

Diagenetic alteration of artificial lime mortar in a Mediterranean soil: ^{14}C and stable carbon isotopic data

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Abstract

Recent research demonstrated a high potential of pedogenic carbonate for paleoenvironmental studies and dating of soils. However, when interpreting radiometric age or stable isotopic composition of pedogenic carbonate, diagenetic alteration of carbonate may present a major problem. Until recently, this process remains comparatively poorly understood. Artificial lime mortars occur in some soils developed in cultural layers and may serve as indicators of diagenetic alteration of carbonate material in soil. The mortar carbonate consist mainly of two initial fractions: the carbonate cement including carbon of the atmospheric CO_2 and residual limestone pieces, which originate from incomplete burning of limestone. Artificial mortar of one generation, deriving from a more or less uniform mixture, has an approximately uniform composition of radioactive and stable isotopes prior to diagenetic alteration in soil. During pedogenesis, different intensity of weathering of carbonate material at different depths should cause a differentiation of isotopic content of lime mortar in a soil profile.

In this work, results of an isotopic study of an artificial lime mortar that has been exposed to weathering in soil within about 1,200 years under Mediterranean climatic conditions are presented. The lime mortar studied covered the walls of the palace at the site Balis (North-Eastern Syria) between about the beginning of the 8th century and the early 9th century A.D. The mortar layers retained their vertical orientation after the downfall of the building and were in form of continuous plates exposed to soil weathering. Soil development is assumed to begin shortly after the end of the occupation phase (c. the early years of the 9th century A.D.). Samples of mortar were taken at 10 cm intervals from two soil profiles (regosols) to a depth of 100 cm. ^{14}C activity and stable carbon isotope ratios were determined. Radiocarbon activity increases from 8-11% at 50-100 cm depth to 17% at 10-20 cm depth. Calculated proportion of diagenetically recrystallized carbonate at 10-20 cm below the soil surface ranges approximately between 10 and 20%. At 50-100 cm depth not more than 10% of mortar carbonate are diagenetically altered. By contrast, stable isotopic composition of carbon in mortar shows no regular vertical trends.

The results suggest that soil development under Mediterranean climatic conditions with a duration of no more than 1200 years might be quite sufficient to cause a diagenetic contamination of carbonate material by environmental ^{14}C at least in upper soil horizons.

Keywords: carbonate, artificial lime mortar, diagenesis in soil, radiocarbon, stable carbon isotopes, Mediterranean

Introduction

Over the last two decades a number of works demonstrated a high potential of pedogenic carbonate for palaeoenvironmental reconstructions (Cerling, 1984, 1991, 1992; Cerling *et al.*, 1989; Amundson *et al.*, 1989; Mora *et al.*, 1991, 1996; Humphrey and Ferring, 1994; Courty *et al.*, 1994; Wang *et al.*, 1993, 1996, 1997; Pustovoytov, 1998; Lee and Hisada, 1999) and geochronology (Ku *et al.*, 1979; Chen and Polach, 1986; Sowers *et al.*, 1988; Amundson *et al.*, 1989, 1994; Vincent *et al.*, 1994; Pendall *et al.*, 1994). For late Quaternary soils the radiocarbon method can be applied effectively to date pedogenic carbonate and/or stages of soil development (Williams and Polach, 1971; Bowler and Polach, 1971; Magaritz *et al.*, 1981; Chen and Polach, 1986; Amundson *et al.*, 1994; Pendall *et al.*, 1994; Wang *et al.*, 1996; Monger *et al.*, 1998).

Interpretation of radiocarbon ages of secondary carbonate accumulations in soils are complicated by two major problems: (1) the "limestone-dilution effect" and (2) diagenetic contamination of carbonate by environmental ^{14}C (Williams and Polach, 1970; Bowler and Polach, 1971; Chen and Polach, 1986; Amundson *et al.*, 1994; Pendall *et al.*, 1994; Wang *et al.*, 1996). The first is that the radiometrically "dead" inorganic carbon from parent substrates may be involved into formation of secondary carbonate, rendering ^{14}C ages too old (Williams and Polach, 1970; Bowler and Polach, 1971; Chen and Polach, 1986). Recent studies suggest, however, that influence of this process is, if at all, rather limited, because formation of pedogenic carbonate occurs in isotopic equilibrium with the CO_2 of soil air (Wang *et al.*, 1993, 1996; Amundson *et al.*, 1994; Pendall *et al.*, 1994).

