Laboratory of Building Materials
Department of Civil Engineering
Aristotle University of Thessaloniki

Proceedings of the

4th Historic Mortars Conference

HMC2016

10th-12th October 2016, Santorini, Greece

Editors

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Published by
Laboratory of Building Materials
Department of Civil Engineering
Aristotle University of Thessaloniki

Thessaloniki, Greece, 2016

ISBN: 978-960-99922-3-7

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PREFACE

The Historic Mortar Conferences (HMC's) resulted from the need of dissemination of the latest information and advances in materials and techniques for the conservation of Heritage Structures. The HMC's have been established and supported by the core members of RILEM Technical Committees on Historic Mortars, Repair Mortars and Grouts during the last twenty years.

HMC2016 has taken place in Santorini, Greece, where one of the famous prehistory civilization, the Cycladitic one, was developed and destroyed by the eruption of the volcano in Thera, before 3300 years, covering with lava and rock fragments the island. This lava, an igneous and amorphous material, the Thera Earth has been used for centuries, from antiquity up to cement years, as natural pozzolan for producing lime-pozzolan hydraulic type mortars. Many monumental structures and great infrastructure projects, such as the Suez Canal (1866) have been erected with Thera Earth.

That is why the Laboratory of Building Materials of the Aristotle University of Thessaloniki, to which was given the task to organize the HMC2016, decided the venue to be in Santorini on Thera Island, a unique place of the world for its geomorphology and natural beauty.

The participants of HMC 2016 have exceeded 150, coming from more than 35 countries all over the world.

Special thanks are going to the Members of the Organizing Committee and the Scientific staff of the Laboratory of Building Materials AUTH for their dedicated work for the organization, as well as to the Members of the Scientific Committee for their great effort on reviewing the papers. All sponsors are gratefully acknowledged for their financial assistance and active support. Last but not least, I would like to thank all participants, chair persons and discussion contributors for the success of HMC2016.

Ioanna Papayianni
Professor AUTH
HMC2016 Chair

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Session IV

Analysis and characterisation of historic mortars

Roman Mortars from the Amphitheatre of *Viminacium*

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Abstract: Although the first archaeological excavations of *Viminacium,* the capital of the Roman province *Moesia Superior*, today's Serbia, began in 1882, only a small amount of laboratory research on applied building materials has been conducted so far. The grandeur and uniqueness of the building of the Roman amphitheatre now being excavated, and the need for its conservation, required the start of the research on the lime mortars used in this building. Some of the results of this particular research are presented in this paper.

Introduction

Viminacium is an archaeological site located in the villages near the confluence of the rivers Mlava and Danube. It is situated by the small town of Kostolac, in eastern Serbia, and lies on the territory of one of the biggest coal mines in the region. This Roman city and legionary camp was founded in the 1st century AD, survived through flourishing and destructive times, and finally fell as a result of the invasions of the 7th century [1].

Excavations of the Viminacium amphitheatre, the only Roman amphitheatre being excavated in present-day Serbia, started in 2007. The foundation of the amphitheatre is dated to the first quarter of the 2nd century and it was in use until the last years of the 3rd, or the beginning of the 4th century [2].

Laboratory research on the building materials of *Viminacium* started in 2002. For the purpose of three PhD dissertations, bricks, pottery and wall paintings were researched, [3, 4 and 5] while in 2015, travertine found in monuments was analysed [6]. The first analysis of mortars was done in 2007, in the C.S.G. Palladio laboratories in Vicenza, Italy. In 2011, the Institute of Archaeology, Belgrade and the Institute for Testing Materials IMS, Serbia, started researching the mortars from the amphitheatre, in order to begin the process of conservation.

Materials used in *Viminacium* lime mortars

The origin of the limestone used in *Viminacium* has not yet been determined. The 19th century Austro-Hungarian travel writer, Felix Kanitz, wrote about the tertiary limestone quarries in the Belgrade area, near the Danube, as the source of the *Viminacium* building stone [7]. According to the geological map [8], the mentioned area is connected with the deposits of Tortonian and Sarmatian limestone, but is also rich in sands and clays. Many of the limestone types are in transition between marl and pure limestone. It is well known that in the central zone of Belgrade, limestone [9] was exploited by the Romans [10]. However, it can not be excluded that the limestone from eastern Serbia area was transported by the river Mlava to *Viminacium*, although no archaeological evidence for this exploitation has been found. Recent research on the stone has shown that the *Viminacium* builders also used imported stones, such as Proconnesian marble [11] and travertine from the quarries near *Aquincum* [6].

