Formulating mortars for use in restoration practice

HUGHES, David C and STARINIERI, Vincenzo <http://orcid.org/0000-0002-7556-0702>

Published version

HUGHES, David C and STARINIERI, Vincenzo (2014). Formulating mortars for use in restoration practice. ZKG International, 2, 48-53.

Formulating mortars for use in restoration practice

DC Hughes[1] and V Starinieri[2]

[1] Professor of Construction Materials, Bradford Centre for Sustainable Environments, University of Bradford, UK, BD7 1DP
[2] Research Fellow, Materials and Engineering Research Institute, Sheffield Hallam University, UK, S1 1WB

ABSTRACT: The principal uses of Roman cement mortars in the field of restoration are for the production of cast decorative elements and renders. The formulation of these mortars differs in terms of mix proportions, workability and workable life. A typical specification for a "cast" mortar is 1:0.5 to 1:1 by volume, flow of 19.5 cm and a workable life in the range 15 – 30 minutes; a render mortar is typically 1:1.5 to 1:2.5 by volume, flow of 15.5 cm and a workable life of between 1 – 2 hours. Whilst the former workable life can be obtained by the use of chemical retarders the prolonged life required for renders is generally not possible without the excessive use of chemicals which impairs the performance of the hardened mortar and an alternative pre-hydration technique has been developed.

Keywords: mortar specifications, cement mortars, hybrid mortars, retardation, pre-hydration, citric acid, strength, water absorption coefficient, water vapour permeability, shrinkage.

1 Introduction

A general introduction to the historical development of Roman cements within a European context, their composition and hydration may be found elsewhere[1-6]. A key point to reinforce is that Roman cement is a generic name, albeit one much despised by some 19th century authorities such as Pasley, covering a broad family of commonly rapid setting cements. As with all "natural" hydraulic binders, the performance of the calcined Roman cement is principally determined by the mineralogy and microstructure of the original marl or coastal septaria as was commonly used in the UK. Thus, it is not possible to define a "typical" Roman cement or Roman cement mortar and a restorer should work closely with the cement supplier to obtain initial guidance in the use of any unfamiliar cement. This paper describes various properties of mortars produced with 3 cements, i.e. Prompt (Vicat, France), Gartenau (Institute of Ceramics and Building Materials, Poland) and Wietersdorfer (Wietersdorf and Peggau, Austria). It should be noted that two Spanish companies, Tigre and Cementos Collet, are also able to supply a range of Roman cements, with the latter also supplying in the USA.

A study of historical Roman cement facades in several European countries was conducted during the ROCEM project[1]. It was found that mortars possessed higher cement contents than might be considered today and that a wide range of aggregate type and grading was evident. The cement content was higher in mortars for cast elements than for renders; the latter might also contain lime. Subsequently, the following specifications (Table 1) were made for restoration mortars after input at workshops by representatives of the conservation industry including Remmers in Germany and Poland and Atelier Gurtner in Austria.

	Cast Elements	***Renders***
Mix proportions (by volume)	1:0.5 – 1:1	1:1.5 – 1:2.5
Flow (cm)	19.5	15.5
Workable life (mins)	15 – 30	60 – 120
Retardation	e.g. citric acid	DARC or citric acid

Table 1: Adopted mix proportions for investigation

The specification for the workable life is exacting. Roman cements typically set within a few minutes; this is a distinguishing feature when comparing European Roman cements and American Natural cements. Consequently, for most uses retardation of the setting is essential. In order to meet the specification for cast mortars, chemicals such as sodium citrate, potassium citrate or, most commonly, citric acid may be used at dosage rates of some 0.5% of the weight of cement. However, the specification for render mortars would generally require such excessive dosage of retarder that the performance of the mortar is severely degraded. A technique based on the pre-hydration of the cement with interesting consequences has been developed; the material has been called De-Activated Roman Cement or DARC. Latterly, it has been discovered that there is some historic basis for this approach although it is not known how widely practiced it would have been[7,8].