Less is known about the diagenetic contamination of carbonate in soils by environmental ^{14}C . Bowler and Polach (1971) found that the difference between the radiocarbon age of pedogenic carbonates in buried soils and the geomorphologically determined age of these buried soils increased with increasing humidity of climate in Australia. This suggests that pedogenic carbonate were not entirely closed systems. Pendall *et al.* (1994) concluded that carbonate in desert soils of Nevada are recrystallized only within the upper 90 cm below the soil surface. Amundson *et al.* (1994) pointed to extremely low ^{14}C concentrations in calcareous laminations on gravel bottoms in soils in early and mid-Pleistocene parent materials, which indicates that pedogenic carbonate can be radiometrically closed systems. However, systematic investigation of diagenetic alteration of carbonate in calcareous soils of different geographical zones were not conducted until now. One of the principal methodological difficulties in studies of radiocarbon contamination of carbonate in soils is a deficit of information about the age of carbonate itself. For this reason, ^{14}C measurements on carbonate samples with independently estimated absolute ages in soils are essential for understanding of the diagenetic processes.

In this work, we present results of an isotopic study of an artificial lime mortar that has been exposed to weathering in soil within about 1,200 years under Mediterranean climatic conditions. The mortar carbonate consists initially of two main fractions: carbonate cement including carbon of the atmospheric CO_2 and residual limestone pieces, which originate from incomplete burning of limestone (Baxter and Walton,

1970; Kedar and Mook, 1978; Folk and Valastro, 1985; Zouridakis *et al.*, 1987). Lime mortar of one generation, deriving from a more or less uniform mixture applied over walls of a room, has an approximately uniform composition of radioactive and stable isotopes prior to diagenetic alteration in a soil. During pedogenesis, different intensity of weathering of carbonate material at different depths should cause a differentiation of isotopic content of lime mortar in a soil profile. Vertical distribution of radiocarbon ages and stable carbon isotopes of artificial mortar may reflect, therefore, patterns of susceptibility of carbonate to diagenesis in soils.

Materials and Methods

The lime mortar studied has been collected at the site Balis, North-Eastern Syria. The site is situated at the western edge of the Euphrates valley. The geological structure of the territory includes three major elements-Cretaceous limestone, Miocene conglomerates and Pliocene-Pleistocene alluvial fans (Wirth, 1971). The climate of the area is Mediterranean with a mean annual temperature of 17.7 °C and a mean annual precipitation of 358 mm (Alex, 1985). The vegetation in the region is presented by Mesopotamian steppes of the *Artemisietea herbae-albae mesopotamica* (Zohary, 1973).

The palace formed a structure of approximately 77 m x 77 m with square towers at its corners. Its curtain walls were made of two parallel rows of large limestone blocks that were carefully dressed only on their outsides. The intermediary space was filled with rubble laid in mud. It appears that the walls were built directly on the hard clay surface without using foundation trenches. The fact that the limestone wall's elevation was more or less equal throughout the building and no larger quantities of dressed blocks were found out of context, suggest that it functioned as the lower level for walls that once rose on top of it but have not been preserved. The assumption that this superstructure consisted of a mudbrick wall is confirmed by the find of fallen mudbricks that were discovered singly and in clusters on both side of the foundation. Both, the limestone foundation and the rising mudbrick walls were plastered on both sides. A series of rooms were excavated on all four sides of the palace. Some were residential and contained remnants of frescoes, mosaics and carved stucco panels while six smaller, elevated rooms on the northern and southern side had concrete floors.

