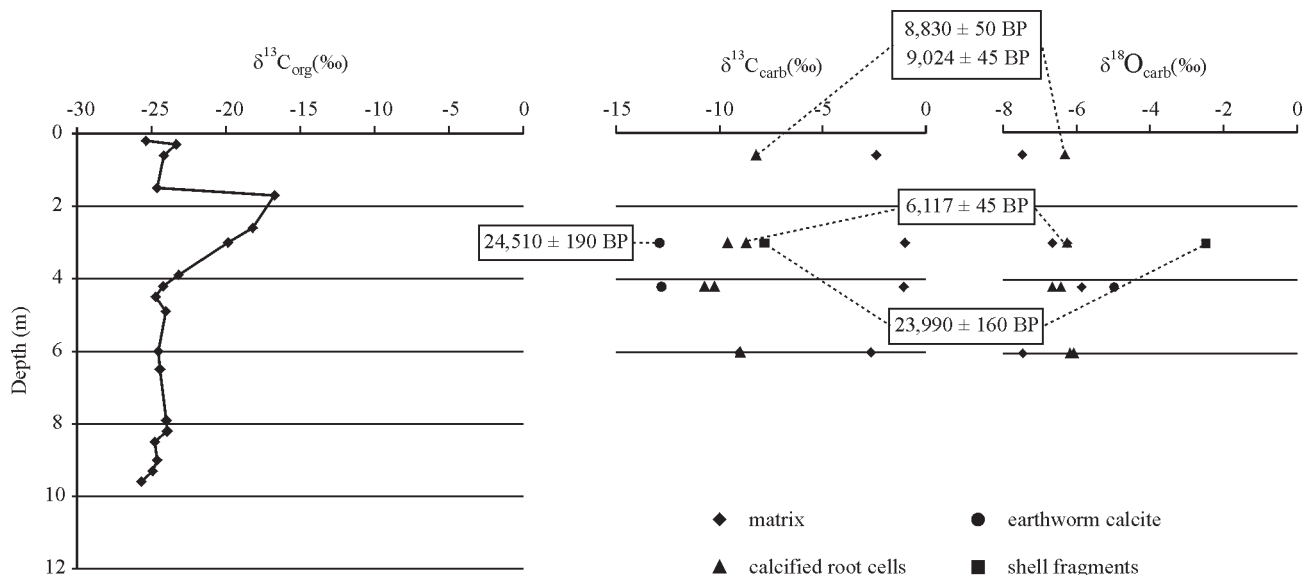For most soils, radiocarbon content in pedogenic

Figure 1. $\delta^{13}C$ of soil organic matter, $\delta^{13}C$ and $\delta^{18}O$ values and ^{14}C age of carbonate from the loess-paleosol sequence at Schattenhausen.

carbonate at the moment of crystallization corresponds to that of soil CO_2, which implies that the radiocarbon age of pedogenic carbonate can be interpreted in terms of the real age (Amundson *et al.*, 1994; Wang *et al.*, 1994, 1996). The so-called "limestone dilution effect" in pedogenic carbonate (Williams and Polach, 1971; Chen and Polach, 1986) is presumably rather limited (Amundson *et al.*, 1994; Wang *et al.*, 1994, 1996). Post-segregational recrystallization can result in ^{14}C ages of pedogenic carbonate that are too young (Chen and Polach, 1986; Amundson *et al.*, 1994).

The radiocarbon ages of calcified root cells in the loess-paleosol sequence are younger than late Pleistocene. These ^{14}C ages can hardly be explained by diagenetic radiocarbon contamination. As mentioned above, there has been no intensive alteration of carbonate at least in unit 7. Data of other researchers suggest that loess deposits can be closed systems with respect to radiocarbon contamination of carbonate material (Evin *et al.*, 1980; Zhou *et al.*, 1999). Calcified root cells formed, therefore, at least in unit 7 in the Holocene. Further work is needed to explore to what extent the calcified root cells in unit 3 were subject to diagenetic alteration.

Radiocarbon ages of carbonate materials, together with stable isotopic data, suggest that the calcified root cells in the loess-paleosol sequence at Schattenhausen formed, at least in part, in the Holocene. The mechanisms of their formation are not known at the moment. Accumulation of carbonate could have proceeded in some way in cells of extinct deep roots of the Holocene vegetation. It is important to stress the significance of this finding for interpreting the pedogenetic features of loess deposits. Previous works supposed calcified root cells to have formed almost synchronously with loess sedimentation. In a micromorphological study of small-scale secondary carbonate accumulations in the European loess belt, Becze-Deak *et al.* (1997) assumed that the formation of calcified root cells is related with the period of loess deposition. Wang and Follmer (1998) explained deviations from the diffusion-production model of T.Cerling in secondary carbonate from a loess-paleosol sequence on the Chinese Loess Plateau by seasonality effects of the Asian monsoons during the last intergalcial-glacial cycle. In doing so, they suggested the age of pedogenic carbonate to be identical with the age of the deposition of the surrounding matrix. A similar view of the phenomenon was the basis for a reconstruction of the paleo-El Niño – Southern Oscillation cycles on a loess-paleosol sequence in the central United States (Wang *et al.*, 2000). Our data demonstrate that the nature of the processes of carbonate accumulation in loess-paleosol sequences is probably more complex than previously suggested. Paleoenvironmental interpretations of the stable carbon isotopic composition of secondary carbonate from loess deposits should be made with more caution. A wider use of direct comparative dating of pedogenic carbonate and other forms of carbonate in loesss-paleosol sequences might approach the solution of the problem.

CONCLUSIONS

An isotopic study of the loess-paleosol sequence at Schattenhausen, close to Heidelberg, gives some new insights into the paleoenvironmental record of late Quaternary loess sediments.

Stable isotope composition of organic carbon in the loess-paleosol sequence demonstrates that a local dominance of C4 vegetation within certain periods of the Upper Pleniglacial in middle Europe is possible. Although most of $^{13}C/^{12}C$ ratios of organic matter fall within the range from -26 to -20‰, there are several layers with $\delta^{13}C$-values values between -16 and -19‰.

Calcified root cells in the loess-paleosol sequence at Schattenhausen formed, at least partially, in the Holocene. $\delta^{13}C$ and $\delta^{18}O$ values of this form of secondary carbonate indicate moderate climatic conditions of this form of secondary carbonate and are definitely different from those characteristic for periglacial environments. Radiocarbon dating indicates early to mid-Holocene ages of the calcified root cells, which cannot be properly explained by recrystallization of calcite. This finding should be taken into account in interpreting pedogenetic features of the loess deposits.

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