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António João Cruz

Sub-Directoras_*Associate Editors*
Francisca Figueira
Maria João Revez

Marketing e Circulação_*Marketing*
Rita Horta e Costa

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Contactos para o envio de colaborações
Addresses for sending collaborations
António João Cruz
ajcruz@ipt.pt

Francisca Figueira
Instituto dos Museus e da Conservação
Rua das Janelas Verdes, 37
1249-018 Lisboa

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Editorial

Entre 24 e 26 de Setembro de 2008 decorreu em Lisboa, no Laboratório Nacional de Engenharia Civil (LNEC), uma conferência internacional intitulada *HMC08 – Historical Mortars Conference*. Esta foi uma iniciativa que resultou da conjugação das actividades desenvolvidas no âmbito de quatro projectos de investigação financiados pela Fundação para a Ciência e Tecnologia, nomeadamente os seguintes: *Cathedral – Caracterização e conservação de argamassas tradicionais históricas de edifícios religiosos do Alentejo*; *Desenvolvimento de metodologias para a avaliação dos efeitos da humidade em paredes antigas*; *Conservarcal – Conservação de rebocos de cal: melhoria das técnicas e materiais de restauro arquitectónico*; e *Estudo de argamassas compatíveis para a preservação do património edificado*.

Mais de uma centena de comunicações foram apresentadas na conferência por investigadores de um grande número de países. Distribuíram-se por quatro temas: *I – Caracterização de argamassas históricas*; *II – Degradação e diagnóstico de alvenarias históricas*; *III – História, salvaguarda e conservação de revestimentos históricos*; e *IV – Soluções compatíveis de argamassas para conservação*.

Tendo em conta o interesse e a qualidade das comunicações e o objectivo de lhes dar maior divulgação, a comissão organizadora da conferência considerou que seria vantajoso aproveitar as páginas de duas revistas – a revista *Conservar Património* e o *International Journal of Architectural Heritage* – para a publicação de algumas das comunicações apresentadas. Para cada uma das revistas, tendo em conta os respectivos interesses e características, foram seleccionados alguns textos que foram depois submetidos seguindo os habituais procedimentos de cada um dos periódicos. Assim, este número da *Conservar Património* é integralmente dedicado à publicação de sete comunicações dos temas I e II. O próximo número incluirá algumas das comunicações dedicadas aos temas III e IV. Os artigos foram escolhidos através do trabalho conjunto da comissão organizadora da conferência e da direcção da revista.

A ARP e a *Conservar Património* agradecem à comissão organizadora da conferência, muito especialmente à eng.ª Rosário Veiga, esta oportunidade de, através da publicação destes dois números temáticos da revista, ficarem envolvidas nesta conferência internacional sobre as argamassas com interesse histórico. Agradecem igualmente o apoio recebido para a sua publicação.

A ARP e a *Conservar Património* agradecem também aos autores das comunicações seleccionadas o seu interesse e a sua disponibilidade para submeterem à *Conservar Património* os respectivos artigos.

PS – Só é possível dedicar este número com data de Junho de 2008 a uma iniciativa que decorreu em Setembro do mesmo ano devido ao facto de a revista geralmente ser publicada alguns meses depois da data de capa.

Editorial

The HMC08 – Historical Mortars Conference took place in Lisbon at the National Laboratory of Civil Engineering (LNEC) between September 24-26, 2008. The conference was a direct result of the joint activities of four research projects, funded by the Fundação para a Ciência e Tecnologia, including: *Cathedral – Characterization and conservation of traditional and historical mortars from Alentejo's religious buildings; Development of methodologies for the evaluation of the effects of humidity in old walls; Conservarcal – Lime mortar conservation - improving repair techniques and materials on architectural heritage;* and *Study of compatible mortars for the preservation of built heritage.*

More than one hundred presentations were given by researchers from several countries, organized under four themes: *I – Characterization of historical mortars; II – Decay and diagnosis of historic masonry structures; III – History, protection and conservation of historical renders and plasters*; *IV – Design of compatible repair mortars.*

The quality and interest of the papers and the wish to make them more widely available led the organizing committee to consider some of them for publication in two journals – *Conservar Património* and the *International Journal of Architectural Heritage.* The texts were selected and submitted to each journal taking into account their respective editorial interests and their usual submission procedures. Thus, the current issue of *Conservar Património* presents seven papers from themes I and II. The forthcoming issue will feature selected papers from themes III and IV. The papers were jointly selected by the conference organizing committee and the journal's board of directors.

ARP and *Conservar Património* wish to thank the conference organizing committee, and particularly Engineer Rosário Veiga, for the opportunity of being involved in this international conference on historical mortars through the publishing of these two special issues, for which they provided invaluable support.

ARP and *Conservar Património* would furthermore like to express their thanks to the authors of the selected papers for their interest and compliance in submitting their papers to *Conservar Património*.

PS - Albeit dated June 2008, this issue refers to an event that occurred in September, that same year. This is possible because the publishing of the journal normally occurs a few months after the cover date.

