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CRETACEOUS AGGREGATE AND RESERVOIR EFFECT IN DATING OF BINDING MATERIALS

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Abstract: Lime mortars may contain carbon from different origins. If the mortars are made of totally burnt lime, radiocarbon dating yields the true age of building construction. The presence of carbonaceous aggregates gives the so-called dead carbon effect, which may generate older ages. Another source of carbon is charcoal present in mortars. An attempt has been made to apply the radiocarbon method to mortars of archaeologically estimated age from the Dead Sea region. Petrographical analyses of these samples show the carbonaceous character of the binder and large amounts of limestone aggregate. Determination of the mineral composition of the mortars and comparison with the geology of the surrounding, allows the provenance of the raw materials to be identified. They probably represent the Cretaceous rocks of the Judea Group.
Separate radiocarbon dates were made on bulk mortar samples, binder, charcoal fragments and separated fractions from mortars. In the case of binder-aggregate mixture the reservoir effect correction has been applied.

Keywords: mortar dating, archaeometry, charcoal, radiocarbon dating.

1. SAMPLE LOCATION

Due to methodological character of the presented paper, it was necessary to carry out the analyses for objects of at least estimated age by archaeologists. The approximate age was known from relative chronology (Humbert and Gunneweg, 2003; Michniewicz 2009), historical sources e.g. Josephus Flavius and Plinius the Older and previous absolute dating of different materials (Jull *et al.,* 1996; Taylor and Higham, 1998; Van Der Plicht *et al.,* 2003; Higham *et al.,* 2003). The investigated carbonate binders originate from the ancient settlements Qumran, situated at the west coast of the Dead Sea. The traces of the settlements in which the investigated material was collected, go back as far as the Iron Age. However, the dated fragments of mortars have been sampled from the ruins of younger buildings, representing the next stage of inhabitation of the settlements – between II BC and I AD. The approximate age established for these samples (indicated by letters Q and QA) by archaeological research is 104 BC - 68 AD (Humbert and Gunneweg, 2003).

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There are two main hypotheses connected with Qumran from where the samples were taken. One of them connects the settlement's existence with the scrolls found in the nearby caves (de Vaux 1953, 1973, Humbert 2006). The second hypothesis rejected the religious character of the settlement and its Essene genesis (Golb, 1980, 1994). Recently, scientists are of the opinion that the presence of Essenes in Qumran is more than probable (Humbert, 2006). The purpose of this paper is to verify the possibility of radiocarbon dating of mortars with various, carbonaceous aggregates.

All the mortar samples are represented by the fragments with carbonate binder applied, what has been confirmed by petrographic observations. The studied mortars contain as their constituent the aggregate, which composition has been macro and microscopically defined as a mixture of flints, limestones and dolomites. The occurrence of charcoal in the mortars has also been noticed. Generally, the petrographic composition of the aggregates in particular samples is similar. However, one can observe the differences in aggregate content concerning individual mortars and sometimes also the divergence in aggregate fraction. Small amounts of clay substances also occur in the mortars. The aggregate from analysed mortars show the distinct presence of scattered shells and crumbled limestone with less dense foraminiferous ones (Nawrocka *et al.,* 2005). Identification of carbonaceous aggregate is very important taking into account the necessity of "dead carbon" elimination.

The knowledge of geological structure and land relief coupled with the research results allow to indicate for the samples from the west coast of the Dead Sea the Cretaceous carbonate rocks as a building material (**Fig. 1**). The aggregate of the mortars, composed of different types of