2 The DARC process

Full details of the research may be found elsewhere[9] and the following is necessarily a summary. The deactivation water is expressed as a percentage of the cement weight within any mortar and is typically in the range 7 – 10% depending upon cement source. In order to obtain a uniform distribution of the deactivation water amongst all cement grains the water is first added to the dry sand and mixed for 2 min at 62 rpm (a Hobart mixer was used for all mortar production). Subsequently, the cement is added to the wet sand and the whole mixed for a further 2 minutes at 62 rpm. The resulting free flowing mixture is then stored in an air-tight container for various periods (storage times) until required for preparation of the mortar. This process generates hydration products initially dominated by monocarboaluminate ($C_4A\dot{C}H_{11}$) which is augmented by carbonated AF_m as the storage period increases; these are the same aluminate phases produced in mortars with practical w/c ratios. Thus, it would be expected that early age strengths of DARC mortars would be low although this is not a problem for render applications. However, it has also been shown that the belite phases are not affected so long-term performance should not be compromised.

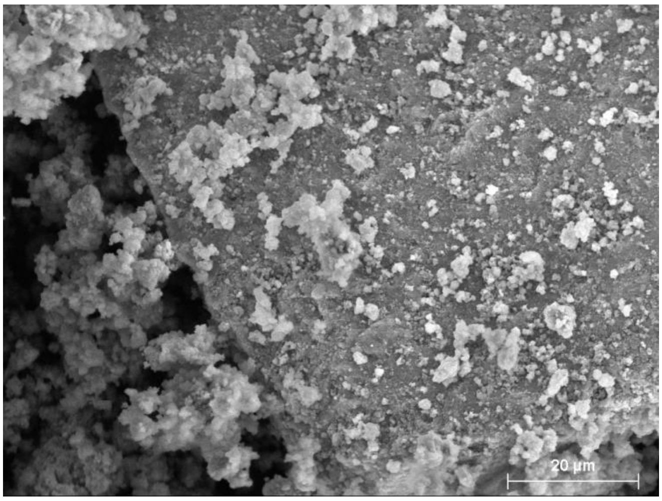

Fig 1: Surfaces of cement grain showing pre-hydration products

Fig 1 shows DARC (Gartenau cement) following de-activation with 10% water having been stored for 30 minutes. The packing density of the aluminate phases increases with both the amount of de-activation water and storage time.

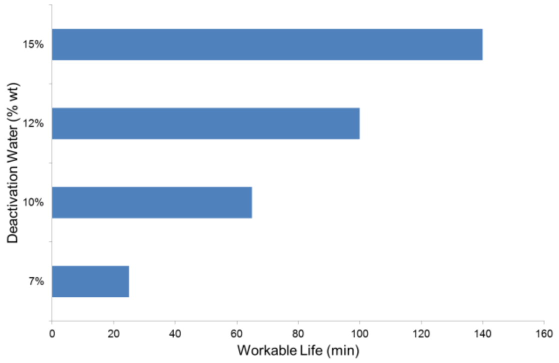

Fig 2: Influence of de-activation water on workable life

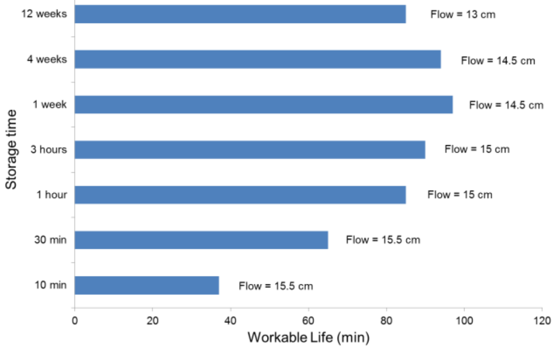

Fig 3: Influence of storage time on workable life

It is apparent in Figs 2 and 3 that the workable life is a function of both the amount of de-activation water and the time the dry mortar is stored prior to use on the façade. Thus, for a given cement the desired workable life may be tailored by a combination of these two parameters.

