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SHORT OBSERVATIONS ON THE POSSIBLE HYDRAULICITY OF VIMINACIUM LIME MORTARS BASED ON THE RESULTS OF LABORATORY RESEARCH

ABSTRACT

During the last decade, laboratory research of different types of ancient Viminacium lime mortars was conducted. It included research of core, bedding, pointing and rendering mortars with or without paintings, originating from the amphitheatre, the small structures beneath its auditorium and the city wall surrounding it, but also from the graves excavated near the Mausoleum central burial structure. Results of the research have led to a variety of discussions regarding the materials used for mortar preparation in Viminacium, but also about its preparation technology. One of the discussions was connected to the possible hydraulicity of Viminacium mortars.

KEYWORDS: VIMINACIUM, LIME MORTAR, HOT LIME TECHNOLOGY, HYDRAULICITY.

INTRODUCTION

During the excavations of ancient *Viminacium* - today an archaeological site and an archaeological park in Serbia near the confluence of the rivers Mlava and Danube and the town of Kostolac, once the capital of the Roman province of *Moesia Superior* and a very important legionary fortress on the Danubian limes, numerous graves and tombs, city baths, streets, roads, aqueducts with other water supply facilities, parts of the city and fortress' walls with gates, villas and suburban settlements, craftsmen's centres for the production of bricks and pottery, as well as the most monumental of all excavated buildings so far - the amphitheatre, were excavated.[1] The excavations have been followed by conservation processes and, for this, laboratory analyses of various types of mortars have been performed during the past decade.The laboratory research included samples of bedding, pointing and rendering mortar with or without paintings, originating from the amphitheatre (Fig. 1) and the small structures found beneath its auditorium (construction no.1 and construction no.2), core mortar coming from the city wall surrounding the mentioned building, together with bedding and core mortar from the graves excavated near the Mausoleum central burial structure in the eastern *Viminacium* necropolis named "Pirivoj" (Fig. 2).[2] The mutual comparison of the loss on ignition

1 The article results from the project *IRS - Viminacium, Roman city and military legion camp – research of the material and nonmaterial culture of inhabitants by using the modern technologies of remote detection, geophysics, GIS, digitalization and 3D visualization* (*no 47018*), funded by the Ministry of Education, Science and Technological Development of the Republic of Serbia.

2 The research of *Viminacium* mortars done in C.S.G. Palladio Laboratories, Vicenza, Italy, in 2007, included the analyses of lime mortars originating from the surrounding wall of the Mausoleum, and the walls of particular graves near it (Cornale, P. and Monni, E. 2007; the research has not been published). Mortar samples from the *Viminacium* amphitheatre, examined in the Institute for Testing Materials (IMS) in 2011, included lime mortars functioning

Fig. 1. Arena wall of the amphitheatre (documentation of the Institute of Archaeology Belgrade).

as bedding, pointing, and rendering mortars (with or without wall paintings), all coming from the arena wall of the amphitheatre and a small structure found beneath its auditorium (construction no.2, assumed to be a small shrine – aedicula), but also a sample of the lime mortar from the core of an outer city wall next to the building (results of the research done by IMS - Delić, Nikolić, I. *et al.* 2011, are partly published in Nikolić *et al*. 2016 and Nikolić and Bogdanović 2012). Samples examined in 2014 in IMS and Vinča Institute of Nuclear Sciences, University of Belgrade, were again those of rendering lime mortar from the arena wall, but also those taken from the small built structure found under the auditorium (construction no.1), all with wall paintings (the research was done for the purpose of Rogić 2014; research done by IMS - Vušović, O. and Ivović, B. 2014, has not been published, while the one done by Vinča Institute - dr M. Gajić-Kvašćev and V. Andrić, is part of Rogić 2014). The C.S.G. Palladio Laboratories research of bedding mortars included the analyses of mineralogic and petrographic composition with the determination of materials. The IMS research of bedding, pointing and rendering mortars without paintings included the analyses of the volume mass, water absorption, compression strength, porosity with the pore distribution, chemical composition, and mineralogic and petrographic composition with the determination of materials, while the research of lime mortars with wall paintings included mineralogic and petrographic composition with the determination of materials. The research of the lime mortars with wall paintings done in Vinča, included XRD analyses of different mortar layers with the determination of the sum of minerals in the samples.