Sediments of the *Viminacium* area consist of loess, sands, alevrites, pebbles, clays and coal [12]. In addition to lime mortars, builders often used mud mortars, and mixtures comprising different combinations of lime and earth [13]. The natural richness in clay materials resulted in large brick production in this Roman town [14], so brick and pottery were often used as mortar additives. The results of laboratory research showed that *Viminacium* bricks and pottery were mostly fired at temperatures lower than 900^{o}C, suggesting pozzolanic features. The overall sum of SiO_2O_3, Al_2O_3 and Fe_2O_3 is bigger than 70%, and the loss on ignition is lower than 10% for almost all the analysed samples [15, 16].

In one part of the wider Belgrade area near the Danube, zeolitic tuffs were found, [17] but there is no evidence of their exploitation through history. Material that can be called "natural brick", which has the common Serbian name "crvenka", and is internationally known as clinker and porcellanite [18, 19], was often used as a building element in the wall and road structures of *Viminacium* [20, 2]. It is visible as a reddish layer in the nearby hill over the former underground coal mine, and represents metamorphosed sedimentary rock, formed during the combustion of the coal layers underneath [18]. Although very rarely, it is found in some ancient Roman structures in Europe [21]. Several studies have determined the pozzolanic features of this type of material [22, 23], but only the research of local deposits can lead us further to the analyses of its possible use in *Viminacium* mortars.

Laboratory research

Ten mortar samples from the amphitheatre of *Viminacium* were chosen for the laboratory research in 2011 and 2014. The results of the conducted analyses on four samples of structural and protective mortars (Fig. 1) are shown here.

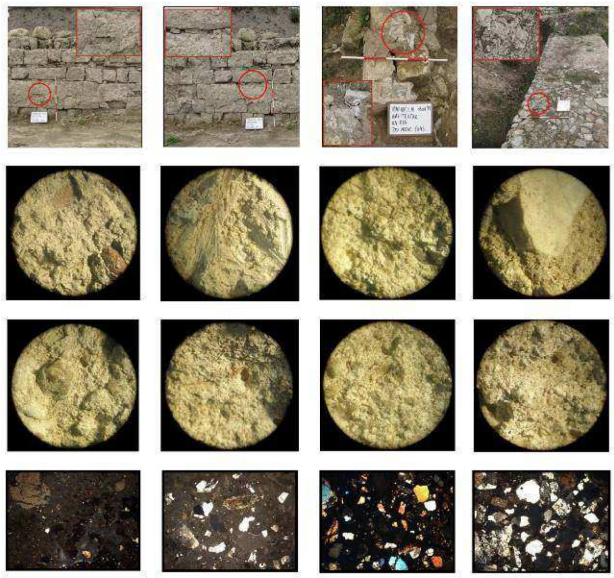

Fig. 1 Position of the sampling mortars and the results of the optical microscopy: views under the laboratory magnifier and micro views (using crossed Nicols, the field is 6mm wide)

The arena wall of the amphitheatre is preserved up to 4m and was built almost completely from limestone blocks bonded with lime mortar, with traces of collapse and repair in the debris. The wall was rendered on both sides, and the side facing the arena was painted. Unfortunately, the city wall surrounding the amphitheatre is preserved only in the foundations, which were built of schists and different stone and brick fragments, bonded with lime mortar. The research of the mortars included the analyses of the volume mass, water absorption, compression strength, porosity, pore distribution, chemical and mineralogical - petrographic composition with the determination of materials (Tables 1-3).

In the rendering mortar sample 039/11 from the arena wall, noticeable is an increased loss on ignition at 450^0C, which indicates an increased quantity of bound moisture and organic substances, in this case the remains of straw that directly influenced the high porosity and the water absorption of the sample. The content of Al_2O_3 comes from the feldspar and mica grains as well as from the fragments of bricks. One part of the CaO content comes from one of the feldspars (anorthite). This sample is characterised by a low volume mass, a high value of water absorption and porosity, and a middle value of compressive strength.

