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beyond the theme

# Experimental rehydroxylation and the dating of early medieval and Byzantine ceramics. A southern Italian case study

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This article explores rehydroxylation dating analyses (RHX) of samples of pottery found in early medieval archaeological contexts is southern Apulia which have, in part, been dated through $^{14}C$. One of the objectives is to test the RHX method and its potential by comparing it with radiocarbon dating of associated organic materials. The application of RHX on coarse wares, that up until now have covered a rather large date-range, has allowed us to formulate some preliminary hypotheses that refine the seriation of ceramic indicators for Byzantine contexts. Indeed, the preliminary dating of some types is confirmed, whilst in other cases (particularly as regards products of the 10$^{th}$ and 11$^{th}$ centuries) it is now possible to distinguish phases in ceramic production, thus allowing us to reconsider the development of ceramics during the transition from Byzantine to Norman times.
**Keywords:** Apulia, dating, RHX, early medieval pottery

*Il contributo offre una riflessione su delle analisi condotte con il metodo della* rehydroxylation dating *(RHX) su campioni ceramici altomedievali provenienti da contesti archeologici della Puglia meridionale, questi ultimi in parte già datati tramite il $^{14}C$. Uno degli intenti è infatti quello di testare l'RHX e le sue potenzialità in congiunzione con la datazione radiometrica dei materiali organici. L'applicazione del RHX su ceramiche d'uso domestico, inquadrabili in orizzonti cronologici piuttosto ampi, ha permesso di formulare alcune ipotesi preliminari per affinare la seriazione degli indicatori ceramici dei contesti di età bizantina. Infatti, la datazione iniziale di alcuni tipi viene del tutto confermata, mentre in alcuni altri casi (in particolare per quanto riguarda i prodotti di X e XI secolo) è ora possibile distinguere fasi di produzione differenti, permettendo di riconsiderare le forme delle ceramiche nel passaggio tra la dominazione bizantina e quella normanna.*
***Parole chiave:*** *Puglia, datazione, RHX, ceramica altomedievale*

## 1. Introduction

The study and dating of ancient ceramics provide a cornerstone to much archaeological investigation and assume particular importance for the dating of later prehistoric or early Middle Ages stratified contexts,

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when other chronological indicators are often rare or missing. Direct or indirect methods of pottery dating, notably luminescence-based approaches (Wintle 2008) or archaeomagnetism (Sternberg 2008), and even radiocarbon dating of surface encrustation or residues absorbed by ceramics (Nakamura *et al.* 2001; Berstan *et al.* 2008) have become valuable tools in dating pottery upon which our understanding of human social and economic development can largely be structured. Stratified contexts, instead, are often dated through finds analogies and relative stratigraphic dating, which over the years have often had to rely on radiocarbon dating of associated organic materials such as bone, shell or carbonised plant remains. These techniques do, however, have limitations in their application and in some contexts are not applicable (Bonsall *et al.* 2002).

A relatively new, alternative or ancillary approach, the rehydroxylation dating method (RHX), was first proposed following investigations into the expansive properties of fired clay bricks (Wilson *et al.* 2003) and has since undergone considerable investigation (Savage *et al.* 2008a, 2008b; Tosheva *et al.* 2010; Bowen *et al.* 2011, 2013; Clegg *et al.* 2012; Bowen *et al.* 2013). This has culminated in its recent application to the dating of bricks and tiles (Wilson *et al.* 2009) and pottery from archaeological contexts (Wilson *et al.* 2012) up to 2000 years old.

Central to this dating method are several assumed properties of fired clays, including:

1) the mass gain curve, associated with uptake of environmental moisture following firing/reheating, is a two-stage process with the second of these, stage II, described by a time1/4-based relationship (Savage *et al.* 2008b);
2) the mass gain behaviour following reheating at a suitable temperature (500°C) is identical to that of the original freshly-fired sample provided the aging temperatures (environmental conditions under which the samples gain mass) are equal (Savage *et al.* 2008a; Tosheva *et al.* 2010);
3) the second stage process continues indefinitely, beyond the age of the ceramic (Wilson *et al.* 2003; Hall *et al.* 2011);
4) the second stage mass gain rate, as a function of t1/4, has an Arrhenius temperature dependence (Wilson *et al.* 2009) plays a significant role in the mass gain over the lifetime of the ceramic (Barrett 2011; Hall *et al.* 2013).