values at different temperatures obtained by the laboratory research of *Viminacium* mortars, and the principles applied in the analogous research, indicated the possible hydraulicity of *Viminacium* mortars (Nikolić *et al*. 2016: 142) and raised questions as to how it could have been gained.[3]

HYDRAULICITY OF VIMINACIUM MORTARS

Hydraulicity of mortar is a feature that includes its water resistance and strength. Lime

3 Some of the results on the mentioned characteristics were published in Nikolić et al. 2016.

Fig. 2. Mausoleum (documentation of the Institute of Archaeology Belgrade).

mortars can achieve hydraulicity in a few ways: using a quarry sand with a high percentage of clay as an aggregate, natural or artificial hydraulic lime as a binder (using a limestone with impurities – natural, or adding certain materials to the limestone before or after its burning - artificial), or using certain materials of natural or artificial origin with pozzolanic features, as an addition to, or replacement for, the aggregate (Nikolić, Rogić and Milovanović 2015: 71-72).

The use of brick as an artificial material with pozzolanic features was most often the method for obtaining the better characteristics of Roman lime mortars in the territory of the present-day Serbia. *Viminacium* builders used brick as an additive in structures directly exposed to water and in humid or warm environments, but their structural mortars rarely contained this addition. (Nikolić *et al*. 2016: 142). However, the results of the laboratory research of the bedding mortar from the amphitheatre arena wall and the core mortar from the city wall surrounding the amphitheatre, without brick in the mixture, showed remarkable strength (Fig. 3) and a high percentage of silicon and aluminium oxides (Fig. 4). This led to the assumption that the builders could have added some natural aluminosilicate materials. However, based on the current understanding of available sources, they did not use any natural material with pozzolanic features. That is why attention is focused on some other methods of gaining hydraulicity. An increased strength and the percentage of silicon and aluminium oxides in mortars could also have been achieved by the introduction of impurities into the mixtures using hot lime technology or by using lime already containing impurities (Nikolić *et al*. 2016: 142).

chemical composition (% vol)							
method	**SRPS B.D8.205, B.D8.210**						
sample	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**Na_2O**	**K_2O**
bedding mortar	66.84	5.12	1.42	13.21	1.19	0.13	0.46
core mortar	53.38	5.15	2.25	20.92	1.08	0.36	0.48

Fig. 3. Physical and mechanical characteristics of the mortar samples from the amphitheatre (data taken and modified from Nikolić et al. 2016, 141, Table 3).

method	**EN 1015-10**	**EN 1015-18**	**EN 1015-11**	**mercury porosimetry**
sample	**volume mass (g/cm³)**	**water absorption (vol.%)**	**compression strength (MPa)**	**porosity (vol.%)**
bedding mortar	1.65	22.3	2.7	33.4
core mortar	1.86	11.7	5.3	24.7

Fig. 4. Results of the chemical analyses of the mortar samples from the amphitheatre (data taken and modified from Nikolić et al. 2016, 140, Table 2).

ORIGIN OF THE VIMINACIUM LIME

Analyses of lime mortars of certain ancient Greek buildings that were not prepared using materials with pozzolanic features showed their great strength, but also the usage of impure limestone (Elsen, Van Balen, Mertens 2012: 125). However, it is thought that ancient people could see the differences between pure limestone and that with certain impurities, but also that they thought the impurities lowered the lime quality (Adam, 1999: 128). In Great Britain, in spite of available impure limestone - with a high percentage of clay, Romans made lime mortars of non-hydraulic lime, and the hydraulicity of the mortars was achieved with the addition of artificial materials with pozzolanic features (Van Balen 2003: 82). From the Middle Ages, hydraulic mortars were intentionally prepared with the addition of materials with pozzolanic features, but also using clay-bearing limestone (Elsen, Van Balen, Mertens 2012: 130). Those impurities in limestone - clay (actually the presence of silicon and aluminium oxides - authors' comment), made the lime created from this limestone naturally hydraulic (Adam, 1999: 129).

