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Contents

This volume is dedicated to the memory of George Pascu Hurezan (1949–2016)

An Ancient Method of Mending a Dacian Vessel. Analysis ATR-FTIR of a binder

Ionuț Ledeți, Adriana Ledeți, Gabriela Vlase, Titus Vlase, Octavian-Cristian Rogozea, Dorel Micle

Abstract: Physical-chemical methods contribute to obtaining very important data regarding the characteristics of a wide range of materials. Techniques such as infrared spectroscopy (FTIR spectroscopy) and thermogravimetric analysis (TG) have been employed in determining the nature of possible traces of repair, namely the structure and characteristics of repairs noted on a pot discovered in a" ritual" pit from the site of Unip-Dealu Cetățuica. The pot is the only of this type discovered in the pit in question, and its typology and breaking have initially led to the conclusion that the item had a ritual function. The obtained results have been analyzed comparatively, i.e. the hypothesis of intentional destruction was partially infirmed by the repairs performed prior to its deposition in the pit.

Keywords: Daci, La Tène, stereoscopy, repair, vessel.

Unip is known in the archaeological biography due to the Bronze Age discoveries made here[1], but especially due to the "Dealul Cetățuica" archaeological site discovered in 1987[2].

This site is located in the Western Plain, namely in its subunit called Timiș Plain, on a hill (Pl. I/1) in the point identified within the following coordinates: GPS 45°40'11.37"N21°17'59.14"E /Stereo70 211911.00618 (long.) 470021.04810 (lat.).

Timiș River grassland situated nearby has been providing favourable conditions for development of human communities in this location, habitation starting in the First Iron Age (Basarabi Culture), followed by the Second Iron Age (I[st] century BC-I[st] century AD) and after that by the Post-Roman period (II[nd]-IV[th] century) continuing during the Early Middle Ages (VI[th]-VII[th] century) and ceasing during the Late Middle Age[3].

Archaeological research carried out here has revealed numerous archaeological complexes with a varied inventory. In the 2012 campaign, among many unbroken or restorable vessels, a vessel provided with an interesting mending system applied over its bottom area has been discovered in a pit.

The Context of Discovery

The vessel has been discovered in Complex 100, outlined in Section 5, on Excavation Plan number 3 at a depth of –50 to –55 cm (Pl. I/3). On the bottom of this complex, at around 1–1.3 m depth from the outlining level, on a bed made of vessel walls, a food vessel supported by a Dacian cup in its neck area, a fruit bowl turned upside down in its belly area and a fruit bowl placed on its edge in its bottom area have been deposited. Noteworthy is the fact that the feet of those two bowls were missing, and were detached (cut) on purpose. In addition to the inventory above mentioned, a sandstone slab, a whole small scale jar made by hand and several fragments of pots also made by hand and decorated with alveolar belts applied under the flaring rim of the vessel or on the belly area have been deposited in the pit near the mended vessel (Pl. I/2).

1 Alexandrescu 1966, 13, 175; Bader 1991, 73; Gumă 1993, 257, 297; Luca 2006, 262.
2 Medeleț, Bugilan 1987, 175
3 Bejan *et al.* 2010; Antoniuc *et al.* 2011, 72–73 2011; Bejan *et al.* 2012; Beldiman 2012; Măruia *et al.* 2011, 505–530; Măruia *et al.* 2013; Micle *et al.* 2016, Rogozea, Berzovan 2016.

Description of vessel

The vessel fired in a reducing atmosphere presents black polished areas (with orange spots on the belly area of the vessel). Fine sand was used as temper in the clay. In terms of size, its rim has a diameter of 15 cm, the bottom 17.5 cm and its largest part (belly) has a diameter of 42 cm. The maximum height of the vessel is 64 cm. The decoration was performed in a very simple manner by modeling a nervure followed by its application on the base of the neck (Pl. I/4–5). The vessel was shaped by hand, therefore the bottom was applied afterwards has detached because of its poor cohesion with the body of the vessel.

This problem has been solved by performing two unidirectional perforations (from outside to inside of vessel with a diameter of approx. 1.1 cm) on opposite position, performed on the wall base and another two unidirectional perforations in the bottom of the vessel (directed from outside to inside) as counterpart of perforations in the wall. These latter perforations have a diameter of 1 cm. Clearly these perforations were used to "sew" the bowl in order to give it mechanical strength. This mending technique, quite common throughout all historical periods, would not give such a distinguished status to this vessel unless the potter/owner would not fill in the cracked area with a black substance probably, with the intention of sealing the vessel (Pl. I/6).