Natural cement and stone restoration of Bourges Cathedral (France)

Cimento natural e restauro de pedra na Catedral de Bourges (França)

C. Gosselin
Laboratory of Construction Materials (LMC), Swiss Federal Institute of Technology (EPFL), Lausanne, Switzerland,
christophe.gosselin@epfl.ch

V. Verges-Belmin
Laboratoire de Recherche des Monuments Historiques (LRMH), Champs sur Marne, France,
veronique.verges-belmin@culture.gouv.fr

A. Royer
Institut National du Patrimoine, Paris, France,
amandine.royer@inp.fr

G. Martinet
Laboratoire d'Etudes et de Recherches sur les Matériaux (LERM), Arles, France,
g.martinet@lerm.fr

Abstract

Natural cement, also called "Roman cement", was invented at the end of the 18th Century and played an important role in the development of civil engineering works until the 1860s. More surprisingly, it was also used to restore historic buildings, such as gothic cathedrals. This paper deals with the mineralogy and the durability of natural cement, in the particular case of the Bourges Cathedral in France. This study illustrates the interest of this material particularly adapted in stone repair or substitution. Contrary to traditional mortars, the present samples are made of neat cement paste, revealed by the absence of mineral additions as quartz or carbonate sand. Several combined techniques (SEM-EDS, TGA, XRD) were carried out to determine the composition of the hydraulic binder rich in calcium aluminate hydrates. The raw marl at the origin of the cement production contains oxidized pyrites which consist in a potential source of sulphate pollution of the surrounding limestone. The exposition of the cement in urban environment leads to some weathering features as atmospheric sulphation. Finally a petrophysical approach, based on water porosity, capillary sorption and compressive strength, has been performed to demonstrate the durability and the compatibility of roman cement applied as a restoration mortar of historical building.

Keywords

Bourges Cathedral; durability; mineralogy; natural cement.

Resumo

O cimento natural, ou "cimento romano", foi inventado no final do século XVIII e desempenhou um papel importante no desenvolvimento de obras de engenharia civil até à década de 1860. Surpreendentemente, foi também utilizado no restauro de edifícios históricos, incluindo catedrais góticas. O presente artigo incide sobre a mineralogia e a durabilidade do cimento natural, no caso particular da Catedral de Bourges, em França. Este estudo ilustra o interesse deste material, particularmente adaptado à reparação ou substituição de pedra. Ao contrário de argamassas tradicionais, as presentes amostras foram executadas com pasta de cimento limpa, revelada pela ausência de adições minerais como areias de quartzo ou calcário. Foi utilizada uma combinação de diversas técnicas (SEM-EDS, TGA, XRD) para determinar a composição do ligante hidráulico, rico em hidratos de aluminato de cálcio. As margas cruas que originaram o cimento continham pirites oxidadas, que constituem potenciais fontes de contaminação por sulfatos para o calcário circundante. A exposição do cimento em ambiente urbano levou a algumas características de alteração como a sulfatação atmosférica. Finalmente, foi conduzida uma abordagem petrofísica, baseada na porosidade acessível à água, absorção capilar e resistência à compressão, para demonstrar a durabilidade e a compatibilidade do cimento romano quando aplicado como argamassa de restauro em edifícios históricos.

Palavras-Chave

Catedral de Bourges; durabilidade; mineralogia; cimento natural.

Introduction

Natural cements, sometimes called “Roman Cements” were discovered at the end of the 18th Century in England [1,2]. Those hydraulic binders, originating from the calcination of marl stones, were used principally for works where rapid set and waterproof properties were required. Their discovery spread quickly all over Europe, where several quarries were found out during the 19th Century. Their properties made these cements of great help in buildings construction, particularly in civil engineering applications, until the development of artificial Portland cements in the 1870s. A minor use emerged in the field of restoration of historic buildings because of specific properties of natural cement, as strong as stone, with a similar colour, but cheaper, at a time when restoration campaigns increased in scale. Some architects were also eager for experiments with new materials and techniques. Dating from the 13th Century and located in central France, the cathedral of Bourges is one of the best examples of the use of natural cements. The cements came from the first quarries discovered in France, in 1824 at Pouilly-en-Auxois and in 1830 at Vassy-les-Avalon (Burgundy). These cements and particularly the one from Vassy, were employed from 1824 to the 1860s, for basement waterproofing masonry, stones repointing mortar and sculpture repair [3].

The literature [4-11] reveals many scientific studies on the different materials employed in the 19th Century. Table 1 gathers several data on the chemical composition of different natural cements, compared to limes and Portland cements. From table 1, Vassy and Pouilly cements show a homogeneous aluminium oxide Al_2O_3 content close to 10 %, while the content of calcium and silica oxides (CaO and SiO_2) are more fluctuating, according to the production sites. The process of natural cement required the firing of marl stones at a temperature estimated between 1000 and 1100°C [3]. The variations in the oxides composition in table 1 may originate from the nature of local marl banks used for the cement production as well as from the quality of the firing process. The other French production sites provide natural cements with a constant composition in the main oxides. Whereas SiO_2 and Al_2O_3 contents are consistent with the Vassy and Pouilly ones, the significant difference resides in the CaO content, slightly lower for the other origins of natural cement. The European and US natural cements are characterized by a large dispersion in silica and calcium. These fluctuations between the European productions sites reveal an actual requirement of gathering and standardization of limes and cements composition, as initiated in France by Vicat, from 1818.