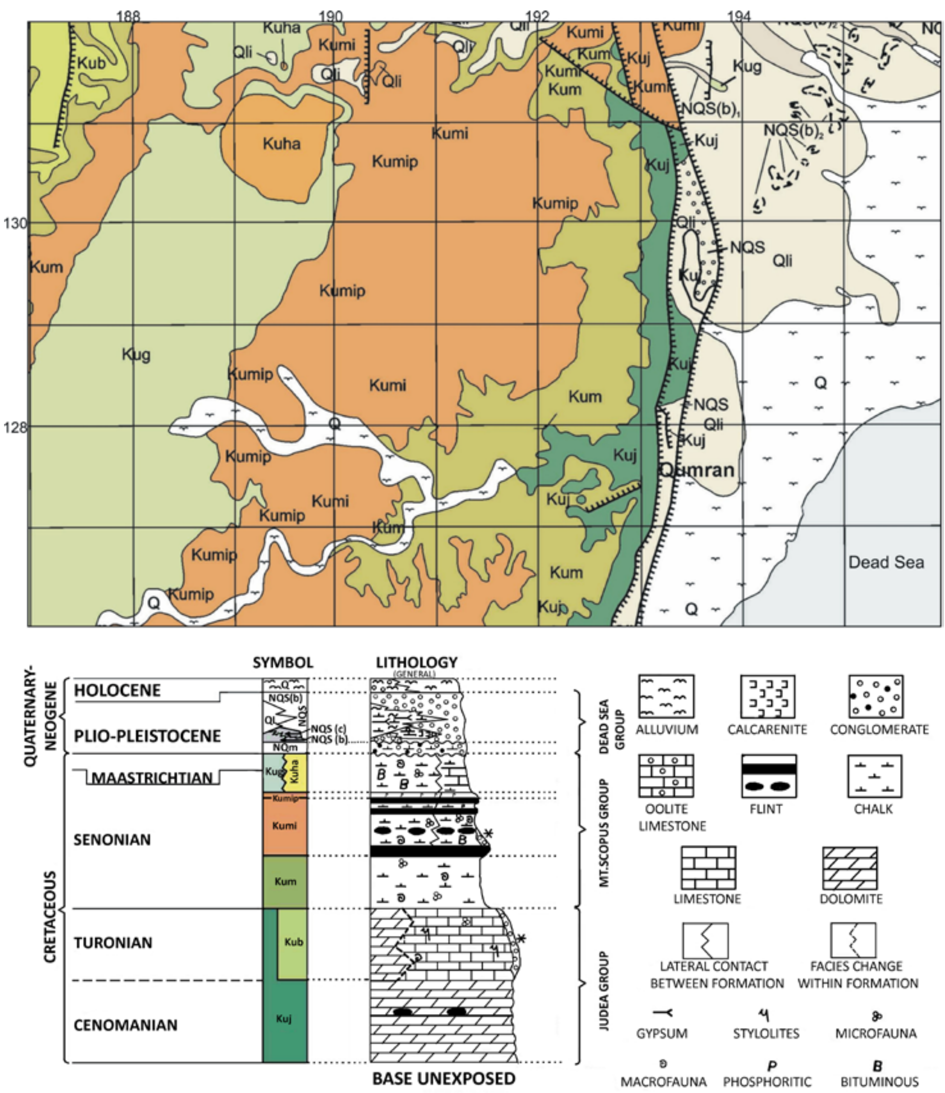

Fig. 1. *Geology of the area (Begin, 1974; Roth, 1974); a – fragment of the geological map 1:50 000, sheet Wadi el Oilt; b – lithostratigraphy of the NW coast of the Dead Sea.*

limestones with flint fragments corresponds to unconsolidated sediments covering the plateau in the neighbourhood of the ruins, which form the weathering detritus of the Cretaceous rocks (Begin, 1974; Roth, 1974; Nawrocka *et al.*, 2005).

2. SAMPLE SELECTION FOR DATING

The sample preparation for dating is performed in different ways, depending on the studied material, on the basis of a procedure established in a given laboratory. Before selecting the samples for dating, in case of carbonate mortars the identification of mortar components is necessary. The first group of the samples has been composed of the mortars dated by the gas proportional counter technique (GPC). It was attempted to separate from them the appropriate amount of binder by alternate freezing and defrosting of the mortar, up to the moment of its intrinsic partitioning into individual elements. Then, the material was selected using binocular microscope. The samples, for which it was impossible to obtain a sufficient amount of binder, were dated in bulk. They were a mixture of aggregate and binder, therefore the application of the reservoir age correction was necessary (**Table 1**, Nawrocka *et al.,* 2005).

In the next step, the suspension fractions separated from the samples QA and Q6 after sieving and the sample Q3 dry sieved have been dated (**Table 3**). The comparison of results has been preceded by observations on rate and course of the leaching reactions for specific fractions

Table 1. *GPC ^{14}C dating results for lime mortars (Nawrocka* et al.*, 2005) together with calibrated dates (Bronk Ramsey 2009, 2010; Reimer* et al.*, 2009).*