Current understanding attributes the source of the two-stage behaviour to a combination of rehydration/rehydroxylation processes (Wilson *et al.* 2012; Bowen *et al.* 2013). In Bowen *et al.* (2013), Stage I is attributed to both the adsorption of water onto the surfaces of the ceramic (physisorption) and the reaction of water with meta-clays in the sample (rehydroxylation). Once physisorption has reached a state of dynamic equilibrium with the environment, Stage I has ceased and rehydroxylation continues alone in Stage II. Wilson *et al.* (2012) go further and ascribe the mass gain in Stage I to a combination of physisorption and chemisorption of molecular water (referred to as Type 1 and Type 2 water, respectively), and rehydroxylation (Type 3). Stage II is attributed only to rehydroxylation and considered to have commenced once the uptake of Type 1 and Type 2 water is complete.

(A.B.)

## 2. Experimental

This experiment nature of this paper regards both a technical aspect of RHX dating (A), and a control of its effectiveness with regard to recent developments in our understanding of the chronology of early medieval ceramics from southern Apulia, in Italy (B).

A. Technically the experiment is concerned in estimating the Stage II mass gain rates, rates which are used directly in calculating the age of the ceramic as part of the RHX method. The exploitation of an assumed linear Stage II mass gain as a function of t1/4 permits dating of archaeological material with reasonable success (Wilson *et al.* 2009, 2012). The set-up experimental procedure is aimed to examine how the mass gain curves are affected during the period of post-firing/reheating cooling by interferent factors.

B. The ceramics analysed in this paper come from stratified contexts revealed by archaeological excavations conducted in the province of Lecce, in the Salento region of Apulia (fig. 1), and more specifically from the deserted medieval village sites of Quattro Macine (Giuggianello, Lecce: Arthur *et al.* 1996), Apigliano (Martano, Lecce: Arthur, Bruno 2009; Arthur, Leo Imperiale, Tinelli 2015), Scorpo (Supersano, Lecce: Arthur, Fiorentino, Leo Imperiale 2008) and from an early medieval pottery workshop in the town of Otranto (Arthur *et al.* 1992; Leo Imperiale 2003 and 2004). Most of these ceramics appear to be of local manufacture, and have been previously dated either through parallels or through associations with other materials, some of which have been dated themselves by radiocarbon analysis conducted by the CEDAD at the University of Salento.

Preliminarily to dating procedure we performed compositional and structural study of the different ceramic samples. Such a systematic survey of all the samples that will subsequently be subjected to the process of heating and cooling will prevent interference at RHX model due to compositional and structural differences.


Fig. 1. Sites considered in the text.

The second step is to systematically evaluate and analyse the processes of RHX subsequent to thermal processes, developing testing device for pre-heating, heating-cooling method and numerical analysis of RHX data.

(P.A., A.B.)

## 3. The samples

The samples of pottery subjected to analysis come from a series of early medieval forms that over the last few years have become index fossils for the dating of Byzantine contexts in southern Apulia. An ever-greater precision in the dating of these forms is, thus, an essential condition for more faithful reconstruction of population dynamics in a territory and for a period in which there is a penury of alternative chronological indicators such as coins, so useful for other periods. This scarcity of chronological evidence has, up to now, conditioned the breakdown of early medieval chronologies into two distinct macro-periods:

1) a period dating between the late 7$^{th}$ and 9$^{th}$ centuries, characterised by globular amphorae, both for commercial transport and domestic use, cooking pots and other forms of types fairly uniformly distributed in the Salento and in Greece, which have defined a 'typical' ceramic assemblage that might be defined as 'western Byzantine' (Arthur 2004; Arthur, Leo Imperiale 2015) (fig. 2);