The literature mentions Portland cements as slow cements in comparison with natural cements, called rapid cements. The opposition in this nomenclature is linked to the setting time of the respective binders. Indeed, Portland cement setting time is controlled by the addition of a small amount of calcium sulphate (less than five percent weight of cement is substituted by gypsum). In contrary, natural cements are not added with calcium sulphate, implying a very quick set. By pursuing table 1, Portland cements differ from natural cements by the Al_2O_3 content, slightly lower in the first case (from 5 to 10 %) and, secondly, by a more controlled CaO amount (from 65 to 70 %) in Portland cement.

Research into natural cements as materials used in the monumental restoration is a new subject, because the use of such materials has been recently rediscovered and their historicity has been taken into account. At Bourges, the presence of natural cement on central and southern portals (figures 1 and 2) has been revealed the last decade [12, 13], during successive preliminary studies for the occidental facade restoration. In 2005, the French Laboratory of Research of Historical Monuments (LRMH) has worked out a scientific study of this cement

Fig. 1 Head and flower shape ornamentations made in natural cement, under a dais (cl. C. Gosselin 2005). In the centre, apparent iron bar shows the sealing system using plaster.

Table 1 Chemical composition of different hydraulic binders used for construction in the 19th Century, after [4-11]

Nature of hydraulic binder		Origin, plant and authors	Oxyde Composition (weight %)						
			CaO	Al_2O_3	SiO_2	MgO	FeO	Fe_2O_3	SO_3
Natural cement Vassy and Pouilly	1	Vassy-les-Avallon [9]	59.60	6.80	17.75	-	-	7.35	4.08
	2	Pouilly en Auxois [9]	49.60	10.00	26.00	-	-	5.10	0.69
	3	Vassy-les-Avallon [8]	56.60	6.90	21.20	1.10	13.70	-	-
	4	Vassy [5]	52.05	8.40	20.00	0.95	5.70	-	2.29
	5	Vassy-les-Avallon [10]	50.90	9.30	20.30	0.30	5.50	-	2.86
	6	Vassy-a [7]	52.69	8.90	22.60	1.15	5.30	-	2.65
	7	Vassy-b [7]	50.68	8.76	23.50	1.80	5.65	-	3.29
	8	Vassy-c [7]	44.12	7.00	24.80	2.08	4.80	-	2.94
	9	Vassy-d [7]	52.20	9.60	22.40	1.44	4.76	-	3.13
	10	Pouilly [7]	46.10	10.39	26.80	1.72	4.61	-	1.42
	11	Vassy (plant Dumarcet) [11]	48.06	10.14	20.26	0.90	-	4.44	2.87
	12	Vassy (Rotton) [11]	46.70	9.64	20.14	1.10	-	4.86	2.86
	13	assy (Faure) [11]	43.46	10.80	22.82	1.60		4.24	1.80
	14	Vassy (plant Millot) [11]	49.90	10.52	20.10	1.06	-	4.00	3.18
	15	Vassy (plant Voyot) [11]	46.04	9.66	21.16	0.97	-	5.12	2.58
	16	Vassy (plant Prevost) [11]	50.14	9.76	19.74	1.18	-	4.94	2.89
	17	Vassy (plant Bougault) [11]	49.46	8.76	19.70	0.86	-	4.98	3.31
	18	Vassy (plant Détang) [11]	42.34	12.32	26.52	1.54	-	3.92	1.34
Other French natural cements	19	L'Albarine (Ain) [4]	47.95	9.25	23.45	1.45	-	3.83	0.57
	20	Argenteuil (Seine-et-Oise) [4]	47.50	8.35	29.55	3.85	-	4.10	1.10
	21	La Bédoule (Bouches-du-Rhône) [4]	49.05	11.60	23.45	1.05	-	4.75	1.02
	22	Cahors (Lot) [4]	50.65	10.75	28.20	1.05	-	3.50	1.71
	23	Champréau (Yonne) [4]	52.05	8.40	21.00	1.00	-	5.10	2.04
	24	Chanaz (Savoie) [4]	46.50	8.95	232.5	1.60	-	4.15	1.14
	25	Chouard-Angély (Yonne) [4]	47.70	12.90	23.40	1.05	-	3.30	2.69
	26	Courterolles (Yonne) [4]	49.15	9.15	22.15	0.7	-	5.45	2.00
	27	Fresnes (Seine) [4]	46.05	7.95	29.05	2.80	-	3.75	0.90
	28	Guétary (Basses-Pyrénées) [4]	53.80	8.85	25.10	1.15	-	3.05	0.94
	29	La Valentine (Bouches -du-Rhône) [4]	47.85	10.85	24.55	1.60	-	5.20	1.31
	30	La Valentine La Méditerranée (Bouches-du-Rhône) [4]	48.60	12.50	29.10	1.70	-	4.65	1.55
	31	La Valentine Ile du Rocher Bleu (Bouches-du-Rhône) [4]	50.45	11.35	24.65	1.15	-	5.25	1.02
	32	Vimines (Savoie) [4]	49.70	9.50	25.50	2.55	-	4.35	1.14
	33	Warcq (Ardennes) [4]	48.70	4.10	27.40	0.65	-	3.75	0.78
	34	Boulogne [9]	49.29	9.58	28.02	2.58	5.73	-	0.42