Sample name/M	Lab. Code	$T_C \pm \Delta T_C$ (BP)	$\delta^{13}C$ (‰ VPDB)	$T_{CCA} \pm \Delta T_{CCA}$ (BP)	Cal. Age (cal BC) 68% conf. intervals	Cal. Age (cal BC) 95% conf. intervals
Q */S	Gd-18007	3810±210	(-9.)^A	3810±210	2570 – 2525 (3.4%) 2500 – 1955 (64.8%)	2875 – 1745 (95.4%)
QA /S	Gd-15262	2810±130	-9.74	2810±130	1130 – 820 (68.2%)	1385 – 770 (95.4%)
Q1/S	Gd-15264	4360±220	(-9)^A	4360±220	3365 – 2850 (59.5%) 2815 – 2745 (6.1%) 2730 – 2695 (2.5%)	3635 – 3560 (2.1%) 3540 – 2470 (93.3%)
Q6/S	Gd-15226	3250±120	-8.11	3250±120	1685 – 1415 (68.2%)	1880 – 1835 (1.9%) 1830 – 1790 (1.5%) 1785 – 1260 (92.1%)
Q1/SK	Gd-15223	6640±110	-7.69	6210±180	5360 – 4940 (68.2%)	5510 – 5500 (0.1%) 5490 – 4720 (95.3%)
Q2/SK	Gd-15225	4730±90	-11.0^B	7180±150	6225 – 5965 (58.9%) 5955 – 5905 (9.3%)	6375 – 5760 (95.4%)
Q3/SK	Gd-15228	8750±130	-7.37	7980±200	7175 – 7155 (1.4%) 7145 – 6635 (66.8)	7455 – 7395 (2.1%) 7375 – 6465 (93.3%)
Q4/SK	Gd-12312	6020±80	-10.41^B	8030±150	7145 – 7095 (5.8%) 7090 – 6690 (62.4%)	7455 – 7405 (1.5%) 7370 – 6590 (93.9%)
Q5/SK	Gd-11609	15080±90	-2.44	5430±350	4690 – 3940 (65.8%) 3860 – 3815 (2.4%)	5210 – 5160 (0.6%) 5120 – 5100 (0.2%) 5080 – 3625 (93.4%) 3600 – 3525 (93.4%)
Q6/SK	Gd-12313	9220±80	-6.13	6970±180	6020 – 5705 (67.5%) 5685 – 5675 (0.7%)	6225 – 5605 (93.7%) 5600 – 5555 (1.7%)
Q10/SK	Gd-12304	15820±200	-2.33	5800±410	5215 – 4320 (67.0%) 4290 – 4265 (1.2%)	5610 – 5595 (0.1%) 5560 – 3910 (94.1%) 3880 – 3800 (1.2%)
QR3/K			-0.06			

Explanatory notes:
A = the estimated $\delta^{13}C$ values;
B = the estimated $\delta^{13}C$ value of the binder (-8.11) is higher than the $\delta^{13}C$ value of the dated material (binder with aggregate). It resulted in T_{CCA} value higher than T_C;
M= type of dated material; S = binder; K = aggregate; SK = binder with aggregate;
T_C = Conventional radiocarbon age of the dated mortar fraction (binder or binder with aggregate)
T_{CCA} = Conventional radiocarbon age of the binder, obtained after taking into account the correction for the content of old, radiocarbon-free aggregate in the dated sample made of binder with aggregate. In case of the binder sample dating, the age is identical with the T_C age;
ΔT_C, ΔT_{CCA} = the estimated uncertainties of the given ages;
Cal age = Calendar (calibrated) age interval on the confidence level of 68.2% (T_{CCA} was calibrated using OxCal program v4.1.7, Bronk Ramsey 2009, 2010; Reimer et al.*, 2009);*
Roman samples from the north-western bank of the Dead Sea, are indicated by letters Q and QA;

of a given sample (Nawrocka *et al.*, 2007; Nawrocka *et al.*, 2009a; Lindroos *et al.*, 2007). From those samples, fractions below 45 µm have been rejected, because in so fine fraction the acid-leaching reaction is very fast for binder and aggregate, especially soft and fragile foraminiferous limestone and crushed shells (Goslar *et al.,* 2009).

For sample Q4, after sieving there was not enough material for dating, but the charcoal separated from binder was measured by AMS technique. The influence of different mortar composition and fraction on radiocarbon dating results is shown in **Fig. 2**.

3. GPC DATING RESULTS AND CALENDAR AGE OF THE FIRST GROUP OF SAMPLES

Radiocarbon dating of the mortar samples has been performed separately for the mixtures of binder with aggregate (samples signed SK) and for the separated binders (samples signed S, see **Table 1**, Nawrocka *et al.,* 2005). The binder contains ^{14}C carbon isotope, whereas the aggregate is devoid of it and contains only old inactive carbon. The amount of inactive carbon has been estimated in the binder itself, as well as in the mixture of binder with aggregate. The concentration of ^{13}C ($\delta^{13}C$) is a measure of its content.