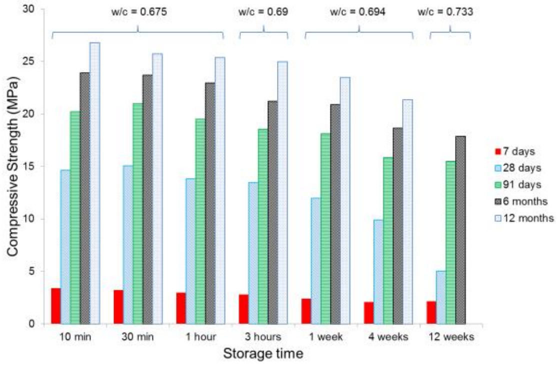

Fig 4: Effect of storage time on compressive strength of a DARC mortar after various ages of curing

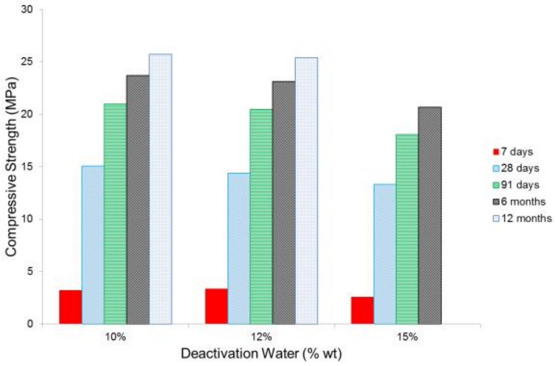

Fig 5: Effect of deactivation water (30 min storage) on compressive strength of a DARC mortar

Fig 4 shows that the strength of a DARC mortar is essentially independent of storage times of up to 1 hour but then decreases for longer storage times up to 12 weeks. Thus, for a site produced mortar the DARC process does not impact on longer term strength. Further work in the laboratory has shown that the addition of quicklime, to combine with excess de-activation water, prevents the reduction in strength associated with prolonged storage periods, so opening the way to factory-based production of ready-mixed mortars suitable for subsequent distribution and storage. Trials are currently under-way in Germany to produce such mortars. Fig 5 shows that whilst the strengths of the mortars with 10% and 12% deactivation water are similar, the strengths for the mortar with 15% de-activation water are significantly lower. However, it can be seen that the impact of deactivation water is lower on strength (Fig 5) than on workable life (Fig. 2): whilst the workable life of the mortar with 15% de-activation water is double that of a mortar with 10% de-activation water, its strength is only reduced by some 13% at 28 days curing.

It was frequently advised in the 19th century literature that Roman cement mortars should not be re-mixed once the onset of setting had been observed. However, that is not the case with DARC mortars. Figure 6 shows the raw data, for a render DARC mortar using Gartenau cement, from the EN Workable Life test which specifies a penetration resistance of 1.5 kg as representing the end of the workable life of a mortar. At this point the mortar was re-mixed without the addition of any extra water. It is apparent that this process has extended the workable life from approximately 50 minutes to some 140 minutes. Samples were manufactured immediately after both first mixing and subsequent re-mixing. Figure 7 shows that the re-mixed mortar has a slightly finer pore structure at an age of 7 days. The re-mixing does not affect the strength development of the mortar whilst only marginally increasing the drying shrinkage as might be expected from a finer pore structure[9].