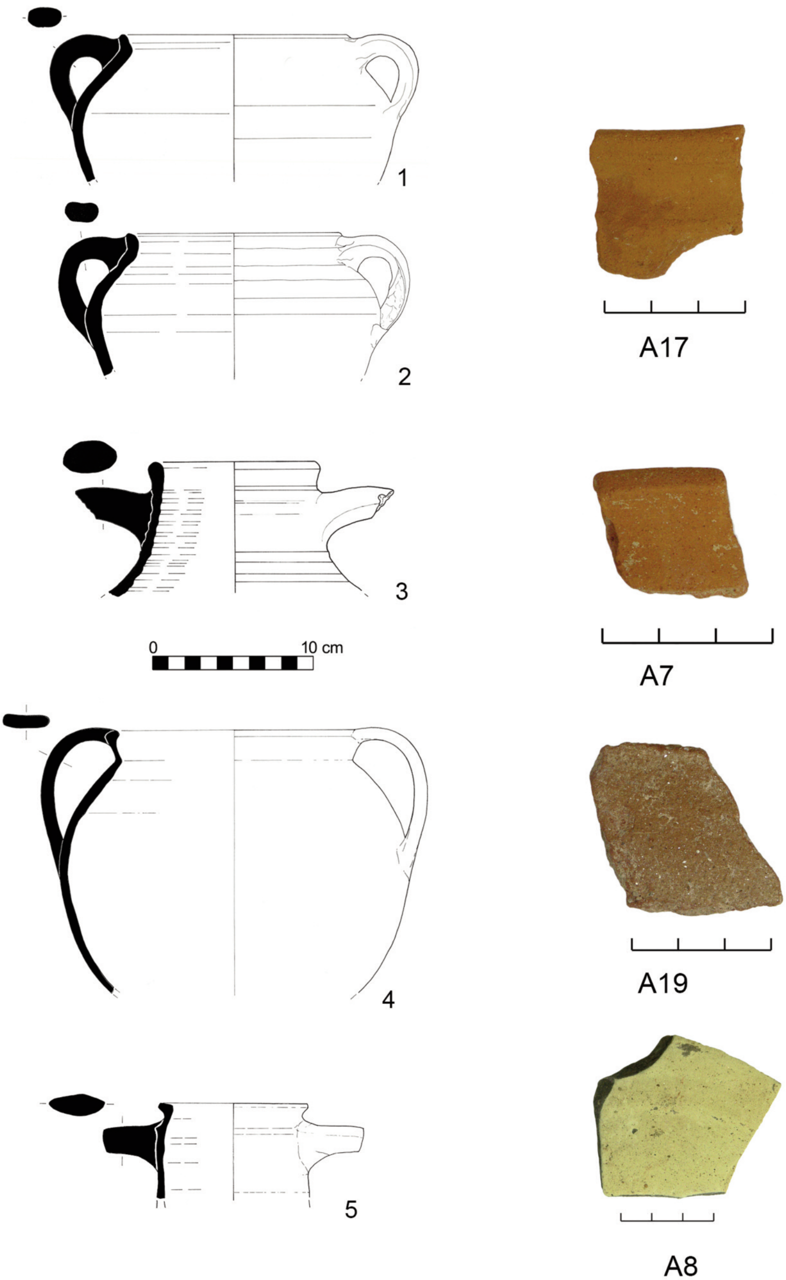


Fig. 2. Early medieval/Byzantine ceramics from southern Apulia (late 6th-9th centuries): 1-2. Cooking pots from Apigliano (as sample A17); 3. Domestic amphora from Supersano (cf. A7); 4. Micaceous cooking ware from Otranto (cf. A19); 5. Domestic amphora from Supersano (cf. A8).