Materials and methods

Thermal stability of the sample was analyzed using a Perkin Elmer DIAMOND thermo-balance, by recording thermoanalytical curves TG, DTG and HF. Samples with mass between 3.3 and 8.3 mg were heated in aluminum crucible between 25 and 500 °C in dynamic air atmosphere (100 mL·min^{-1}) with a heating rate β of 10 °C·min^{-1}. For determining the thermal effects, the DTA data (in μV) were converted in HF (Heat Flow) data (mW). In order to assure the coherence of the data, each analysis was repeated three times and results were practically identical.

FTIR spectra were recorded on two techniques: on a JASCO 670 plus device using the KBr pellet dispersion method vs. UATR-FTIR analysis on a Perkin Elmer SPECTRUM 100 device. Spectra were collected after 64 co-added scans, with a resolution of 4 cm^{-1}, on the spectral domain 4000–600 cm^{-1}.

A comparative discussion of the obtained results by the employed instrumental techniques was carried out.

Results and discussions

As first investigational tool, ATR-FTIR spectroscopy was employed (Figure 1). The FTIR spectrum of the sample is relatively simplistic, consisting in several vibrational bands. A large band in the spectral region 3010–3700 cm^{-1} with a peak at 3326 cm^{-1} suggests the presence of some O-H groups in the structure of the resin. Other characteristic bands were observed at lower wavenumbers, i.e. 2926 and 2896 cm^{-1}, and can be associated with the characteristic vibrations of C-H bonds from alkyl moieties. These bands confirm the presence of organic compounds in the structure of the sample. At lower wavenumbers, different bands appear and their attribution is difficult to be assed – at 1731, 1453, 1370, 1217, 1155, 1078, 1021 and 717 cm^{-1}. The results using the classical KBr dispersing technique revealed a similar spectrum, with the same broad band in the 3010–3700 cm^{-1}, even after the sample was maintained at 50 °C for 24h in order to eliminate the physically adsorbed water. This fact suggests that in the presence of the compound, O-H moieties are still present.

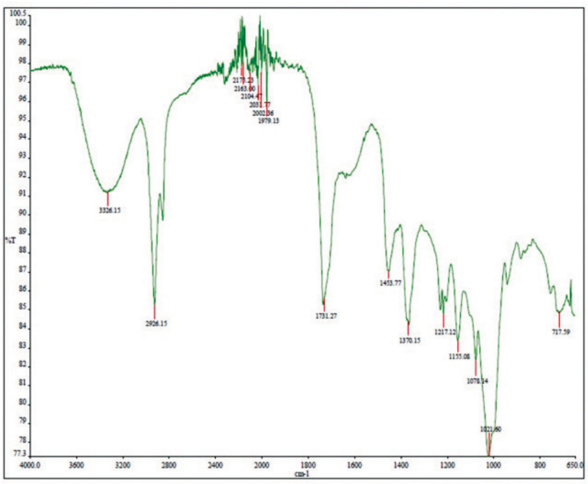

Fig. 1. ATR-FTIR spectra of investigated sample.

In order to predict the composition of the sample, the Sadtler Database of Infrared Spectra-FTIR Spectral Library was used as finding tool. The search revealed some potential compounds to be associated with this spectrum. The results are presented in table 1.

0.774 SE3892	Polycaprolactone triol, average mn ca. 300
0.770 SE6932	11b-Hydroxyetiocholanolone glucuronide
0.768 SE0948	Polyester Polyol 0301 Monomer
0.766 SE0212	Poly(caprolactone)triol (nom mw: 300)
0.766 SE1738	Disperse AYD-6 Anionic Surfactants/Fatty Esters Additive
0.762 SE0485	Methyl acetyl ricinoleate
0.762 SE6878	Etiocholan-3a-ol-17-one glucuronide
0.754 SE0484	Glycerol tri(acetylricinoleate)
0.752 SE7141	K-Strophanthoside
0.749 SE0487	n-Butyl acetyl ricinoleate
0.748 SE8426	Erysimoside
0.745 SE7140	Strophanthin K S6751
0.744 SE4779	Midecamycin, from streptomyces mycarofaciens
0.743 SE3913	(-)-Bornyl Acetate
0.743 SE0159	Poly(caprolactone)triol (nom mw: 300)
0.740 SE1430	4-t-Butylcyclohexyl acetate
0.738 SE0104	5-Isopropenyl-2-methylcyclohexyl acetate; Dihydrocarvyl acetate
0.737 SE3908	(1R)-(-)-Menthyl Acetate
0.733 SE7037	5b-Pregnane-3a,20a-diol diacetate

Tabl. 1. Sadtler Database of Infrared Spectra- Spectral Library prediction results.