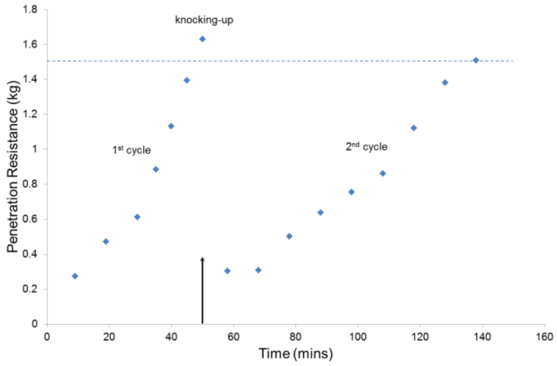

Fig 6: Workability of a re-mixed render mortar

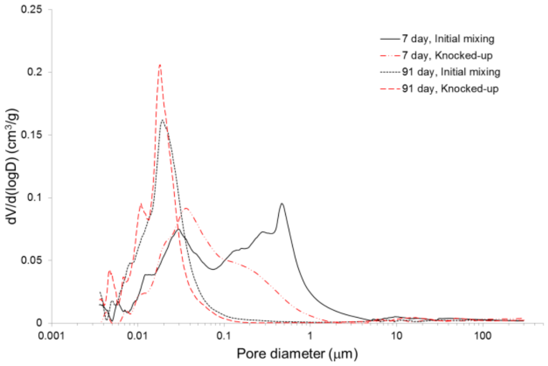

Fig 7: Influence of re-mixing process on pore structure

3 Mortar properties

3.1 Casting mortars

Mortars of volumetric composition 1:0.5 and 1:1 were manufactured using Vicat's Prompt and two ROCARE cements, i.e. W&P Wietersdorfer and MBM Gartenau requiring citric acid concentrations of 0.5%, 0.4% and 1.0% of the weight of cement respectively in order to achieve the target workable life. A carbonate sand was used for all mortars.

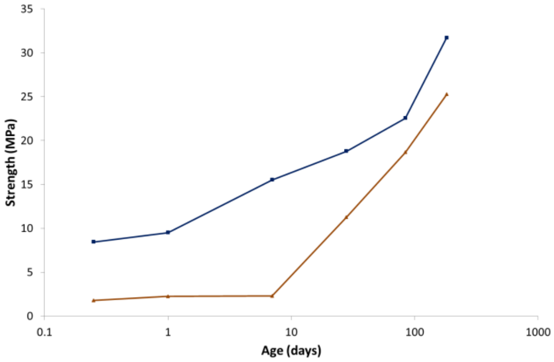

Fig 8: Strength envelope for 1:0.5 mortars

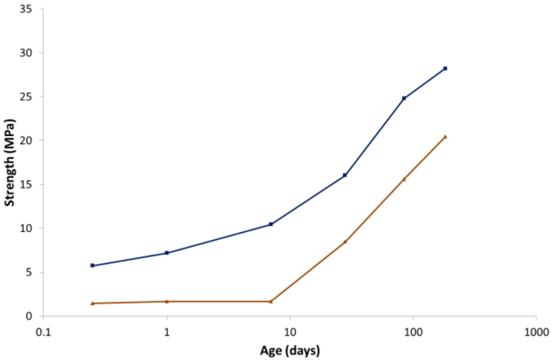

Fig 9: Strength envelope for 1:1 mortars

The actual mortar strength will depend upon cement, mortar formulation and content of citric acid. Figures 8 and 9 show the likely envelopes for the mortars tested. The w/c ratio depends upon the characteristics of the cement but a value within the range 0.45 – 0.55 is a good starting point for trials. The lower curve in each case is typical of many Roman cements which exhibit a dormant period lasting days or weeks before substantial hydration is observed. It is known that Gartenau cement is particularly susceptible to excess retarder concentration and trials with a 1:2.5 mortar have shown that increasing the concentration of citric acid from 0.5% to 1.0% may yield a strength reduction of some 66%. Thus, it may be possible to raise the upper envelope but only at the expense of a shorter workable life for a given workability and formulation. The early age strengths of the Roman cement mortars are higher than for both comparable PC and NHL5 mortars making them ideal for cast elements requiring stripping at an early age. By way of comparison, for the 1:0.5 mortars at an age of 28 days the PC mortar achieved a strength of 43 MPa and the NHL5 mortar achieved only 2.7 MPa, thus indicating the unique position of Roman cements within the continuum of hydraulic binders.

Breathability is an important yet poorly defined element of compatibility. It comprises the transport of both water and water vapour through the mortar's pore structure. The Water Absorption Coefficient and Water Vapour Permeability after 4 weeks curing in water are more sensitive to the cement used rather than mix formulation. The WAC is higher than the value of 2 $kg/m^2/hr^{0.5}$ considered critical for cast elements; the typical range may be 3 – 8 kg/m^2/hr$^{0.5}$. This range compares to 2 kg/m^2/hr$^{0.5}$ for Portland cement mortars and 25 kg/m^2/hr$^{0.5}$ for an NHL 5 mortar. The range of values of WVP is 6 – 17 x10^{-12} kg/m^2/Pa/s. This range compares to 3 x10^{-12} kg/m^2/Pa/s for Portland cement mortars and 24 x10^{-12} kg/m^2/Pa/s for an NHL 5 mortar.