Nature of hydraulic binder		Origin, plant and authors	Oxyde Composition (weight %)						
			CaO	Al_2O_3	SiO_2	MgO	FeO	Fe_2O_3	SO_3
European and US natural cements	35	Krienberg (D) [6]	58.38	6.40	28.83	5.00	-	4.80	-
	36	Sheppey (GB) [6]	55.50	6.96	25.00	1.73	-	6.63	-
	37	Tarnowitz (P) [6]	47.83	1.50	5.80	24.26	-	20.80	-
	38	Hausbergen (Alsace) [6]	58.88	7.24	23.66	2.25	-	7.97	-
	39	Medina Parker Nouveau [9]	43.45	5.60	19.50	13.95	-	12.15	0.65
	40	Zumaya (ES) [7]	33.04	7.82	30.80	0.93	-	5.13	2.37
	41	San Sebastien (ES) [9]	38.34	17.53	37.65	0.00	-	-	2.73
	42	Cumberland (US) [7]	36.12	10.66	29.40	2.42	-	5.37	1.55
	43	Rosendale (US) [7]	33.02	7.16	27.50	19.50	-	4.64	1.09
	44	Lehigh (US) [7]	48.24	9.12	24.10	2.42	-	3.18	1.34
	45	Kentucky (US) [7]	43.40	4.82	23.00	12.80	-	3.18	1.81
	46	Podolski, société Moscou [7]	40.15	0.00	16.66	18.19	-	6.84	1.83
Low hydraulic limes	47	Ablancourt (Marne) [4]	59.50	4.90	12.20	0.90	-	2.50	-
	48	Beaume (Côte-d'Or) [4]	60.90	1.75	13.85	0.70	-	1.35	0.16
	49	Les Côtes d'Alun (Haute Marne) [4]	61.70	5.65	13.35	1.43	-	2.75	0.16
	50	Malain (Côte d'Or) [4]	65.85	4.45	10.60	0.50	-	1.35	0.65
	51	Fresnes (Seine) [4]	59.00	5.80	11.63	0.30	-	2.05	0.73
	52	Les Ormes (Vienne) [4]	64.50	3.05	9.80	0.80	-	1.30	-
	53	Cardalou (Tarn) [4]	67.30	3.60	13.60	3.20	-	2.35	0.49
	54	Doué (Maine-et-Loire) [4]	63.85	2.90	14.35	0.80	-	2.20	0.33
	55	Echoisy La Grave (Charente) [4]	59.20	4.60	11.70	1.40	-	2.30	-
	56	Massay (Cher) [4]	64.75	2.40	10.30	0.85	-	1.45	0.73
	57	Les Pomets (Var) [4]	62.40	4.75	12.15	0.75	-	1.60	0.57
Hydraulic limes	58	Argenteuil (Seine et Oise) [4]	56.80	5.20	17.85	1.35	-	2.40	1.06
	59	Bar sur Seine (Aube) [4]	59.20	5.20	17.30	1.90	-	2.60	-
	60	La Bédoule (Bouches-du-Rhône) [4]	58.80	3.45	16.80	0.85	-	3.45	0.57
	61	Beffes (Cher) [4]	61.35	4.25	13.50	1.05	-	3.20	0.37
	62	Bougival (Seinet Oise) [4]	57.80	5.00	16.35	1.00	-	2.10	0.09
	63	Les Cordeliers (Vienne) [4]	60.60	6.25	13.65	1.45	-	2.35	0.78
	64	Cruas (Arcèche) [4]	65.80	2.00	21.60	0.35	-	1.25	0.12
	65	Saint Astier (Dordogne) [4]	62.25	1.35	21.85	1.05	-	2.83	0.41
	66	Saint Michel (Savoie) [4]	62.20	5.40	14.20	2.40	-	2.10	-
	67	Lafarge du Teil (Ardèche) [4]	63.76	1.72	23.13	0.97	-	0.73	-
High hydraulic limes	68	Albi (Tarn) [4]	49.40	6.30	19.60	5.75	-	2.60	0.29
	69	Bertaucout (Ardennes) [4]	47.85	4.20	19.35	0.60	-	3.45	1.31
	70	Chanaz (Savoie) [4]	54.85	7.85	18.10	1.30	-	2.50	0.78