From textual sources, the origin of the palace can be linked with the activities of a prominent member of the Umayyad family, the first Islamic dynasty in this area during the beginning of the 8th century A.D. The palace did presumably not function as such more than fifty years, i.e. until the downfall of the Umayyad dynasty in 750. A.D. Some secondary installations and alterations must originate from squatters who moved in after this event. Evidence for the approximate latest use of its space is provided by two coins dated to the early years of the 9th century. The pristine condition of some frescoes and plaster facings, now sealed under the sediment of collapsed mudbrick walls suggests that the process of deterioration proceeded rapidly after the place was abandoned (Finkbeiner and Leisten, 2000).

Although the history of pedogenesis at the site is not exactly known, two general considerations should be taken into account. First, it is obvious that the duration of soil development cannot exceed c. 1,200 years. Second, geomorphological position of the site suggests that substantial erosion (both natural and anthropogenic (Wirth, 1971)) of the ruin surface did not take place after its stabilisation and the soils of the site are likely to be intact. Soil development is assumed to have begun shortly after the end of the

occupation phase, i.e. in the second half of the 8th century A.D. Soil profiles are weakly developed and are accounted to as regosols (FAO, 1990).

The mortar layers retained their vertical orientation on the rest of the walls after the downfall of the building and stabilization of the ruin surface and were exposed to soil weathering in form of continuous plates enclosed into homogeneous loamy substrate. Samples were taken from the mortar plate vertical edges (Figure 1), which are presumably more subjected to soil weathering and are therefore better indicators of diagenetic alteration of carbonate than the rest of the plates. Sampling was carried out at 10 cm intervals in two soil profiles: from 0-90 cm of the soil surface in profile 1 and from 20-100 cm in profile 2 (Figure 1). Relative ¹⁴C specific activity (in the subsequent text referred to as radiocarbon or ¹⁴C activity) of 15 samples (7 from profile 1 and 8 from profile 2) was determined by the liquid scintillation procedure (Radiocarbon Laboratory, Institute of Geography of the Russian Academy of Sciences, Moscow) and is expressed as a percentage of a modern standard (95% of NBS oxalic acid). Furthermore, stable carbon isotope ratios in mortar were measured (Institute of Geology, University of Erlangen). The results are presented as relative deviations in % from the isotopic ratio in the PDB standard (Craig, 1957).

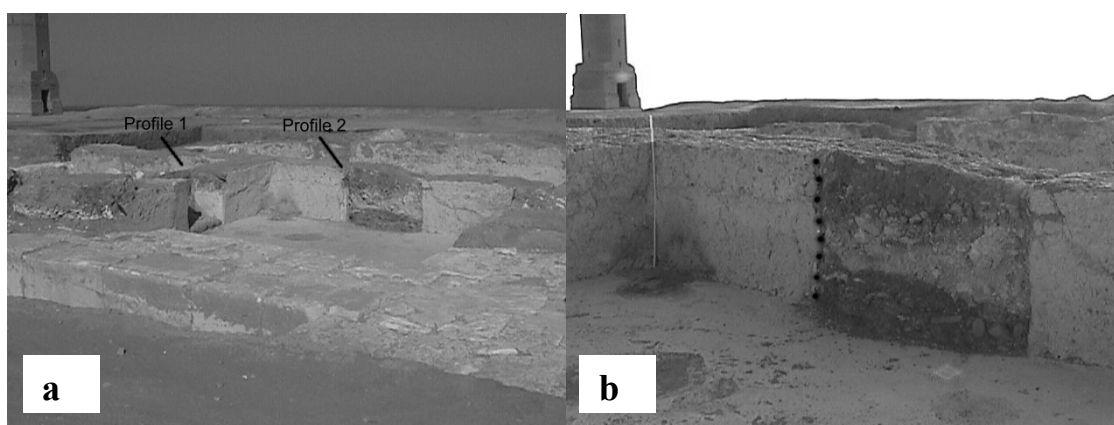


Figure 1 Field views of artificial mortars in Brasil: a) general view of the excavation with the location of profiles 1 and 2; b) profile 2, black points indicate positions of samples.