Associated with the transport properties is the porosity which in the case of the Roman cement mortars lies in the range 22 – 32% with NHL 5 being higher at 34% and Portland cement much lower at 13%.

3.2 Render mortars

Mortars of volumetric composition 1:1.5 and 1:2.5 were manufactured using Vicat's Prompt and two ROCARE cements, i.e. W&P Wietersdorfer and MBM Gartenau and a carbonate sand. Whilst the Prompt mortars were retarded with 1.5% citric acid which would often be considered to be excessive, the ROCARE mortars were retarded using the DARC approach. It should be noted that we have not been able to successfully use the DARC process with Prompt and it has been suggested that this is because the DARC process is incomplete within a system which produces high water demand AF$_t$ rather than AF$_m$ as the initial hydration phase[5].

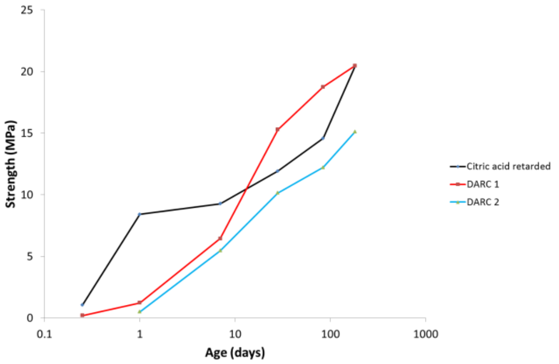

Fig 10: Strength for 1:1.5 mortars

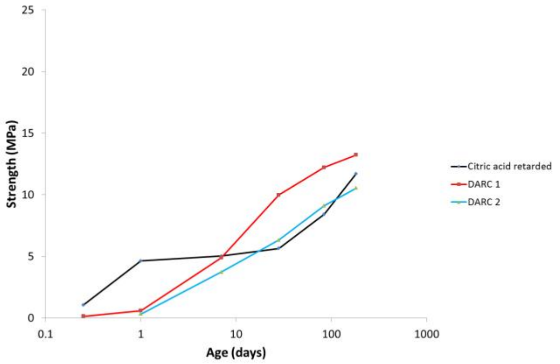

Fig 11: Strength for 1:2.5 mortars

Figs 10 and 11 show data for render mortars manufactured with 3 different cements in order to illustrate key features. The mortar retarded using citric acid required 1.5% citric acid to achieve the specified workable life. Whilst the strength after 6 hours is low, it has rapidly developed strength at 24 hours. However, the high dosage of citric acid has also retarded the hydration of the belite phases which then show an acceleration between 91 and 180 days. As expected, the DARC mortars possess low strength at 1 day; the DARC process produces the hydrates which would contribute to strength at this age which are then not able to be formed in the mortar. However, the strength then increases at a steady pace up to an age of 180 days when there is a narrow window within which the strength of all mortars is located. As previously observed for the casting mortars, mortars made with PC are much stronger than the Roman cement mortars and those made with the NHL 5 are much weaker; the strengths of the 1:1.5 mortars are 61 MPa and 6.1 MPa respectively at an age of 6months.

There does not appear to be a consistent trend between mortar composition and Water Absorption Coefficient and Water Vapour Permeability for all binder types after 4 weeks curing. The WAC is higher than the value of 4 kg/m^2/hr$^{0.5}$ considered critical for renders; the typical range may be 4 –10 kg/m^2/hr$^{0.5}$. This range compares to 2 kg/m^2/hr$^{0.5}$ for Portland cement mortars and 14 kg/m^2/hr$^{0.5}$ for an NHL 5 mortar. The range of values of WVP is 7 – 16 x10-^{12} kg/m^2/Pa/s. This range compares to 6 x10-^{12} kg/m^2/Pa/s for Portland cement mortars and 15 x10-^{12} kg/m^2/Pa/s for an NHL 5 mortar.

Associated with the transport properties is the porosity which in the case of the Roman cement mortars lies in the range 18 – 22% with NHL 5 being higher at 23% and Portland cement much lower at 11%.