2) a period dating between the end of the 9th and the later 11th centuries, in which the locally made domestic wares change significantly (Arthur 2004; Leo Imperiale 2014). This is probably due to different influences, in a composite framework that combines technology transfer and costumes, in part directly from Byzantium, in part from other southern Italian regions and perhaps also under the influence of immigrant populations and their material culture. In this period we see the appearance of jugs with excised decoration (or fine orange-burnished ware), the painted wares are very similar to those made in other parts of southern Italy, whilst the cooking pots distance themselves from the Byzantine models (fig. 3). Also characteristic of this period are the so-called "Otranto-type" amphorae, partly produced in Apulia and similar to other types circulating in the Byzantine Empire and beyond at this time.

Nineteen samples were submitted to analysis, of which nine have been dated so far. All the samples were taken from recognisable ceramic types and from closed contexts that had been previously dated on association with other finds, through stratigraphic grounds, and sometimes through $^{14}C$ dating of associated organic material (bone or charcoal).

Samples A1-A3 and A17 come from Apigliano. The first three are from vases that were within the fill of a pit, which also provided two radiocarbon dates of 870-1000 cal AD and 960-1040 cal AD, and a *terminus post quem* from a bronze *follis* or coin of Constantine VII and Zoe of AD 913-919. Sample A17, instead, came from the fill of a possible sunken featured building (SFB), which has also yielded a $^{14}C$ date of 770-980 cal AD, but for which we preferred the tighter date range of late 8th to early 10th century on ceramic analogies (Arthur, Leo Imperiale 2015; Leo Imperiale 2014).

Samples A7 and A8 were obtained from vessels found in two separate pits, both interpreted as SFBs, excavated at the early medieval site of loc. Scorpo, Supersano. The vessels represent two different types of globular amphorae, principally for domestic use, almost certainly from two distinct local or regional workshops. $^{14}C$ dating of associated organic materials chronological horizon of later 7th or 8th century for both, although stratigraphic considerations suggested the amphora of sample A8 may have been the later of the two.

Samples A11 and A12 come from ceramics found in a pit fill excavated in 1991 at Quattro Macine, not far from Otranto. The ceramic assemblage, which includes cooking vessels, broad-line wares, spot-painted ware, excised ware and examples of the so-called Otranto-type commercial amphorae, would appear to date to the later 9th-11th centuries.

Last but not least, sample A19 represents a micaceous fabric cooking pot, possibly of Aegean origin, from an early medieval context at