Results and Discussion

Radiocarbon content in the mortar

¹⁴C content in the mortar samples is relatively low, which suggests a high proportion of primary limestone fraction in all samples (Table 1, Figure 2). Radiocarbon activity definitely increases from bottom (8-11%) to top (17%) of the soil profiles (Figure 2). Since the initial distribution of radiocarbon in the mortar (prior to downfall of the palace and the beginning of soil development) was presumably more or less uniform in space, the vertical distribution of measured ¹⁴C activity can be only explained as a result of pedogenesis, namely, a more intensive dissolution-precipitation of calcium carbonate in upper soil horizons. This finding suggests that soil development under Mediterranean climatic conditions with a duration of no more than 1,200 years might be quite sufficient to cause a diagenetic contamination of carbonate material by environmental ¹⁴C at least in upper soil horizons.

Table 1 ¹⁴C activity, ¹⁴C ages and δ¹³C of artificial mortar from Balis.

Profile	Depth, cm	Sample	¹⁴ C-Lab. index (IGAS-)	¹⁴ C activity, %	Calculated ¹⁴ C- age (uncalibrated), years BP	δ ¹³ C, ‰
1	0-10	bulk mortar				-0.51
1	10-20	- „ -	2216	17.11±0.82	14090±430	-0.43
1	20-30	- „ -				0.88
1	30-40	- „ -	2153	9.91±0.48	18710±410	-0.6
1	40-50	- „ -	2143	13.91±0.72	15850±300	-0.23
1	50-60	- „ -	2142	10.55±0.50	18070±240	-0.5
1	60-70	- „ -	2144	8.76±0.37	19570±340	-0.73
1	70-80	- „ -	2145	9.14±0.36	19230±400	-0.49
1	80-90	- „ -	2140	9.19±0.69	19290±600	-0.62
1	20-30	limestone				-0.78
1	60-70	bulk mortar				-0.64
1	60-70	- „ -				-0.61
1	60-70	- „ -				-0.65
2	20-30	bulk mortar	2224	10.68±0.43	17970±810	-0.7
2	30-40	- „ -	2223	13.49±0.48	16130±620	-0.92
2	40-50	- „ -	2222	9.67±0.52	18780±410	-0.55
2	50-60	- „ -	2221	9.83±0.57	18670±420	-0.45
2	60-70	- „ -	2220	8.96±0.53	19390±1030	-0.41
2	70-80	- „ -	2219	8.41±0.41	19910±450	-0.45
2	80-90	- „ -	2218	9.97±0.44	18530±680	-0.63
2	90-100	- „ -	2217	11.18±0.60	17640±720	-0.56
2	70-80	limestone				0.15

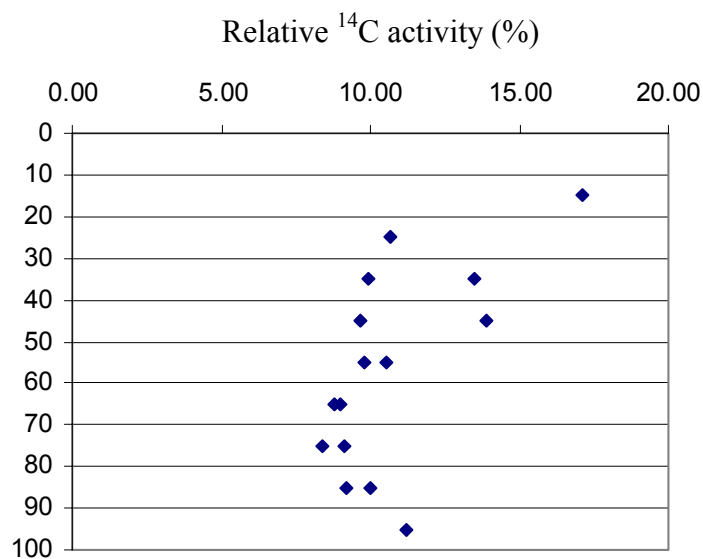


Figure 2 Vertical distribution of relative ¹⁴C specific activity in profiles 1 and 2.

It is notable that Pendall *et al.* (1994) concluded that an about 1000 years long soil formation is too short to manifest recrystallization of carbonate in a desert soil of Nevada. There may be two explanations for the difference between their and our results. First, the more humid climate of the study site in our case could have been responsible for more active processes of carbonate dissolution in Balis. Second, lithogenic carbonate has obviously in most cases a much broader ^{14}C range prior to pedogenesis than that in the artificial mortar and is probably a less sensitive object in terms of diagenetic alteration.