3.3 Hybrid Render mortars

During the study of historical mortars[1] it was observed that render mortars often contained lime. Whilst contemporary handbooks[10-12] contain details of mortar formulations for various purposes no specification has been found for the inclusion of lime, e.g. lime type, cement/lime proportions or property development. It has been shown that the addition of lime in the form of both CL90 and NHL 5 does not increase the workable life of hybrid mortars[13]; consequently, retardation is essential. A study has been undertaken using both NHL 5 and CL90 as a partial replacement for Gartenau cement in a 1:1.5 render mortar. Two mix formulations were used in which the lime content formed 50% and 67% by volume of the binder phase.

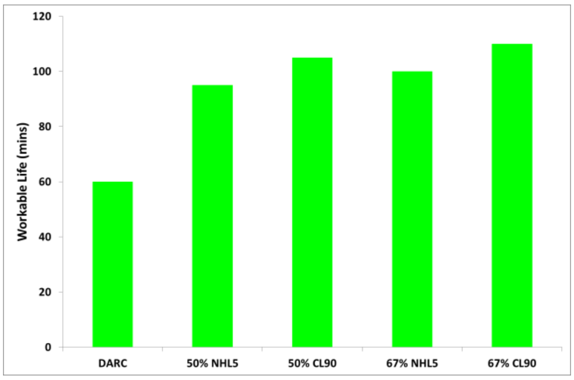

Fig 12: Workable life of hybrid mortars

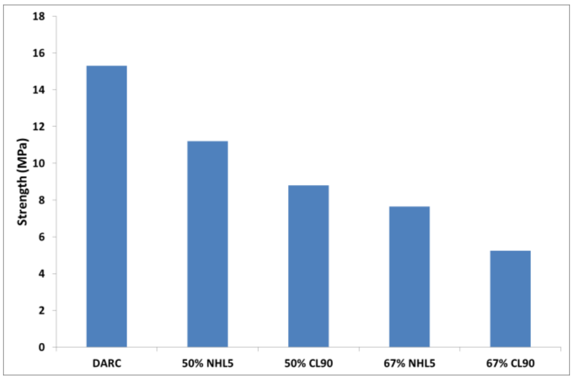

Fig 13: Strength of hybrid mortars

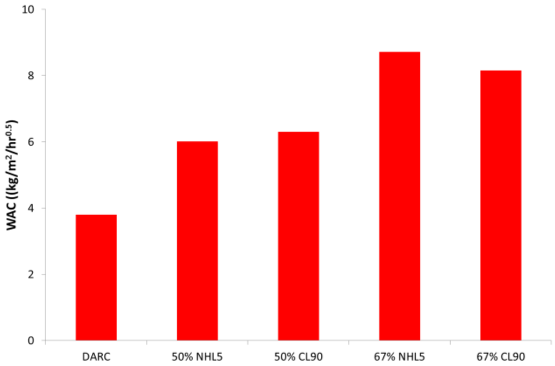

Fig 14: WAC of hybrid mortars

It is apparent the inclusion of lime to create hybrid mortars affects the performance of a DARC mortar in both the fresh and hardened states. The hybrid mortars exhibit longer workable lives than the original mortar although neither the lime type nor replacement level are significant factors (Fig 12). As expected, the use of lime decreases the mortar strength with 50% NHL 5 > 50% CL90 > 67% NHL 5 > 67% CL90 (Fig 13). The hybrid mortars possess higher values of water absorption coefficient such that it is possible to double the WAC in comparison to the original DARC mortar (Fig 14).

Further details on pore structure, shrinkage and the influence of curing regime on mortar properties are available elsewhere[13]. Shrinkage cracking is a characteristic feature of many historic Roman cement constructions. The susceptibility of such mortars to cracking has been comprehensively reported by Wilk *et al*[14,15].

The appropriate use of lime permits the refinement of mortar properties to achieve specified properties. If NHL is to be used then it is recommended that trials are conducted since they vary in performance just as do Roman cements since they reflect the characteristics of the limestone from which they were calcined.