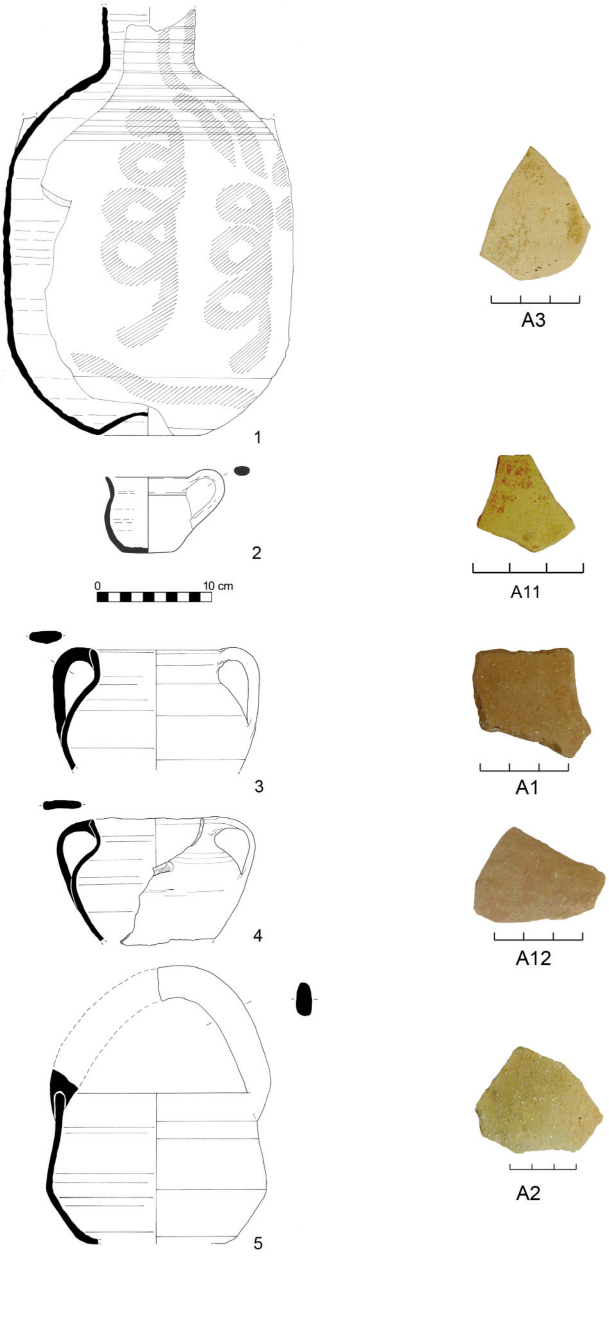


Fig. 3. Medieval/Byzantine ceramics from southern Apulia (10th-11th centuries): 1. Broad-line painted jar from Apigliano (cf. sample A3); 2. Beaker from Quattro Macine (sample A11); 7. 3. Cooking pot from Apigliano (cf. A1); 4. Cooking pot from Quattro Macine (sample A12); 5. Ceramic pail from Apigliano (sample A2).

| Sample | Type | Proposed context date | $^{14}C$ dating | Notes |
|---|---|---|---|---|
| A1 | Cooking pot (Apigliano, US 1061) | 10th-11th cent. | 960-1040 cal AD | Coin of Constantine VII and Zoe (913-919 AD) |
| A2 | *Paiolo* (Apigliano, US 1058) | 10th-11th cent. | 870-1000 cal AD | |
| A3 | Painted amphora (Apigliano, US 1058) | 10th-11th cent. | 870-1000 cal AD | |
| A7 | Domestic amphora (Scorpo, pit n. 1, US 5) | Late 7th-8th cent. | 530-660 cal AD<br>650-780 cal AD | |
| A8 | Amphora with pale brown fabric (Scorpo, pit n. 3, US 89) | 8th cent. ? | not available | Later than pit n. 1? |
| A11 | Cooking pot (Quattro Macine, US 33) | 10th-early 11th cent. | not available | |
| A12 | Broad-line ware (Quattro Macine, US 33) | 10th-11th cent. | not available | |
| A17 | Cooking pot (Apigliano, US 2221) | Late 8th-9th cent. | 760-980 cal AD<br>770-990 cal AD<br>540-670 cal AD<br>660-780 cal AD<br>770-980 cal AD<br>(samples from various layers of filling of the pit) | A sherd of GWW came from an upper layer |
| A19 | Imported cooking pot (Otranto Mitello, US 113) | 8th-9th cent. | not available | |

Tab. 1. Different types of chronological data (autoptic dating of finds, $^{14}C$ and RHX) available for the samples.

Otranto. The type appears to be attested in at least two different early medieval contexts within the town.

The tab. 1 summarises the different types of chronological data (autoptic dating of finds, $^{14}C$ and RHX) available for the samples.

(M.L.I.)

## 4. Results

RHX measurements were performed according to methodology suggested by Wilson *et al.* 2014.

First the sample is dried at 105°C until it reaches constant mass, $m_A$. At this point all physically absorbed moisture is driven off and only the chemisorbed water acquired over the sample's lifetime remains.

The sample is then heated to 500°C until it is "emptied" of all water both physisorbed and chemisorbed, in order to perform a complete dehydroxylation of the sample, until reaching a constant $m_{RHX}$ mass value.

The sample is then placed under engineered system in our laboratory in specific and carefully controlled conditions of temperature, pressure and relative humidity. The operating parameters were 20°C, 800 mbar and 30% RH respectively. The dating methods consisted of repeatedly weighting the sample during several months in the constant environmental conditions until the mass gain rate becomes constant. So we use the following power law to fit the RHX mass data. In this equation the parameter *a* is a proportionality constant. The power law allows one, through extrapolation to $m_A$, to determine the time spent since archaeological heating of the sample.