A quantitative estimation of the percentage of diagenetically recrystallized (^{14}C -contaminated) carbonate is of theoretical and practical interest. As already mentioned, besides carbonate cement with carbon of atmospheric origin, artificial mortars contain originally a certain proportion of primary limestone with radiometrically "dead" carbon (Baxter and Walton, 1970; Kedar and Mook, 1978; Folk and Valastro, 1985; Zouridakis *et al.*, 1987). Although the ^{14}C content and radiocarbon ages (Table 1, Figure 2) indicate that the percentage of primary limestone in samples is relatively high, an exact value of this percentage in initial mortar mixture is unknown and a precise quantification of reprecipitated carbonate is, therefore, problematic. However, some limits of the proportion of this carbonate can be estimated. Because radiocarbon activity of mortar carbonate does not exceed c. 17% (profile 1, 10-20 cm), which corresponds to approximately 19% in the 7th century A.D., the proportion of diagenetically altered calcium carbonate in the mortar is not higher than of the order of 20%. Carbonate in the lower 0.5 m of the profiles is better preserved with ^{14}C activity ranging from 8 to 11% which corresponds to roughly 9 to 12% in the 7th century AD. It implies that not more than of the order of 10% of mortar carbonate is diagenetically recrystallized. Furthermore, it is also evident that the proportion of recrystallized carbonate in the 10-20 cm horizon is not lower than the difference between the above-mentioned limits, i.e. of the order of $20-10=10\%$.

Stable carbon isotope composition of the mortar

$^{13}\text{C}/^{12}\text{C}$ ratios of carbonate in mortar samples vary only slightly over the profiles with most $\delta^{13}\text{C}$ values lying in the range from -1 to 0‰ (Figure 2). Three separate fragments of mortar from the same depth 60-70 cm in the profile 1 have very similar $\delta^{13}\text{C}$ values (Table 1, Figure 2), which indicate that the differences in carbon isotopic content at diverse depths over the profiles is a function of diagenetic alteration rather than a variation of initial concentration of primary limestone in the mortar. The distribution of $\delta^{13}\text{C}$ values show no definite correlation with the diffusion-production model (Cerling, 1984; 1991; Cerling *et al.*, 1989), which implies that the mortar recrystallized on a very limited scale and is in general agreement with the radiocarbon data. An absence of vertical trends in ^{14}C content was found in some desert soils in young substrates (c.1,000 years old), whereas carbonate in soils in older substrates (3,800 and 6,300 years old) was mostly recrystallized and had $^{13}\text{C}/^{12}\text{C}$ ratios as described by the diffusion-production model (Pendall *et al.*, 1994).

A closer look at the data shows that the relationship between radiocarbon content and $^{13}\text{C}/^{12}\text{C}$ ratios in the mortar in the profile 1 is opposite to that in the profile 2 (Figure 3). The lower is the ^{14}C content in the samples from the profile 1, the lower are the $\delta^{13}\text{C}$ values, whereas the correspondence between these parameters in the profile 2 is mostly

inverse. A plausible explanation for this phenomenon could be that the $\delta^{13}\text{C}$ value of the limestone species used for preparation of a mortar mixture in the palace room located at the site of the profile 1 was different from that in the room at the site of the profile 2. If $\delta^{13}\text{C}$ of recrystallized carbonate lies within the range of values of the two limestone species, diagenesis should cause an equalizing of $^{13}\text{C}/^{12}\text{C}$ ratios of mortar carbonate in the two profiles and produce $\delta^{13}\text{C}$ patterns like the observed ones. To verify this suggestion, relatively large (2-10 mm in length) and dense limestone fragments (presumably weakly or not weathered) found in the mortar samples from every profile were analysed. Indeed, the $\delta^{13}\text{C}$ value of carbonate in the profile 1 (20-30 cm) was -0.78‰ , whereas that for the profile 2 (70-80 cm) was $0,15\text{‰}$ (Table 1). Because of a high porosity and a high concentration of the atmospheric CO_2 in the mortar, it is possible that $\delta^{13}\text{C}$ of recrystallized carbonate lies in the range between these values (Cerling, 1984; 1991; Cerling *et al.*, 1989). The $\delta^{13}\text{C}$ values of limestone fragments support, therefore, the suggested explanation of the opposite trend in relationships between radiocarbon and $^{13}\text{C}/^{12}\text{C}$ data in the profiles 1 and 2. It should be stressed, however, that much more further data are needed to prove the hypothesis.