The research to date has shown that local and regional limestones dominated as building stones in *Viminacium* (63% of the examined stone samples), while the other stones used were travertine and marble (Đurić, Kale and Rižnar 2018: 474-475). The Austro-Hungarian travel writer from the XIX century, Felix Kanitz, wrote that tertiary limestone quarried in the surroundings of Belgrade – today Višnjica, was exploited by *Viminacium* builders (Каниц 1987: 157). The area from Belgrade to Kostolac, along the Danube, can be connected with the deposits of crypocrystal

limestones, corallinacean limestone (Leitha limestone), sandy limestone, sandy calcarenites and oolitic limestones (Ivković 1975: 19-22), but also with Sarmatian sediments of clays (most often marly), sands, sandstones and limestones (Pavlović 1980: 42, 22-23; Ivković 1975: 20). Sarmatian limestones are present as building blocks in the arena wall of the *Viminacium* amphitheatre. Oolitic limestones from the mentioned area have "sparry" calcites in their composition (Ivković 1975: 20) which is interesting to mention because those calcites were found in the analyses of *Viminacium* bedding mortars from the graves in the zone of the Mausoleum. Also, sandy calcarenites of this area have cherts (Ivković 1975: 20), found in a rendering mortar with the paintings from the construction no.2, and in the core mortar from the city wall. It is also interesting that in this area Leitha limestones are often very marly (Ivković 1975: 20).

Following this short review, it can be assumed that during the exploitation of the limestone for the lime used in *Viminacium*, there was a chance that the clayish and marly sediments, which could have been a source of hydraulic lime, were also exploited. However, the intention of using impure limestones by *Viminacium* builders, to obtain better mortar characteristics, can not be presumed based on the current understanding.

HOT LIME TECHNOLOGY AND DIFFERENT ADDITIONS

In the visual examination of *Viminacium* mortars from graves excavated in the southern Viminacium necropolis named "Pirivoj", done by the conservators long before the first laboratory research, the presence of impurities was highlighted. The aggregate of these lime mortars was often river sand with the presence of soil, indicating that the sand that was not rinsed (Станојловић 1992: 58). In the laboratory analysis of a sample of the rendering mortar with paintings from the construction no.2, the presence of mud-alevrite interlayers was observed. The first possibility for the presence these impurities is that they got into the mortars accidentally during its preparation, as a result of the carelessness of the worker or the fast process of building. The second possibility is that the mortar came into contact with the earth-lime mortar used for the building of the wall structure on which it was applied (the structure was probably built very fast). The third possibility is that the impurities were put in the mixture on purpose. In all the cases, they could have given the mortar mild hydraulicity. However, it could happen only when the lime was slaked *in situ,* whilst using hot lime technology. The traces of organic fibres are also found in the samples of analyzed mortars from *Viminacium*, that is, in rendering and pointing mortars of the arena wall and rendering mortars with wall paintings of the construction no.1 and construction no.2 (Fig. 5). Their presence in mortars, especially renders and plasters, is often ascribed to the tendency for the improvement of the tensile strength (Elsen 2006: 1419), but they can also be introduced to the mixtures accidentally.

It was often the case in Roman buildings that baked limestone was transported to the site and slaked there with a small amount of water and sand (Adam, 1999: 128). This hot lime technology, when done properly, produces distinctly strong mortars (Moropolou *et al*. 1996: 158), with good adhesion of lime and sand (Balksten 2007: 7). In the analysed rendering mortar of *Viminacium* which was painted, from the construction no.1 and construction no.2), we can find traces of this process. It is presented with lime inclusions, often noted in historical structures as a consequence of the use of dry slaked lime (Callebaut et al 1999: 118-119), actually the use of lime not slaked enough, because of the simplification and speed of the process (Станојловић 1992: 58), or during the use of hot lime technology (Pecchioni, Fratini and Cantisani 2006: 258; Станојловић 1992: 58).

The impurities, during the slaking of the lime *in situ*, with the development of temperature,