In fig. 4 the principles of the rehydroxylation dating method are reported. Curve (a) represents the mass data obtained for sample A8 after its heating to 105°C and while it remains in constant environmental conditions. The data are plotted against time$^{1/4}$. After the first heating the mass data become constant, in this way we can to define the sample archaeological mass $m_A$. After the heating at 500°C (curve b), the mass

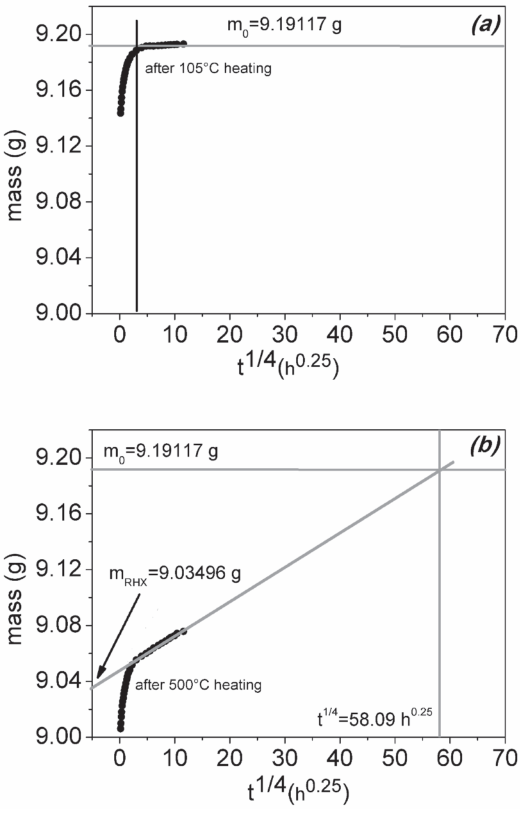


Fig. 4. Experimental mass-gain data subsequent to pre-heating and controlled heating treatment (sample A8).

| Sample | $m_{RHX}$ (g) | $m_0$ (g) | Age (years) |
|---|---|---|---|
| A1 | 17.68374 | 17.86284 | AD 915 +/- 25 |
| A2 | 18.84222 | 19.01199 | AD 990 +/- 25 |
| A3 | 8.95486 | 9.22746 | AD 960 +/- 25 |
| A7 | 5.02622 | 5.07437 | AD 710 +/- 30 |
| A8 | 9.03496 | 9.19117 | AD 715 +/- 30 |
| A11 | 2.611787 | 2.67154 | AD 1065 +/- 25 |
| A12 | 5.140357 | 5.40241 | AD 1060 +/- 25 |
| A17 | 10.07241 | 10.43180 | AD 760 +/- 30 |
| A19 | 12.51973 | 12.92873 | AD 805 +/- 30 |

Tab. 2. Results.

values show an initial linear trend (stage I) followed by a slow rehydroxylation process (stage II). The extrapolation of this behaviour to $m_A$ gives its archaeological age.

By fitting parameters the results in tab. 2 are obtained.

(A.B.)