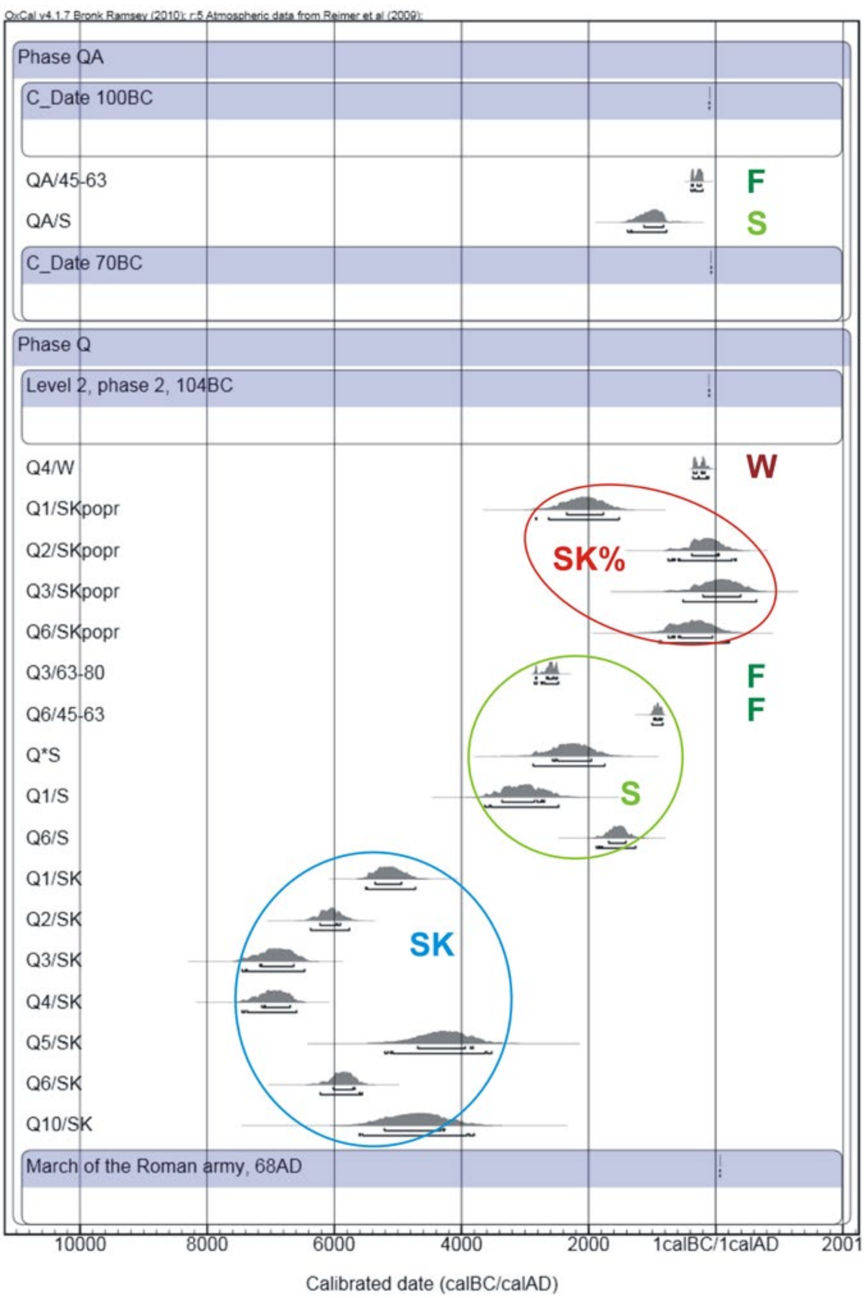

Fig. 2. *Calibration of dating results of the mortar samples prepared in different ways for dating; The binder- results marked with circle in green and signed S; the mixture of aggregate with binder- marked in blue and signed SK; SK% - SK after considering percentage content of aggregate, marked with red circle; W – charcoal; F – mortar fraction. The age uncertainty increased after applying the corrections for carbonate components content. The age was calibrated using OxCal program v4.1.7, (Bronk Ramsey 2009, 2010; Reimer* et al., *2009).*

Different reservoirs of carbon on Earth are characterised by specific $\delta^{13}C$ signatures, therefore it was essential to become familiar with the geology of the region and to recognize the material applied in mortar production (see: Chapter 1). In our case, the carbonate rocks of Cretaceous age were used, which are characterised by $\delta^{13}C$ values close to zero, what is confirmed by the $\delta^{13}C$ value measured in the aggregate (see **Table 1**). In the estimations of the reservoir age (T_R) performed for the investigated samples, constant $\delta^{13}C$ value in the binder equal to -8.11 ‰VPDB, has been assumed for all the samples.