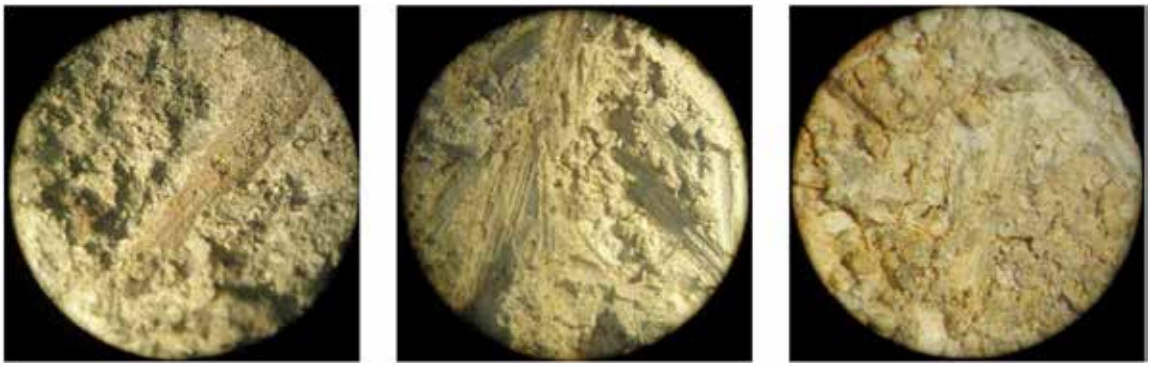

Fig. 5. Straw traces visible in the mortar samples from the amphitheatre (views under the laboratory magnifier): a. rendering mortar from the arena wall (Delić Nikolić, I. et al. 2011); b. pointing mortar from the arena wall (Nikolić *et al.* 2016, 139, Fig.1; Nikolić, E. and Bogdanović, I. 2012, 60, Sl.2); c. rendering mortar with wall paintings from the construction no.2 (Delić Nikolić, I. *et al.* 2011).

would react with lime, making artificial hydraulic lime, sometimes forming mortars with remarkable strength (Moropolou *et al.* 1997: 119, 129, 151, 159). The strength of the mortar samples taken from the walls of a monastery from Mount Athos, dated to the 16th century, was attributed to hot lime technology and clay additions. Here, the fine ground magnesium-alumino-silicate dust of the clays in the area was mixed with the *in situ* slaked lime and reacted as a pozzolanic addition (Moropolou *et al*. 1996: 151, 158-159). However, intentions in these processes are often hard to prove (Elsen 2006: 1419) and the properties of the particular clays are also very important (Pinheiro, Montenegro and Gumieri, 2010). In the case of *Viminacium*, the properties of the mud mortar used to build a brick kiln showed a total of the oxides SiO_2, Al_2O_3 and Fe_2O_3exceeding 70%,[4] and in one of them as high as 90,25% (Raičković 2012, tab. 8), indicating the pozzolanic features of the clay and the technology for making building materials from it (Nikolić, Rogić and Milovanović 2015, 76). It can be assumed that the *Viminacium* builders knew about the possibility of making mortar stronger by adding clays during the use of hot lime technology because they were well acquainted with brick making and, consequently, soil selection.

During the *in situ* slaking of lime, other minerals of aggregate could also have reacted as the addition with pozzolanic features when heated. Regarding the rock fragments found during the laboratory research of *Viminacium* lime mortars, mostly metamorphic and sedimentary rocks are present. Schist, created by the metamorphosis of volcanic rocks, can have a small amount of reactive silicon and will react with lime, giving a mild hydraulic feature to mortar (Henry, Stewart 2011: 58). Volcanic rocks are natural materials with pozzolanic features (Elsen 2006: 1419). In one sample of the bedding lime mortar from a Viminacium grave found around the Mausoleum, the presence of a volcanic rock is noted, in a percentage of 10% of the aggregate, This is very interesting because the zeolithised tuff is found around the settlements of Slanci and Veliko Selo, also near the Danube, a few kilometres from Višnjica (Ivković 1975: 39-40; Kašić *et al.* 2017, 51-52, 54-56). However their exploitation throughout history is unknown and modern exploitation has only just begun. As already mentioned previously, chert was found in a small amount in the rendering mortar with paintings originating from the construction no. 2, but also in the core mortar of the city wall. Granitoid was present in the pointing mortar and rendering mortar with paintings from the arena wall, and the core mortar from the city wall.[5] During the

4 In the Vienna and Belgrade area, it was exploited in the Roman times (Moshammer *et al*. 2015: 255; Bogojević 1968: 81, Бојовић 1977: 5-22).