In order to predict the composition of the sample, the Sadtler Database of Infrared Spectra-FTIR Spectral Library was used as finding tool. The search revealed some potential compounds to be associated with this spectrum. The results are presented in table 1.

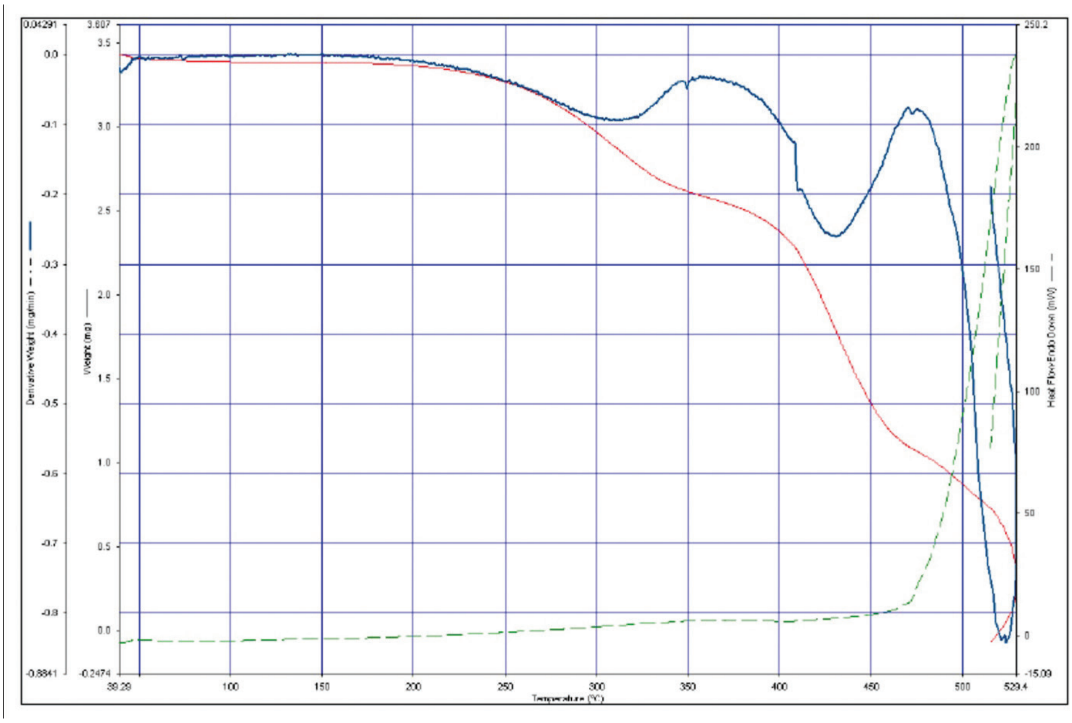

Fig. 2. Thermoanlaytical data (TG/DTG/HF) determined in oxidative atmosphere at a hating rate β = 10 °C/min.

After the prediction, the probability of containing one or more of the mentioned compounds in Table 1 is between 0.774 and 0.733. most of the compounds are naturally-occuring ones, especially esters (ricionoleates, acetates), but as well polyols and terpene derivatives (bornyl acetate, menthyl acetate), but as well steroid glicosides, that are generally found in numerous plants. All these information lead to the conclusion of an organic-type sample, probably a resin one.

As secondary investigational tool, thermoanalytical curves were drawn up (Fig. 2). The sample is stable up to 177 °C, when the mass loss occurs. According to TG/DTG curves, the mass is constant up to this temperature. The results confirm the lack of adsorbed water (generally eliminated up to 120 °C) on the sample surface, and confirm the FTIR data of the grafted OH moieties on the organic skeleton. DTG curve reveal three well-differentiated processes, the first in the 177–350 °C temperature range with DTG_{max} at 318 °C, the second process in the 350–471 °C temperature range with DTG_{max} at 429 °C, and respectively the third process between 471–530 °C, with a DTG_{max} at 521 °C. Heat Flow curve reveal solely an exothermic process between 460–530 °C, due to intense thermo-oxidations accompanying the final mass loss.

The fact that no thermal event was observed on the HF curve at lower temperatures is probably due to sensitivity of the thermobalance correlated with the small quantities of compounds in the sample.

Conclusions

The preliminary instrumental investigation study confirms the presence of organic compounds in the sample, mainly of natural sources (vegetal) as esters (ricionoleates, acetates), but as well polyols and terpene derivatives (bornyl acetate, menthyl acetate) or steroid glicosides. However, a complete composition can be realized after carrying complementary analysis like elemental analysis, mass spectroscopy, NMR and PXRD studies.