Table 1 Mineralogical and petrographic composition and the determination of materials

	AGGREGATE												
		composition (% Vol)											
sample	granulation (mm)	quartz	feldspar	pyroxene	mica	calcite	chert	granitoid	quartzite	schist	brick	limestone	B/A (Vol.)
039/11	0-1(98%)	94	5	-	1	-	-	-	-	-	-	-	80/
	1-10(2%)	-	-	-	-	-	-	-	50	-	50	-	20
040/11	0-1(95%)	94	3	-	2	-	-	-	-	-	1	-	75/
	1-4(5%)	-	-	-	-	-	-	50		-	50	-	25
041/11	0-2(93%)	90	5	2		-	-	-	3		-	-	65/35
	2-5(5%)	-	-	-	-	-	-	-	100		-	-	
	5-10(2%)	-	-	-	-	-	-	-	100		-	-	
042/11	0-4(87%)	84	5	-	1		10				-	-	70/30
	4-10(3%)	40	-	30	-	-	-	-	30	-	-	-	
	15-60(10%)	-	-	-	-	-	-	-	-	-	-	100	

While observing the pointing mortar sample 040/11 from the arena wall, remains of chopped straw were also found, which caused the high values of porosity and water absorption. A decreased loss on ignition at 950^0C determined a lower content of the carbonate binding component. The content of SiO_2 is noticeably higher than in the previous sample. The highest proportion of this oxide comes from quartz minerals, but the other parts are connected to other minerals. Although Al_2O_3 comes from the present feldspar and mica grains, as well as from brick fragments, its high content together with the high content of SiO_2 indicates that, during the preparation of the mortar, some other mineral aluminosilicate additive was used in a small quantity. The volume mass is higher than that of the previous sample, water absorption is lower, compression strength is still medium and porosity is high.

Table 2 Results of the chemical analyses

chemical composition (% vol)								loss on ignition	
method	SRPS B.D8.205, B.D8.210								
sample	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	950^0C	450^0C
039/11	16.50	2.79	1.18	43.05	0.95	0.33	0.46	33.72	5.80
040/11	40.25	4.67	1.18	29.17	0.59	0.21	0.45	22.70	3.01
041/11	66.84	5.12	1.42	13.21	1.19	0.13	0.46	10.73	3.23
042/11	53.38	5.15	2.25	20.92	1.08	0.36	0.48	15.82	2.02

Table 3 Physical and mechanical characteristics of the samples

method	EN 1015-10	EN 1015-18	EN 1015-11	mercury porosimetry			
sample	volume mass (g/cm³)	water absorption (vol.%)	compres sive strength (MPa)	overall pore volume (cm³/g)	porosity (vol.%)	middle pore diameter (nm)	specific pore area (m²/g)
039/11	1.10	42.4	2.4	0.34	45.8	1342	7.9
040/11	1.27	35.5	2.0	0.36	47.1	1950	6.1
041/11	1.65	22.3	2.7	0.20	33.4	74	14.1
042/11	1.86	11.7	5.3	0.13	24.7	5402	4.3

The bedding mortar sample 041/11 from the arena wall has a very low loss on ignition at 950^{0}C, as well as the content of CaO, which indicates a decreased content of the carbonate binding component compared to the previous two samples. The content of SiO_2 is higher compared to the quantity of the quartz minerals. One part of SiO_2 comes from other minerals, i.e. feldspar, mica, schist and pyroxene. However, the high content of SiO_2 and Al_2O_3 again point to a mineral additive of aluminosilicate composition, even in a higher percentage than in the previous sample. According to the available data, the percentage of this additive could even be 30%. The compression strength was higher in this sample, and the volume mass is noticeably higher than in the previous two samples (even 50% higher than in the first sample). Water absorption is noticeably lower (twice as low as in the first sample) and porosity and the overall volume of pores are decreased.

A notable characteristic of the city wall core mortar sample 042/11 is the presence of large grains in the aggregate. Limestone fragments (sized from 1.5cmx2cm to 4.5cmx6cm) are present in a percentage of 10%. The content of SiO_2 is much higher than the quartz mineral content, also determined by mineralogical and petrographic analyses. The content of Al_2O_3 is higher than in other samples (a little higher than in the third one). High contents of SiO_2 and Al_2O_3 again indicate that a mineral aluminosilicate additive was used, probably in a percentage of 25%. With this data, the physical and mechanical characteristics of the sample are correlated. Compression strength is high for lime mortars, which can also be the result of the presence of large limestone fragments. The first descriptions of *Viminacium* mortar were given in 1907 by Miloje Vasić, the first qualified archaeologist to excavate *Viminacium*, who wrote that a very hard mortar from a grave was made of lime, crushed, but not baked, limestone and grains of quartz in the form of pebbles [14]. The value of the volume mass is noticeably higher in this sample than in all the previous three. This characteristic is also high for lime mortars, along with the lowest value of water absorption. Correlating to this are the lowest overall pore volumes and the lowest volume percentage of porosity.