5 According to international standards, the sum of

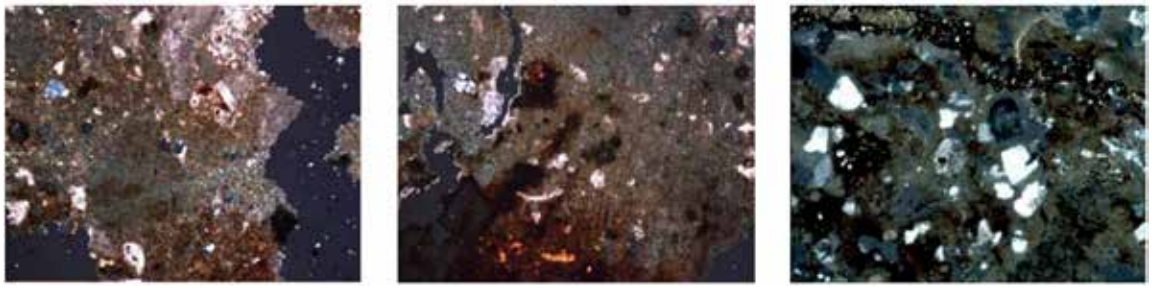

Fig. 6. Micro views of the mortar samples from the amphitheatre in which lime inclusions were found: a. b. construction no.1 (Vušović, O. and Ivović, B. 2014; c. construction no. 2 (Delić Nikolić, I. *et al.* 2011).

laboratory research of bedding mortars found in the graves found in the zone of the Mausoleum, a small amount of flint, which was mentioned by some authors as an admixture with possible pozzolanic features (Elsen, Van Balen, Mertens 2012: 129), was also found in many samples.

Viminacium is situated in the Kostolac lignite basin, one of the most important industrial areas in Serbia. However, the earliest written records of organised lignite exploitation in Kostolac date to the XIX century (Симић 1971, 75). By the 1870's, coal was exploited in very small quantities in Serbia. Considering the richness of the forests, it was not much needed until the second half of the century, when industrial complexes and the infrastructure were rapidly developing, also influencing the increased destruction of forests (Пејић, Јаношевић 1971, 62). We do not know if the Romans of *Viminacium* used the coal as a fuel, but it can be assumed that they knew about its presence, because the coal layers are very close to the surface in some parts of the area, and soil burnt by coal fires was used by them as a building material ("crvenka").[6] This can also be assumed from the presence of impurities, noted as "fragments of coal", visually observed in *Viminacium* mud mortars used for the building of the graves in the southern *Viminacium* necropolis (Станојловић 1992: 57), although they could be charcoal, left after the burning of the wood used as fuel. The remains of the wood and coal, used as fuels during the limestone burning, (Elsen 2006: 1419) are often present in ancient mortars. They may have originated in the process of the lime preparation, when quicklime was not sieved enough after the limestone burning (Callebaut *et al*. 1999: 118). Their ash, which could have been introduced to lime in this way, could have had slight a hydraulic feature (Henry, Stewart 2012: 57) The slag, visually observed in lime mortars with a small amount of lime and of lower quality, originating from the graves found in the southern *Viminacium* necropolises and used for the strengthening of the walls of the graves from their outer side (Станојловић 1992: 59) is, with charcoal, mentioned in literature, as a possible mortar admixture with pozzolanic features (Elsen, Van Balen, Mertens 2012: 129).

It is often very difficult to distinguish between the deliberate and accidental use of certain impurities found in the composition of mortar (Elsen, Van Balen, Mertens 2012: 129). However, their presence, together with other data acquired from the laboratory research of mortar samples, can offer precious conclusions about the way builders and artists prepared mortars for different functions in the structure.

percentages of the oxide content of SiO_2, Al_2O_3 and Fe_2O_3 in natural pozzolanic materials should be higher than 70% (ASTM C618-12a: 2012, Pinheiro, Montenegro, Gumieri 2010: 2).

6 Considering very small amount of granitoid and chert present in the sample of the core mortar from the city wall, its presence was omitted in Nikolić *et al*. 2016, 140, Table 1.

CONCLUSION

Results of the laboratory research of *Viminacium* mortars conducted until today have provoked different discussions regarding the materials used for mortar preparation, but also about the preparation technology.

It is very probable that, besides the deliberate hydraulicity achieved by the use of an artificial material with pozzolanic features - brick, and the possible use of some natural material with pozzolanic material, the hydraulicity of *Viminacium* lime mortars was very often produced by accident. It was done by using a natural hydraulic lime - created by burning impure limestone, or by the negligent introduction of different impurities to the mixture - which reacted as materials with mild pozzolanic features during the hot lime technology process. These impurities could have been: soil or mud with a clay component present in unrinsed sand; coal, wood, their ash, or charcoal present in the lime which was unsieved after burning; or different rocks present in the sand. However, they are mostly present in a very small percentage of the mortar mixture, and these assumptions have to be taken carefully and analysed separately in each particular mortar sample.