Nature of hydraulic binder		Origin, plant and authors	Oxyde Composition (weight %)						
			CaO	Al_2O_3	SiO_2	MgO	FeO	Fe_2O_3	SO_3
	71	Charleville (Ardennes) [4]	47.85	4.55	21.85	0.75	-	3.75	0.86
	72	Chatenoy (Haut-Rhin) [4]	57.90	9.25	21.95	0.25	-	3.20	0.08
	73	Côtes d'Alun (Haute-Marne) [4]	54.00	7.75	16.30	0.30	-	1.65	2.49
	74	Sigonce (Basses-Alpes) [4]	55.65	3.70	20.80	0.25	-	1.95	0.94
	75	Virieu-le-Grand (Ain) [4]	56.10	5.75	19.90	1.50	-	2.70	0.82
French Portland cements	76	Argenteuil (Seine et Oise) [4]	57.90	8.50	24.50	1.50	-	4.25	0.65
	77	Bassin Paris (Seine) [4]	59.80	9.35	22.30	1.15	-	3.90	0.41
	78	Boulogne-sur-Mer (Pas-de-Calais) [4]	59.40	7.20	24.10	0.95	-	3.35	0.43
	79	Campbon (Loire Inférieure) [4]	51.80	9.65	18.75	12.95	-	3.80	0.49
	80	Charleville (Ardennes) [4]	51.30	4.70	23.65	0.25	-	4.35	1.39
	81	Chouard-Angély (Yonne) [4]	49.40	9.05	22.60	0.90	-	5.95	2.82
	82	Frangey (Yonne) [4]	63.70	7.53	21.61	1.22	-	3.17	0.50
	83	Marseille (Bouches-du-Rhône) [4]	53.00	7.60	19.90	1.20	-	3.60	0.65
	84	Saint-Banzille (Hérault) [4]	60.85	9.55	22.10	1.30	-	4.25	0.78
	85	Lafarge du Teil (Ardèche) [4]	59.10	3.25	25.70	0.95	-	1.40	0.24
	86	Tenay (Ain) [4]	53.25	6.90	24.30	1.70	-	3.85	0.57
	87	La Valentine (Bouches-du-Rhône) [4]	50.45	8.60	21.25	2.05	-	4.20	1.35
	88	Candlot [11]	63.70	8.30	19.50	0.74	-	3.30	0.51
	89	Sollier [11]	64.03	6.03	22.30	0.97	-	2.94	0.65
	90	plant A [11]	63.23	7.80	22.03	0.97	-	2.57	0.76
	91	Couronne [11]	64.33	6.26	21.70	1.10	-	2.47	0.71
	92	Darsy [11]	60.35	9.30	21.35	0.90	-	2.30	0.98
	93	Cambier [11]	63.05	7.80	20.75	0.75	-	3.03	0.61
	94	Delbende [11]	63.40	8.30	21.07	1.10	-	2.47	0.54
	95	Cie Nouvelle [11]	62.68	8.32	22.00	0.80	-	2.23	0.53
	96	plant B [11]	62.95	8.15	22.05	0.85	-	2.40	0.74
	97	Quillot [11]	64.00	8.30	20.00	1.65	-	2.15	0.29

applied in Bourges Cathedral [14, 15]. The characterization aimed at better understanding the composition and physical properties of the material, in order to select the most appropriate restoration product and procedure for both natural cement and adjacent stones in the Bourges monument.

Sampling and experimental techniques

Sampling

By the end of the 20th Century, many fragments of decayed stones and mortars were about to fall on the cathedral forecourt [12]. Due to long lasting and severe stone and mortar decay, many pieces of repointing mortar and sculptures made of natural cement were

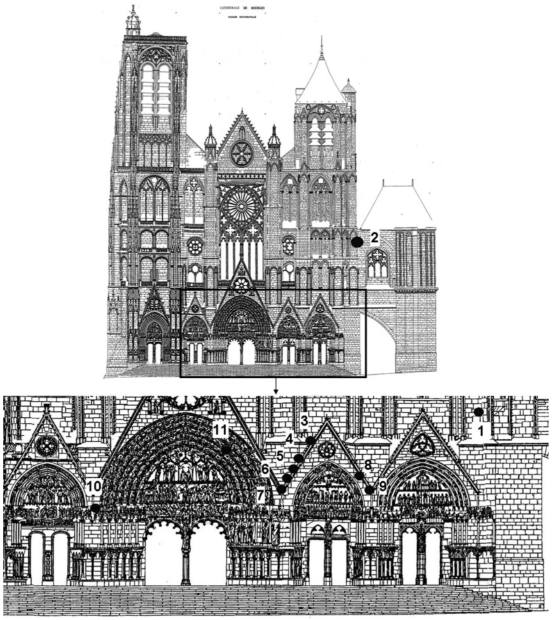
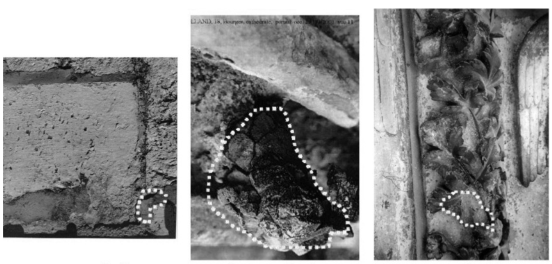


Fig. 2 Location of samples on the occidental façade (scheme from Société Française de Stéréotopographie 1969). The view of central and southern portals is detailed to locate the samples.



a: sample 1 b: sample 4 c: sample 11

Fig. 3 Different natural cement applications on the cathedral: a-stone repointing (cl. O. Rolland 2001), b-hook shaped sculpture (cl. O. Rolland 2001), c-leaf shaped sculpture (cl. V. Verges-Belmin 2003).

removed in 2001 within the frame of a safety removal campaign. The present 11 samples were selected amongst the pieces collected during this campaign and are located in figure 2.