Discussion

From the presented laboratory results it can be concluded that all the mortars sampled were made as mixtures of a carbonate binder and fine natural aggregate which, according to the shape of the grains, was assumed to be of a river origin. The voids are present in 3% for the 039/11-041/11 samples, and 2% for sample 042/1. Samples 039/11 and 040/11 show a significant level of mutually similar characteristics, such as granulation, mineralogical and petrographic composition, the presence of straw and brick, as well as physical-mechanical characteristics (Table 3 and Fig. 2). A similar case is seen with the samples 041/11 and 042/11, where the aggregate content is higher, the grains are bigger and the SiO_2 and Al_2O_3 content is higher. (Table 2 and Fig. 2) The results of the porosity tests showed that, in the examined interval of pore size (7.5nm – 15000.0nm), sample 040/11 had the highest value of overall porosity, while the lowest is in sample 042/11(Fig. 2).

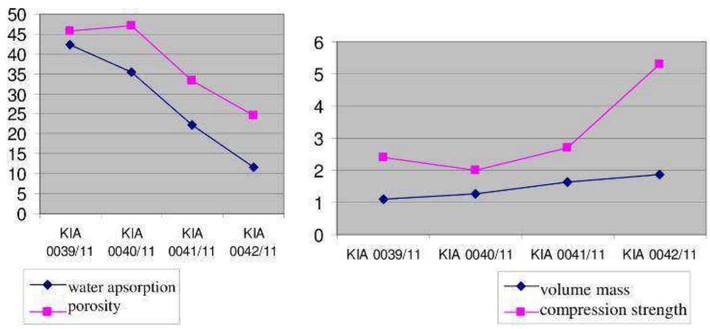

Fig. 2 Diagrams showing the correlated research values, mutually compared

The mutual comparison of the loss on ignition values at the temperatures of 950º and 450º obtained by this research, and the principles applied in the analogous research [24, 25, 26 and 27] can indicate the possible hydraulicity of *Viminacium* mortars. In the region of the present-day Serbia, the use of brick was the usual way for obtaining the better characteristics of Roman mortars. The *Viminacium* builders used brick as an additive in wall finishing and floor mortar layers [28], a practice expected in structures directly exposed to water and in humid or warm environments [29], but their structural mortars rarely contained brick [28]. The results of the laboratory research of the samples without brick in the mixture can lead us to the assumption that the builders could have added some natural aluminosilicate materials. Unfortunately, based on current understanding, they had no natural material with pozzolanic features for making mortars stronger. Strength and an increased percentage of silicon and aluminium oxides in these mortars could also have been achieved by the introduction of impurities into the mixtures whilst using hot lime technology or by using lime already containing certain impurities that the builders may not always have recognised. This can be further explored by means of research of the available limestone and the role of different additives and lime inclusions visible in many samples analysed during 2007, 2011 and 2014.

Conclusion

Provincial Roman architecture always depended on local resources. One of the problems in determining the natural resources of *Viminacium* is the coal exploitation, whose development is constantly changing the landscape.

The laboratory research of the lime mortars from the *Viminacium* amphitheatre showed that they were prepared in a variety of ways, depending on their role in the particular structure. This is most noticeable in the case of the city wall of *Viminacium,* which was built with the mortar of the highest compressive strength.

Future laboratory research of the natural materials that may have been used in the production of lime mortar, as well as research of a larger number of mortar samples from different monuments and periods of the seven century lifespan of *Viminacium*, with special attention given to their possible hydraulicity, will probably provide some answers to the questions this paper has raised.

Acknowledgments

The article is a result of the project: *IRS* - *Viminacium, Roman city and military legion camp – research of the material and non-material 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. Authors of the paper thank Ivan Bogdanović, a researcher from The Institute of Archaeology, Belgrade, for sampling the mortars and guiding the laboratory team through the archaeological site.

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