The conventional age (T_{CCA}) has been calibrated using OxCal programme (Bronk Ramsey, 2009, 2010) and IntCal04 curve (Reimer *et al.,* 2009) (**Figs. 2**, **3**; **Tables 1-4**). All the measurements of ^{14}C activity necessary for age determination have been carried out by the proportional gas counter technique in the Gliwice C-14 Laboratory (Pazdur *et al.*, 1999, Pazdur *et al.*, 2000). In case of the two samples of binder-aggregate mixture (Q4 and Q2), the $\delta^{13}C$ values are considerably lower than in all the remaining samples (-11.0 and -10.41 ‰). Here, the reservoir age correction results in higher value of conventional age. This means that the applied model of estimation of the correction gives erroneous results. It may be suspected that the low $\delta^{13}C$ values in these samples are caused by application in the mortar production also the aggregate with the isotope composition differing from the Cretaceous carbonates, e.g. secondarily precipitated carbonates. The answer to this question, as well as more precise reservoir age determinations, may be given by additional isotopic analyses made for the aggregate, and by the $\delta^{13}C$ measurements in binders for all the samples studied. **Table 1** includes also the most probable calendar time intervals, obtained after the calibration of the corrected conventional ages T_{CCA}. In case of the samples Q2 and Q4, the calibration has been applied to the conventional age T_C. In spite of the efforts made to eliminate carbonate aggregate from the binder and after taking into account the $\delta^{13}C$ values, there is still a dramatic difference between the historical data, archaeological estimations and the obtained radiocarbon dating results. The results of mortar dating presented in the paper are far from the real age of the mortar origin, however some relationship is noted.

The age of the binder-aggregate mixture is markedly more over-estimated than the age obtained for the binder separated from the mortar (**Table 1**, **Fig. 2**). This fact confirms the enormous influence of aggregate on the radiocarbon dating results and indicates the necessity of its elimination. The dating has been performed on the samples from the settlement well recognized by archaeologists and previously dated (Humbert, 2006; Higham *et al.*, 2003; Van Der Plicht *et al.*, 2003), thus it is realized that the applied method of separating binder from aggregate did not give the expected effects. The age over-estimation for the samples signed as binder is lower than for the bulk mortars. But in case of binder the determination of "over-ageing" components content is impossible. In case of the bulk mortars, an attempt of the reservoir age calculation considering the percentage content of carbonate aggregate in a