Sample 1 (Fig.3-a) is a fragment of repointing mortar while samples 3 to 11 come from ornamentation sculptures standing on the third portal gable (Fig.3-b) or on the central portal vaults (Fig.3-c). Only one sample of limestone (sample 2) comes from a hook shaped sculpture.

Experimental techniques

The present experimental procedures are inspired from the literature on historical mortars characterization [16-19]. Preliminary phenolphtalein (concentration 1 %) was pulverized on freshly fractured samples to distinguish carbonated from non carbonated areas. The latter were then preferentially studied, in what concerned the mineralogy of the cementitious matrix, because their content of calcium carbonate is lesser [20].

Mortar macrostructure was observed on thin and polished sections, using natural or polarised reflected light optical microscopy (Leica DM) equipped with a digital camera.

The microstructure was observed by scanning electron microscopy (JEOL JSM 5600 LV) and analysed by electron probe microanalysis. Backscattered electron technique (Low Vacuum 17 Pa, 15 kV acceleration voltage) on polished sections was used for elementary chemical analysis. Secondary electron imaging, on carbon coated fresh fractures (High Vacuum, 25-30 kV acceleration voltage), provided high resolution images of the microstructure morphology.

Crystallized phases were determined by X-ray diffraction, using a Brücker D8 Advance diffractometer (100µm sieved fraction powder method, Cu tube, 2theta = 5-65°) with long time acquisition parameters (step size = 0.01°, step time = 10s, rotation speed = 10 rpm).

A complementary mineralogical analysis was performed by the LERM in Arles. This includes a chemical analysis of the acid soluble fraction (HNO_3 1:50) according to the protocol described in [18]. A complementary thermogravimetric/differential thermal analysis (TGA/DTA Netzsch), until 1000°C and under N_2 atmosphere is used. These coupled methods aim at determining the mineralogical composition of the mortar. The computation principle is based on oxides Bogue calculation and the results are expressed in weight percent of binder, aggregate and carbonated fraction [21]. This qualitative and quantitative approach, usually known as "Calcul Mineraux LCPC" method, was applied only on sample 8.

The separation of aggregates from hydraulic binder was performed using diluted (1:3) HCl acid etching [16]. After etching, the filtrate was rinsed with distilled water, dried and weighted before optical observation and XRD analysis.

Petrophysical characterization of cement and limestone has been comparatively done on prismatic specimens. The total porosity N_t, the 48 hours porosity N_{48} and the kinetics of capillary rise were measured according to the Rilem recommendations [22].

Finally, compressive strength tests were performed using an Instron 5500R hydraulic press, and managed at a controlled displacement rate of 0.5 mm/min.

In this article, the chemical formulas of phases are simplified using the cementitious notations for oxides: C: CaO; $\underline{C}$: CO_2; S: SiO_2; $: SO_3; A: Al_2O_3; H: H_2O.

Results

Macroscopic observations

Cement samples present common weathering forms, such as biological colonization (lichens, mosses) or black crusts, as a signature of rich sulphur urban atmosphere on calcareous materials [23]. Some samples present a network of deep cracks but no corrosion products have been observed, especially in the casting marks of the copper sealing rebars.

The samples present an original aspect and show a very fine, beige coloured and homogenous texture. In most cases a sub-millimetre thick brownish oily-aspect layer underlines the surface, as previously observed by Weber [24] on roman cement mortars sampled from European monuments. Traditional repair mortars contain mineral additives (sand, stone powder, pozzolans and/or tile fragments) but the matrix of our samples does not contain any of these coarse inclusions. Given these preliminary observations, all available specimens seem to come from the same restoration campaign. While the general texture looks similar for every sample, some nuances of colour are distinguishable in the matrix. After pulverising phenolphthalein solution on fresh fractures, this difference coincides with carbonated and non carbonated areas.

Microstructure

The microstructure has been investigated using optical and scanning electron microscopy on thin and polished sections. From optical examination, the matrix is composed of a binder including distinct and small inclusions. The porosity is defined by spherical and oblong pores with a large range of sizes, from 50 µm to 1 mm, and with a mean diameter estimated at 200 µm. The few microcracks, probably originating from preparation artifacts, are isolated and do not constitute a well defined network.