The use of certain organic binders for mending (sticking) the pottery is not a technique specific only to the LaTéne Age, such technique being encountered even since the Neolithic when bitumen[4] was used or during the First Iron Age, where in the settlement from Teleac a mixture of coal particles, “siliceous materials”, and a natural resin, more likely rosin found in the composition of the resin of conifers[5] was used for sealing the cracks of a vessel. Returning to the Dacian civilization, vessel wall perforation practice followed by “sewing” with organic or lead wires[6] is rather common.

Noteworthy is the fact that this vessel is the only container submitted to “mending” discovered so far in a “pit with deposits of pots” from Unip-*Dealul Cetățuica*. This repair contradicts to some extent the assumption of “deliberate destruction of the inventory” practices suspected in the case of the bowls with detached feet, the Dacian cup with its broken handle or the cups having their rims chipped[7].

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4 Dooijes, Nieuwenhuyse 2009.
5 Ciugudean 1994, 529.
6 Daicoviciu 1972, fig.41/1,2.
7 Berzovan 2013, 322–323; Berzovan *et al.* 2014, 17–18.

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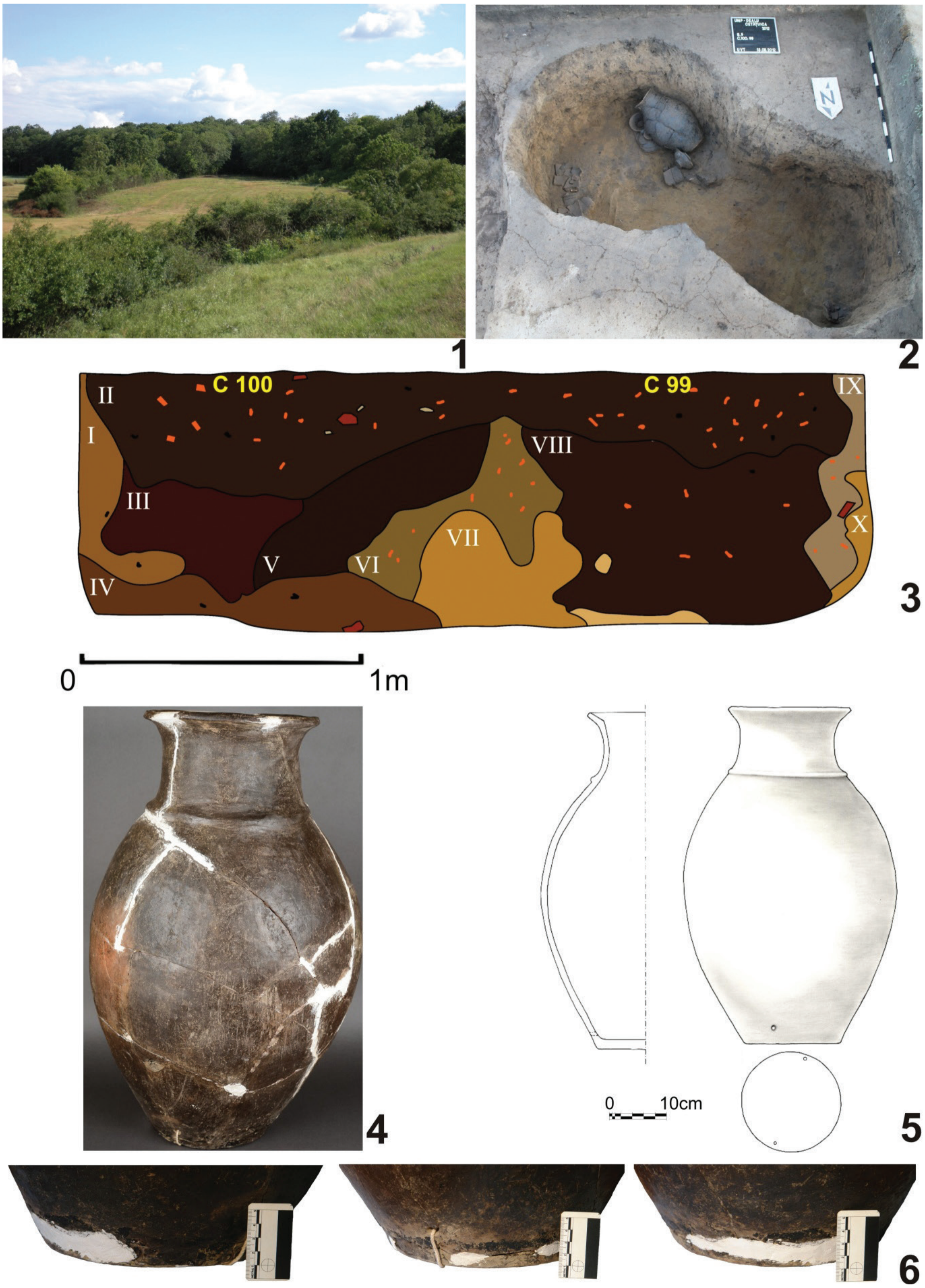