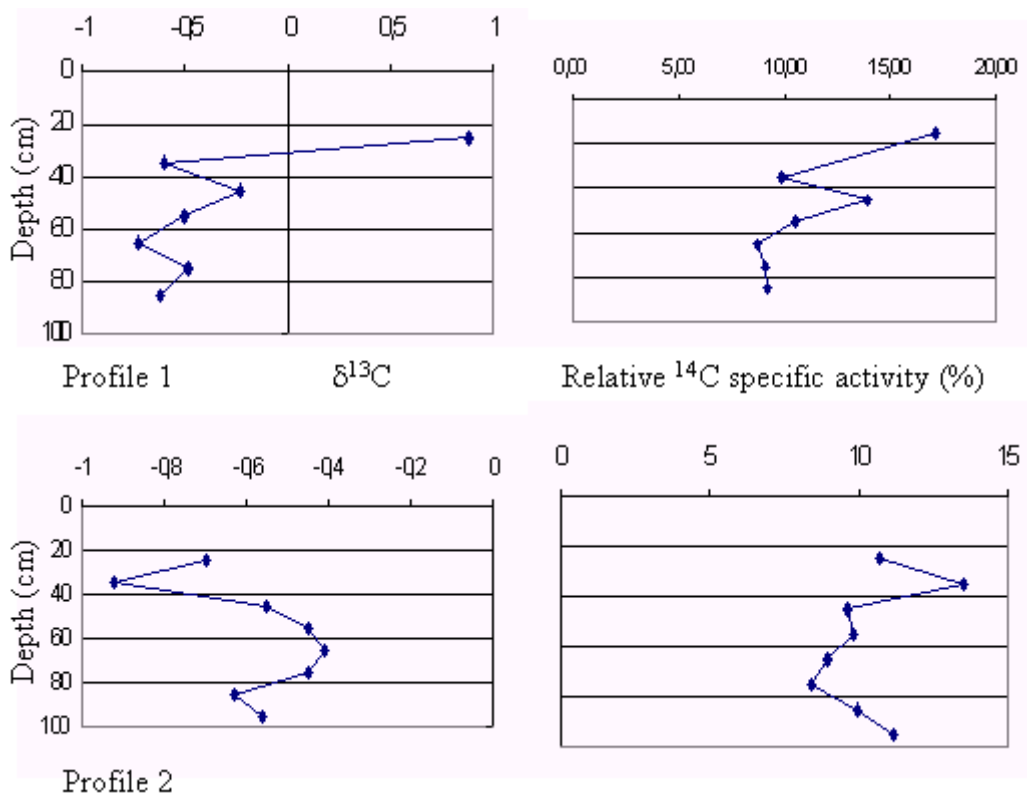


Figure 3 Stable carbon isotopic composition of the mortar and its comparison with radiocarbon activity in the mortar.

Conclusions

Radiocarbon data on an artificial lime mortar demonstrate that a not longer than c. 1,200 years period of soil formation under Mediterranean climatic conditions is sufficient to produce a definite pattern of diagenetic contamination of soil carbonate by environmental ^{14}C . Two profiles of mortar carbonate in soil show a pronounced increase

of radiocarbon activity from the bottom upwards today, whereas there was presumably no vertical differentiation of ^{14}C until the early years of the 9th century A.D. Radiocarbon activity increases from 8-11% at 50-100 cm depth to 17% at 10-20 cm depth. Calculated proportion of diagenetically recrystallized carbonate at 10-20 cm below the soil surface ranges approximately between 10 and 20%. At 50-100 cm depth not more than 10% of mortar carbonate are diagenetically altered. By contrast, stable isotopic composition of carbon in mortar shows no regular vertical trends.

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