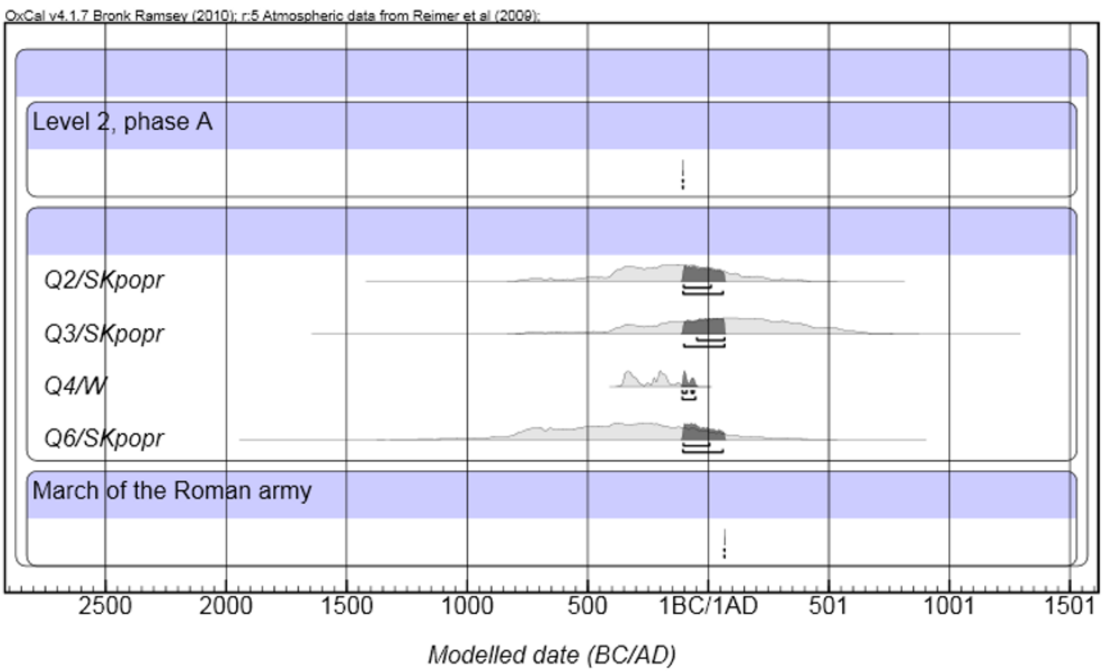

Fig. 3. *Radiocarbon dating interpretation using boundary options in the OxCal programme v4.1.7, (Bronk Ramsey, 2009, 2010; Reimer* et al.*, 2009).*

Table 2. *[14]C age of mortar samples after considering the carbonate components content in mortar during reservoir age calculation; the age estimated by archaeologists basing on artefacts (Humbert and Gunneweg, 2003): 104 BC-68AD. The age was calibrated using OxCal program v4.1.7, (Bronk Ramsey 2009, 2010; Reimer* et al., *2009). The uncertainties of the given ages increase after applying the correction for carbonate aggregate content in bulk mortars, the uncertainties of applied program and phase analysis by man were taken into account. Symbols like in Table 1.*

Sample name	Lab. Code	Binder (%)	Aggregate (%)	Carbonate aggregate (%)	$T_{CCA} \pm \Delta T_{CCA}$ (BP)	Cal. age (cal AD/BC) 68% conf. intervals	Cal. age (cal AD/BC) 95% conf. intervals
Q1/SKpopr	Gd-15223	67	33	30	3670±210	2345 – 1765 BC (68.2%)	2630 – 1515 BC (95.4%)
Q2/SKpopr	Gd-15225	70	30	27	2110±180	380 BC – 30 AD (66.3%) 35 – 55 AD (1.9%)	750 – 685 BC (1.8%) 670 – 640 BC (0.6%) 595 – 575 BC (0.3%) 570 BC – 260 AD (92.2%) 295 – 320 AD (0.5%)
Q3/SKpopr	Gd-15228	38	62	51	1915±250	204 BC – 395 AD (68.2%)	520 BC – 645 AD (95.4%)
Q6/SKpopr	Gd-12313	40	60	55	2270±230	750 – 685 BC (5.9%) 655 – 645 BC (1.9%) 590 – 580 BC (0.6%) 550 – 50 BC (59.8%)	900 – 865 BC (0.6%) 860 BC – 180 AD (94.3%) 185 – 215 AD (0.5%)

Table 3. *Radiocarbon dating results of mortar samples by accelerator technique; symbols like in Table 1 ($\delta^{13}C$ obtained during AMS measurement).W – charcoal; QA/45-63/5s – sample name QA dated in fraction 45-63 µm, gas portion collected in first 5 seconds of leaching reaction; The age was calibrated using OxCal program v4,1,7, (Bronk Ramsey 2009, 2010; Reimer* et al., *2009).*

Sample name	Lab. Code	$T_C \pm \Delta T_C$ (BP)	$\delta^{13}C$ (‰)	Cal. age (cal BC) 68% conf. intervals	Cal. age (cal BC) 95% conf. intervals
QA/45-63/5s	Poz-9818	2240±30	-13.3	385 – 350 (20.8%) 295 – 230 (47.4%)	390 – 345 (26.8%) 325 – 205 (68.6%)
Q 4/W	Poz-5089	2165±30	-22.3	355 – 295 (37.3%) 230 – 220 (3.1%) 215 – 170 (27.8%)	365 – 270 (44.8%) 265 – 110 (50.6%)
Q3/63-80	Poz-18022	4060±40	-10.7	2835 – 2815 (6.0%) 2665 – 2645 (3.9%) 2635 – 2560 (39.1%) 2535 – 2490 (19.2%)	2855 – 2810 (10.4%) 2745 – 2725 (2.4%) 2700 – 2475 (82.6%)
Q6/45-63	Poz-18060	2770±35	-5.8	975 – 955 (11.3%) 945 – 890 (37.1%) 880 – 840 (19.7%)	1005 – 830 (95.4%)

sample will be made. For comparison purposes, also the radiocarbon dating of charcoals from the mortars (Nawrocka *et al*., 2007; 2009a) and fractions separated from the mortars will be helpful (Goslar *et al*., 2009; Heinemeier *et al*., 1997; Sonninen and Jungner, 2001; Lindroos *et al.,* 2007, Nawrocka *et al.,* 2009a).