## 5. Conclusions

Despite the scepticism of some scholars, the preliminary RHX analyses of early medieval ceramics presented in this paper, which should soon be followed by further analyses, appears to illustrate the potential of the method in improving the dating and the chronological seriation of ceramics. The range of the individual dates obtained is currently restricted to within fifty or sixty years. There therefore appears to be enormous potential in the dating method, particularly in cases of rather conservative forms, such as the Byzantine vessels analysed in this paper, which appear to have maintained the same basic morphology for around two hundred years or more in southern Apulia, and which may have persisted much later in parts of Greece (see below). Apart from the dating of archaeological contexts, which precision, however, depends largely on the representativity of the ceramics analysed, we believe that a more extended use of the technique may soon favour a re-examination of morphological types, permitting an assessment of the contemporary and diachronic relationships between the various known types and productions, whether local or imported. Clearly RHX dating should not be considered as alternative to the $^{14}C$ dating of organic materials, which use remains fundamental as well as being now extraordinarily well tested, but can fruitfully be used in conjunction. Furthermore, ceramics in preindustrial societies can sometimes have a very long life, often more than fifty years, and occasionally much longer, and their use could continue even after they had lost their original function. In this sense RHX, which dates the creation of the artefact, might need the help of other dating methods so as to better date their context. This is the case for the dating of samples A1, A2 and A3, which come from a closed context that was probably formed over a very short space of time. The chronological range provided by RHX analysis, whilst precise for each single fragment, nonetheless extends over a period of almost a century, presumably indicating that whilst some artefacts present in the context were manufactured at the beginning of the 10$^{th}$ century, the date of closure of the context should, instead, date to the second half of the century.

This preliminary application of RHX to Byzantine ceramics, though limited in the number of samples, leads us to the conclusion that our work on the chronology of early medieval artefacts carried out over the last twenty years has proved to be well founded. Indeed, the RHX chronologies largely confirm our prior thoughts as to their dating. Furthermore, greater chronological certainty and precision dispels a series of doubts and now permits the formulation of new hypotheses regarding ceramic manufacture, use and their archaeological contexts.

The two samples from the site at Supersano (A7 and A8), up until now believed to date to the end of the 7$^{th}$-8$^{th}$ centuries, provide RHX dates in the first half of the 8$^{th}$ century for two distinct productions of domestic amphorae previously thought to have been not necessarily contemporary. Marginally later (AD 760 +/- 30), though belonging to the same chronological horizon, is the rim of a cooking pot A17, that other evidence suggests could have dated principally to the 8$^{th}$ and 9$^{th}$ centuries. The form is one of the commonest in Byzantine Salento, having been produced at Otranto, and elsewhere, and representing a type fossil for Byzantine sites in the eastern part of the peninsula. Interestingly, the same form appears at Butrint, as well as in Byzantine areas of western Greece and the Peloponnese, including Aegina, Athens, Argos and Corinth. At Corinth, the excavator Guy Sanders, who has been reformulating the chronology of the site (see Sanders 2000), sees it as typical of the 11$^{th}$ century, perhaps continuing into the early 12$^{th}$ (pers. comm.). Joanita Vroom (2005, pp. 104-105) even ventures to date the continuity of the type into the early 13$^{th}$ century, well over a couple of centuries after it had apparently stopped being made and used in Italy. Thus, RHX and archaeological dating of the Salento examples would seem contradictory with the Greek dating, although it is not impossible that the form in Greece witnessed a longer life-span, perhaps because of a more conservative life-style until the Frankish conquest of the 13th century. In such a case, RHX dating would clearly be more reliable than dating based on formal analogy.

Within a substantial period that we envisaged as extending from the 10th (or late 9th) to the end of the 11th century, concluding with the consolidation of Norman rule in southern Italy, the analyses would seem to indicate the existence of two distinct phases, one datable to the 10th (A1, A2, A3) and one to the 11th century (A11, A12). This new understanding, for instance, allows a re-examination of the morphological evolution of cooking pots, taking us beyond the initial idea of these vessel types that we saw as being "different but contemporaneous". Furthermore, the dating from the late 8th to beginning of the 9th century for a cooking vessel in a distinctly micaceous ware, with possible Aegean analogies (ex inf. Rossana Valente), allows us to assess the phenomenon of the importation of pottery to Salento in the early Middle Ages, up until now attested principally by early medieval globular amphorae (Arthur 1998; Leo Imperiale 2015) in a new light. Thus, in providing securer dates, the new technology provided by RHX analysis allows us to add a further dimension to works such as that of Michael McCormick (2001), who has explored innovative routes and indicators for the characters and scale of early medieval exchange in the Mediterranean.

(P.A., M.L.I.)

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