4 Conclusions

The results of mortar testing clearly show that Roman cement produces mortars with properties different to those yielded by the use of either Portland cement or Natural Hydraulic Lime. The water transport properties are higher than those commonly required for restoration materials indicating a good basis for compatibility with existing products. Retardation is a key factor and a "de-activation process" is likely to give the best results for render mortars requiring a substantial workable life. This technique is not appropriate for mortars for cast elements since the early age strength is low and would prevent rapid demoulding. The use of lime to yield hybrid mortars permits the fine tuning of mortar performance across a range of properties.

5 Acknowledgements

The authors would like to express their appreciation of the support provided by the EU and the contributions of many colleagues from the ROCEM (Contract EVK4-CT-2002-00084) and ROCARE (Call identifier FP7-ENV-2008-1, Project number 226898) projects, particularly Brigitte Pestitschek and Blanka Kolinkeova.

REFERENCES

1. Weber J, Gadermayr N, Bayer K, Hughes DC, Kozlowski R, Stillhammerova M, et al. Roman Cement Mortars in Europe's Architectural Heritage of the 19th Century. In: Edison MP, editor. Natural Cement, STP 1494, West Conshohocken: ASTM; 2007, p. 69-83.
2. R. Kozlowski, D. Hughes, J. Weber, Roman cements - key materials of the built heritage of the nineteenth century, in: M. Bostenaru Dan, R. Přikryl, Á. Török (Eds.), Materials, Technologies and Practice in Historic Heritage Structures, Springer, Berlin, 2010.
3. Hughes, D., Swann, S., Gardener, A., Starinieri, V.: The History, Use and Analysis of Roman Cements. In Building Limes in Construction. Ed: Ian Brocklebank. Shaftesbury: Donhead Publishing Ltd. 107 – 130, (2012).
4. D.C. Hughes, D. Jaglin, R. Kozlowski, D. Mucha, Roman cements – Belite cements calcined at low temperatures, Cem. Concr. Res., 39 (2009) 77–89.
5. Gosselin C. Composition and hydration of some Roman (Natural) cements. Institute of Concrete Technology Yearbook 2013-2014
6. J.Weber, N. Gadermayr, R. Kozlowski, D. Mucha, D.C. Hughes, D. Jaglin, W. Schwarz, Microstructure and mineral composition of Roman cements produced at defined calcination conditions, Mater. Charact. 58 (2007) 1217–1228.
7. Chateau T. Technologie du Batiment, Tome premier, Librairie D'Architecture de B. Bance, Paris, 1863.
8. Prévost J. Les Travaux en Ciment, in Le Ciment de Vassy, Societe Anonyme des Ciments de Vassy, Paris, 1906.
9. Starinieri V, Hughes DC, Gosselin C, Wilk D, Bayer K. Pre-hydration as a technique for retardation of Roman cement mortars, Cement and Concrete Research 46 (2013) 1-13.
10. Bohnhagen A. Der Stukkateur und Gipser (1914). Leipzig: Reprint - Verlag Leipzig; 2003.
11. Issel H. Illustriertes Lexikon der Baustoffe (1902). Leipzig: Reprint – Verlag Leipzig; 2000.
12. Koch E. Gesammelte Erfahrungen über die Verarbeitung und die verschiedenen Anwendungen des Cementes aus den Cementfabriken von Ernst Koch in Hessen-Kallel und Hanau. Kassel und Leipzig: Kriegersche Buchhandlung Theodor Fischer; 1938.
13. Starinieri V, Hughes DC, Wilk D. Influence of the combination of Roman cement and lime as the binder phase in render mortars for restoration. Construction and Building Materials 44 (2013) 192–199.
14. Wilk D, Bratasz Ł, Kozłowski R. Shrinkage cracking in Roman cement pastes and mortars. Cement and Concrete Research 53 (2013) 168-175.
15. Wilk D., Bratasz Ł., Kozłowski R., 2011, Acoustic Emission for monitoring crack formation in Roman cement mortars [in:] Cultural Heritage Preservation. Proceedings of the European Workshop on Cultural Heritage Preservation, Krüger M. (ed.), Fraunhofer IRB Verlag: 177-181