Concerns about the hydraulicity of *Viminacium* mortars and the ways it could have been gained can be partially removed only by future laboratory analyses of a large number of samples originating from various buildings and several historical periods of *Viminacium*, as well as by deeper research into the origin of the materials used for the preparation of these mortars.

NOTE

As the authors of the paper Nikolić E., Rogić D. and Milovanović, B. 2015 we use this opportunity to give two corrections. Page 80 - instead of: "In structural mortars, lower mortar layers of floor constructions and mortars for rendering, brick admixture is visible in the mortar structure in the form of smaller or larger fragments, (Fig. 11, Fig. 12) while in those mortars used for plastering, wall-paintings and finishing floor layers, the brick appears in the form of small fragments, but also in the form of a dust. (Fig. 13, Fig. 14)", it should be written: "In structural mortars, lower mortar layers of floor constructions and leveling layers of the mortars for the walls exposed to water, brick admixture is visible in the mortar structure in the form of smaller or larger fragments, (Fig. 11, Fig. 12) while in those mortars used for rendering and plastering – with or without wall-paintings, and finishing floor layers, the brick appears in the form of small fragments, but also in the form of a dust. (Fig. 13, Fig. 14)." Page 81 - instead of "Fig. 12. Rendering layers of Viminacium thermae with brick in the form of large fragments", it should be written: "Fig. 12. Leveling layer of the mortar on the wall exposed to water, with brick in the form of large fragments".

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REZIME
KRATKA ZAPAŽANJA O MOGUĆOJ HIDRAULIČNOSTI KREČNIH MALTERA VIMINACIJUMA ZASNOVANA NA REZULTATIMA LABORATORIJSKIH ISTRAŽIVANJA

KLJUČNE REČI: VIMINACIUM, KREČNI MALTER, HIDRAULIČNOST.

Tokom protekle decenije, sprovedena su laboratorijska istraživanja različitih vrsta krečnih maltera antičkog Viminacijuma, odnosno maltera za zidanje, maltera za malterisanje i dekorativnih maltera iz građevine amfiteatra, maltera iz jezgra gradskog zida koji je okruživao amfiteatar, i više grobnih struktura.

Rezultati ovih istraživanja, zajedno za vizuelnim opažanjima istraživača vezanih za maltere upotrebljavane u Viminacijumu, doveli su do razvoja različitih diskusija o poreklu materijala i tehnikama pripreme maltera u ovom rimskom centru, ali i o mogućoj hidrauličnosti ovih maltera i načinima na koji su ovu osobinu mogli dostići.

Vrlo je verovatno da je osim namerne hidrauličnosti dostizane upotrebom opeke, ili eventualnih prirodnih materijala sa pucolanskim svojstvima, ova osobina krečnih maltera nastajala često slučajno, upotrebom prirodnog hidrauličnog kreča nastalog od pečenja nečistog krečnjaka ili nemarnim uvođenjem nečistoća koje su tokom tehnologije spravljanja matera uz gašenje kreča na listu mesta, reagovale kao materijali sa blagim pucolanskim svojstvima. Ovi dodaci su mogli biti zemlja i mulj sa glinovitom komponentom, uneti u mešavinu uz neispran pesak, ugljen ili pepeo nastali nakon pečenja krečnjaka sagorevanjem uglja ili drveta, uvedeni u malter uz kreč, ili pak, neka stena koja je mogla biti nosilac blage pucolanske aktivnosti, a koja je bila deo peska. S obzirom da su ovi dodaci bili zastuoljeni u malom procentu, prethodne pretpostavke se moraju uzeti uslovno, odnosno proveriti na svakom pojedinačnom uzorku maltera.

Nedoumice vezane za hidrauličnost viminacijumskih maltera i načine njenog dostizanja mogu biti delimično otklonjene samo budućim laboratorijskim analizama uz veliki broj uzoraka, koji potiču iz različitih građevina i iz više istorijskih perioda antičkog Viminacijuma, kao i dubljim istraživanjima porekla materijala upotrebljivanih za pripremu ovih maltera.