4. CORRECTION FOR CARBONATE COMPONENTS CONTENT

The conventional radiocarbon age of the binder has been determined using the appropriate procedures for radiocarbon age calculation (Stuiver and Polach, 1977; Pazdur *et al*., 1999) and the equations mentioned by Nawrocka *et al*., (2005). In this step, the percentage of carbonate aggregate has been considered). The possibility of age calculation after considering the correction for the content of old, radiocarbon-free aggregate in the dated sample concerns only the samples of "raw" mortars (**Table 1**).

The applied correction was made using the isotopic analysis, taking into account the percentage of carbonates aggregate in mortars.

The samples from the Dead Sea region were a mixture of old, inactive carbon present in the aggregate and binder containing the same concentration of carbon isotopes as in the atmospheric CO_2 during the hardening process. The overestimation of the ages of a bulk sample could be estimated on the basis of $\delta^{13}C$ value in the binder and in the binder-aggregate mixture.

The percentage content of the aggregate has been determined basing mainly on microscopic images of the mortar samples using e.g. the AnalySIS computer program, option "phase analysis" and "arbitrary area" (Soft Imaging System, 1999). The first command is an automatic algorithm which determines areas covered by different (grey scale) fractions. The second one, allows a manual choice of measured fractions (aggregate fragments). To calculate the reservoir age, the carbonate components content has been recalculated to 100%. Due to the limitations caused by size of the available samples, the analysis was possible only for four mortar samples (see **Table 2**).

The correction for percentage content of carbonate components in mortars, which was applied during reservoir age calculation, enabled obtaining results contained within time boundaries of the settlement existence. However, attention must be paid to large measurement uncertainty. The result for the sample Q1 still departs from predictions. It may be caused either by presence of numerous, very fine fraction of crushed shells in this mortar or by wrong estimation of carbonate aggregate percentage content on the basis of microscopic image. One should remember that microscopic thin section is a small-size object (2×3 cm) and the mortar fragment observed under the microscope may not be representative of the whole sample, even if the measurements were performed several times. If larger amount of the sample would be available, the doubts could be removed by preparation of several thin sections of the mortar coming from a given wall of the object.

5. DATING BY AMS TECHNIQUE

The accelerator mass spectrometry (AMS) technique has been used for dating of charcoal (sample Q4) and two binder fractions separated from mortar (**Table 3**). The Poznan Radiocarbon Laboratory is equipped with an accelerator mass spectrometer type 1,5 SDH-Pelletron, Model "Compact Carbon AMS".

The samples Q3 and Q6 display similar proportions of binder and aggregate, thus two fractions have been selected for dating in order to evaluate the impact of inactive carbon on dating results of a given fraction.

6. SUMMARY OF THE RESEARCH RESULTS FOR CARBONATE MORTAR SAMPLES

The carbonate mortar samples coming from the ruins of the settlements at the west coast of the Dead Sea have been dated by accelerator technique and by proportional gas counter technique. The first results obtained for the "raw" samples and for separated binder (**Table 1**, **Fig. 2**), although indicated good tendency in the applied method of sample preparation, have been far from the real age of those mortars, which was known from archaeological-historical context. Recognition of the sources of material applied for the mortar production and estimation of percentage content of its components had considerable significance for working out the reservoir age correction.

The determination of percentage content of aggregate and binder in the studied samples allowed taking into account these values in the calculation of reservoir age. The applied correction enabled to obtain the calendar age intervals contained in the time frame indicated by relative dating methods. The dating of two fractions from the mortars indicates their contamination with older carbonate aggregate. In such a case, jointly with the fraction size the content of "over-estimating" components also increases (Goslar *et al.*, 2009; Nawrocka *et al.*, 2009a). The dating result of the charcoal from mortar, slightly shifted towards ages older than expected, may suggest its origin from older wood fragments or, what is more probable in this case, the accidental addition of small charcoal fragments in the mortar during production process. The material chosen for dating were collected after leaching the carbonaceous fractions of mortar and collecting the charcoal "residue" (instead of one charcoal fragment) to give mass adequate for radiocarbon dating. The comparison of all the radiocarbon dating results received for the carbonate mortars reveals enormous impact of the applied preparation method on the obtained age (**Figs. 2**, **3**, **Tables 2**, **3**, **4**). Dating of samples after alternate freezing and defrosting of a mortar, up to the moment of its intrinsic partitioning reflect the attempt of carbonate aggregate elimination, showing at the same time the results younger (e.g. Q6/S) than for bulk material (Q6/SK), but still overestimated. In spite of the mechanical separation and considering the value of $\delta^{13}C$, the dead carbon effect was still visible.