Optical microscopic exams are performed on polished sections etched by borax. This etching method reveals the major mineral phases like clinker grains not reacted with water during or after the mixing of mortar. Figure 4 shows two types of anhydrous grains of cement (50 to 200 µm size) present in the matrix. The first type of encountered cement grains, illustrated in Fig 4-a, is composed of blue to brown spherical and oblong particles in a white matrix. These spherical particles represent mainly dicalcium silicate grains (C_2S) contained in a white colored solid solution composed of calcium--aluminates such as tetra-calcium-alumino-ferrite (C_4AF) [25, 26]. The second type of anhydrous clinker grain is illustrated in Fig. 4-b. These grains are only composed of calcium-aluminates such as tricalcium-aluminates (C_3A, grey coloration) or tetra-calcium-alumino-ferrite (C_4AF, white coloration).

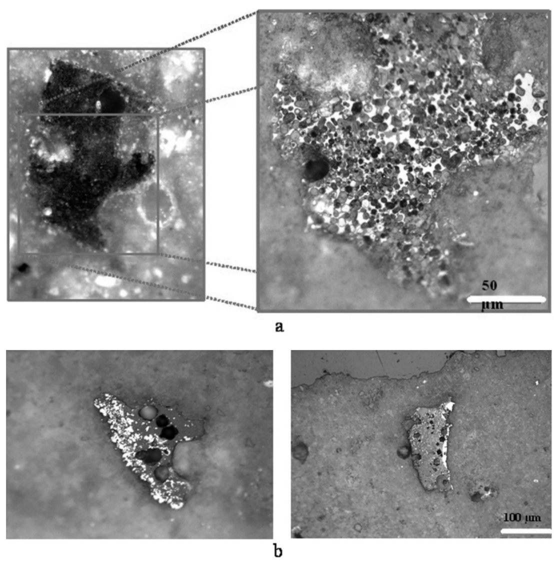


Fig. 4 Photomicrograph of anhydrous grains of clinker on etched polished section – a/:reflected natural light (left) and reflected polarized light (right) to show rounded C_2S grains in a (white) rich C_4AF matrix – b/ reflected polarized light view (grey) C_3A and (white) C_4AF rich grains clinker.

The rarity of clinker grains in the matrix shows a high degree of hydration of the cement. The heterogeneous nature and form of clinker grains could originate from a non optimized process of marls firing. Indeed, the kiln temperature must be constant to obtain a constant composition of clinker.

Analysis of aggregates

Optical microscopy examination of thin sections confirmed the naked eye observations on the absence of quartz sand or stone fragments in the mortar. As a complementary investigation, we proceeded to an acid etching of the bulk mortar that allowed separating siliceous inclusions from the binder. Filtrate residue (which represents

Table 2 Results of XRD semi quantitative analysis of carbonated and non carbonated (7, 8, in bold) samples: significant (+++), average (++), weak (+), trace (t) conformity of diffraction peaks according to reference file.

	Carbonated samples								Non carbonated samples	
	1	3	4	5	6	9	10	11	7	8
Calcite $C\underline{C}$	+++	+++	+++	+++	+++	+++	+++	+++	**++**	**++**
Vaterite $C\underline{C}$	++	++	++	++	++	++	++	++	**-**	**-**
Quartz SiO_2	++	++	++	++	++	++	++	+	**+**	**+**
Calcium silicate hydrate $C_2SH_{0.5}$	-	-	-	-	-	-	-	-	**++**	**++**
Hydrogarnet C_3AH_6	-	-	-	-	-	-	-	-	**++**	**+**
Hydrocalumite C_4AH_{13}	-	-	-	-	-	-	-	-	**++**	**++**
Gypsum $C\$H_2$	+	+	+	+	-	t	t	+	**++**	**+++**
Ettringite $C_3A.3\underline{C}\$H_{32}$	-	-	-	-	-	-	-	-	**+++**	**+**

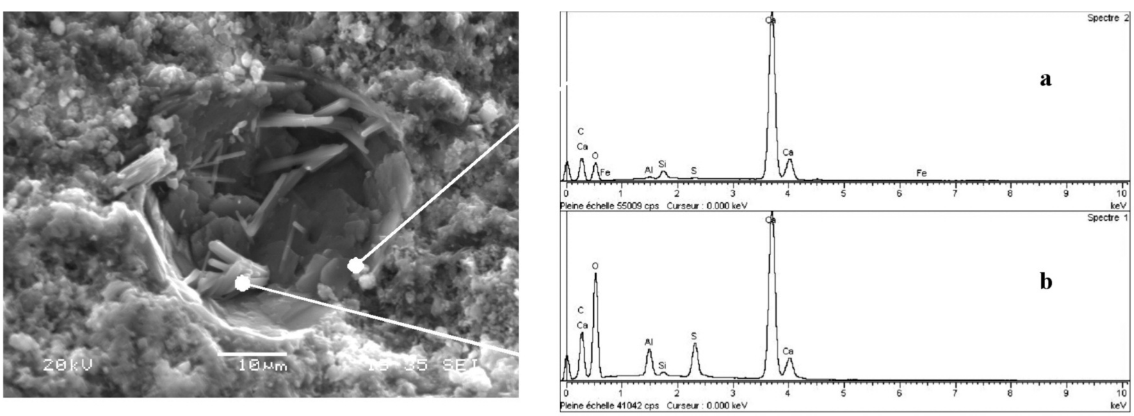


Fig. 5 SEM-EDS analysis of a pore recovered by portlandite (EDS spectrum a) and ettringite needles (EDS spectrum b).