Plate I. 1. Situl de la Unip (vedere înspre sud-est); 2. Complexele 99 și 100 pe grundul suprafeței; 3. Profil longitudinal prin complexele 99 și 100; 4. Vasul supus reparației (foto); 5. Vasul supus reparației (desen); 6. Detaliu cu zona reparată.

Abbreviations

AAASH	Acta Archaeologica Academiae Scientiarum Hungaricae, Budapesta.
AAC	Acta Archaeologica Carpathica, Cracovia.
Alba Regia	Alba Regia. Annales Musei Stephani regis, Székesferhérvár.
Angvstia	Angvstia. Sfântu Gheorghe.
Arabona	Győri Xántus János Múzeum, Győr.
ArchÉrt	Archaeologiai Értesitó, Budapesta.
ArchHung	Archaeologia Hungarica, Series Nova, Budapest.
ArhMold	ArheologiaMoldovei. Iași.
Arheologija/Archeologiya	Arheologija/ Archeologiya. Sofia.
Apulum	Acta MuseiApulensis – Apulum. Alba-Iulia.
AMN	Acta Musei Napocensis, Cluj-Napoca.
AMP	Acta Musei Porolissensis, Zalău.
BAM	Brvkenthal Acta Mvsei. Sibiu.
BHAB	(Museum Banaticum Temesiense) Bibliotheca Historica et Archaeologica Banatica.
BMMK	A Békés Megyei Múzeumok Közleményei. Békéscsaba.
BMA	Bibliotheca Memoriae Antiquitatis, Piatra Neamț.
BMN	Bibliotheca Musei Napocensis.
BudRég	Budapest Régiségei. Budapest.
CCA	Cronica Cercetărilor Arheologice din România, București.
CRSCRCR	Coins from Roman sites and collections of Roman coins from Romania.
Dacia N.S.	Dacia. Revue d'archéologie et d'histoire ancienne. Nouvelle serie. București.
EphNap	Ephemeris Napocensis. Cluj-Napoca.
Ethnographia	Ethnographia. A Magyar NéprajziTársaságFolyóirata. Budapest.
FADDP/GMADP	Führer zu archäologischen Denkmälern in Dacia Porolissensis/Ghid al monumentelor arheologice din Dacia Porolissensis
FolArch	Folia Archaeologica. Budapest.
Hesperia	Hesperia: The Journal of the American School of Classical Studies at Athens. Athens.
MCA	MaterialeșiCercetăriArheologice. Bucharest.
MFMÉ	A Móra Ferenc Múz. Évkönyve. Szeged.
MFMÉ SE	Móra Ferenc MúzeumÉvkönyve: StudiaEthnographica. Szeged.
MFMÉ-StudArch	A Móra Ferenc Múzeum Évkönyve, Studia Archaelogica. Szeged.
MGTSZ	Magyar GazdaságtörténetiSzemle. Budapest.
MMA	Monumenta Avarorum Archaeologica, Budapest.
Mousaios	Mousaios. BuletinŞtiinţific al Muzeului Judeţean Buzău, Buzău.
NyJAMÉ	Jósa András Múzeum Évkönyve. Nyiregyháza.
OM	Orbis Mediaevalis, Arad.
PAT	Patrimonium Archaeologicum Transylvanicum. Cluj Napoca.
PBF	Praehistorische Bronzefunde. Berlin.
PeuceS.N	PEUCE. Studii și cercetări de istorie și arheologie. Serie nouă. Tulcea.
RI, SN	Revista Istorică, Serie Nouă. București.
RMM-MIA	Revista Muzeelor și Monumentelor. seria Monumente istorice și de artă. București.
SA	Sovietskaia Arheologija. Moscova.
SCIV(A)	Studii și Cercetări de Istorie Veche. București.
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WMMM	Wosinsky Mór Megyei Múzeum, Szekszárd
ZSA	Ziridava. StudiaArchaeologica. Arad.