Dating of separated grain fractions in two cases – suspension from samples QA and Q6 - gives ages closer to historical premises than results obtained for the same samples after freezing and defrosting disintegration process. The dried sieved samples Q3/63-80 still show large "dead carbon effect". It could be connected also with the presence in this fraction of crushed shells, observed in bulk samples during petrographical analyses. Previous research showed that elimination of small, crumbled pieces of formaminiferous limestone and shells scattered in a sample is very difficult and practically preclude its ^{14}C dating (Goslar *et al.*, 2009).

The whole range of fractions for all samples could not be applied in this case because of the lack of a sufficient

Table 4. *Calibration results in the tabular form, after application of boundary options for the samples of carbonate mortars.*

Sample name	Lab. Code	^{14}C age $\pm\Delta T_C$ (BP)	Cal. Age (cal AD/BC) 68% conf. intervals	Cal. Age (cal AD/BC) 95% conf. intervals
Q2/SKpopr	Gd-15225	2110±180	102 BC – 5 AD (68.2%)	104 BC – 62 AD (95.4%)
Q3/SKpopr	Gd-15228	1915±250	50 BC – 68 AD (68.2%)	101 BC – 68 AD (95.4%)
Q4/W	Poz-5089	2165±30	108 – 91 BC (50.0%) 71 – 62 BC (18.2%)	108 – 54 BC (95.4%)
Q6/SKpopr	Gd-12313	2270±230	104 BC – 3 AD (68.2%)	106 BC – 61 AD (95.4%)

amount of material. Comparing all the obtained results, it must be noted that every step, such as mechanical disintegration of samples, consideration of $\delta^{13}C$, or grain fractions dating were less overestimated than the first dating results for these samples from the Dead Sea region, but did not allow for total elimination of the dead carbon effect. The closest to relative chronology were the results obtained after applying correction for carbonaceous aggregate content in bulk samples (except Q1/SKpopr), marked as SK% in **Table 2**.

The results within the time interval established by the scientists who investigated the settlements have been additionally analysed (**Figs. 2**, **3**). The year 104BC regarded as the beginning of the level 2, phase A (after Humbert and Gunneweg, 2003) can be treated as previously called *terminus poste quem* (TPQ) – i.e. any dated sample should not be older than this age, and the year 68 AD – the date of the Roman army marching into the settlement – may define the so called *terminus ante quem* (TAQ), thus any sample should not be younger. The radiocarbon ages and the above-mentioned information have been joined using *boundary* options in the OxCal programme (**Fig. 3**, **Table 4**).

CONCLUSION

Building mortars, as the material directly linked to the construction of the building, can be a valuable source of information about its age, being aware of the limitations resulting from the presence of carbonate aggregate and recrystallization of binder (Lindroos *et al.*, 2007, Nawrocka *et al.*, 2009a, 2009b), (causing rejuvenations of results; not present in analyzed samples), as both those factors have considerable influence on the ^{14}C measurement results. Radiocarbon dating of mortars requires special sample preparation methodology, larger than standard amount of a sample and somewhat longer time for its preparation.

The necessary part of the investigations is petrographic observations in macro- and microscale. They supply basic information about the possibility of dating particular mortars or their exclusion from measurements. They also allow for establishing proper steps of sample preparation (sieving, fraction selection, choice of analytical technique). The measurements by AMS technique may be applied for very small samples facilitating selection of appropriate material. However, it does not remove error sources – especially in case of the samples containing very fine, crumbled fragments of shells (Nawrocka *et al.,* 2009a). The inaccuracies can be eliminated by dating the selected fractions of the mortar using time-resolved leaching, but it implies dating of several gas portions coming from one sample (Goslar *et al*., 2009).

For dating by GPC technique, larger samples are required. Thus, the efforts to carry out the separation of carbonate aggregate only diminish the over-ageing effect, but do not exclude it completely. However, the first attempts to apply the reservoir age correction for the mortars (bulk samples) taking into consideration the percentage amount of aggregate, seem to be promising. The analyses would need verification on the more extensive group of mortars. Positive validation of the method including the introduced correction would allow dating of bulk mortar samples without special preparation. For this purpose a sound knowledge of geology of the material source area, coupled with detailed petrographic investigations of the mortars, would be required. The information about carbonate aggregate content, their provenance and geological age will be very important. In case of carbonate aggregate with different geological age, the application of appropriate correction will be much more complicated.

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