12 to 15 % of bulk mortar mass) is composed of fine grains (5 to 10 µm) or grains groups (20 to 50 µm). XRD analysis gives the following crystallographic nature of these grains: quartz SiO_2, tridymite SiO_2 and goethite FeOOH.

XRD Analysis

The main crystallized phases of the samples are detected by XRD and the results are given in table 2.

As mentioned above, calcite and vaterite are the two major phases detected in carbonated samples. Among the main crystalline phases characterizing the binder, calcium silicate hydrate is a long term hydration product of C_2S, known to be slowly reactive. Then C_4AH_{13} is a member of hydrocalumite minerals and results from the hydration of calcium aluminates, such as C_3A grains observed on thin sections. This reaction is favoured in presence of Portlandite CH [27]. The latter seems to be weakly crystallized because undetected by XRD even within non carbonated samples. However, SEM-EDS examinations reveal the presence of CH in very small pores (Fig. 5).

Hydrogarnet C_3AH_6 is a stable phase corresponding to the complete thermodynamical conversion of C_4AH_{13}. Finally, ettringite and gypsum indicate a reaction between sulphates and the calcareous binder as commented below.

TGA/DTA results

The TGA/DTA results are given in table 3. This method allows the quantification of hydrated phases and calcium carbonate, for which the thermal decomposition corresponds to distinct ranges of temperature. In case of sample 8, 15.6 % of mass is attributed to hydrated phases (ettringite and calcium aluminate hydrates) and 12.7 % is related to calcium carbonate.

Table 3 Thermal analysis (bound water and decarbonation) of sample 8 and chemical analysis (expressed in wt. % of oxides) of its soluble fraction after nitric etching.

	Elements	% weight
Thermal analysis	Free water (<60°C)	0.2
	Bound water (60 to 550°C)	15.6
	from ettringite	*3.3*
	from hydrated aluminates	*6.1*
	Loss for 550 to 1000°C	12.7
	CO_2 from calcite	*11.6*
	corresponding calcite	*26.4*
	Total loss	28.4
Chemical analysis	SiO_2	13.83
	Al_2O_3	5.49
	Fe_2O_3	2.13
	CaO	40.87
	MgO	0.94
	SO_3	1.48
	P_2O_5	0.16
	Na_2O	0.04
	K_2O	0.10
	TiO_2	0.14
	MnO	0.19
	Cr_2O_3	0.01
	SrO	0.06
	Cl	0.01
	Insoluble fraction	6.15
	Loss ignition	28.40
	Total	100.00

Chemical composition

The nitric acid etching of sample 8 confirms that the mortar contains a low amount of insoluble fraction (7 %). The analysis of the soluble fraction, expressed in wt % of oxides is given in table 3. The high loss on ignition could confirm the high degree of carbonation but as well the high degree of hydration of the cement. By combining chemical and thermal results, a calculation is carried out to approximate initial mortar composition: hydraulic binder 60.2 %, siliceous fraction 7.0 % and carbonate fraction 30.5 %. Concerning the siliceous fraction, this composition corresponds to the preliminary observations of insoluble residue after HCl (1:3) etching and confirms the presence of quartz only in raw marl stones.

Concerning the carbonated fraction, results of mineral computation have to be taken into account carefully. Indeed, this method is limited for highly carbonated materials because the distinction between calcite from calcareous aggregates and matrix carbonation is impossible at this stage of investigation. Moreover, the calcareous fraction could also correspond to incompletely burnt marl fragments but no typical grains of calcite, from limestone aggregates, have been observed on thin sections.

Table 3 shows a high content of SO_3 in the binder. Sulphates are linked to the presence of ettringite and gypsum (table 2 and section 3-7) but also to unfired marl blocks or combustibles used for cement production. From table 1, among all French natural cements, Pouilly and especially Vassy cements contain a high amount of sulphate, with an average of 2.8 % SO_3, much higher than for French Portland cements (0.75 %) and hydraulic limes (0.48, 0.48 and 0.94 % respectively for low, medium and high hydraulicity).

Source of sulphates

The hydrated phases containing sulphur are significantly detected as gypsum and ettringite. Those two phases are secondary formation products coming from the effect of sulphur or sulphates sources on calcium and aluminium hydrates phases of cement. Gypsum is present in bulk mortar (XRD results) as well as on external subsurface (revealed by elemental EDX mapping of Ca and S, performed on polished sections where the external border was covered with a thin black crust). Ettringite is mainly present in the pores of the matrix and shows different degrees and forms of crystallization.

Concerning the internal sources of sulphur, several grains of pyrite (iron sulphur FeS_2) have been observed enclosed in the cement matrix. Pyrites originate from the raw marl stones used for the cement process. Figure 6 displays a grain of oxidized pyrite using optical microscopy (left) and the corresponding SEM image (right). X-Ray mapping illustrates the repartition of iron Fe, sulphur S and oxygen O in such oxidised pyrite grain. More generally, the pyrites observed under SEM present different degrees of oxidation.

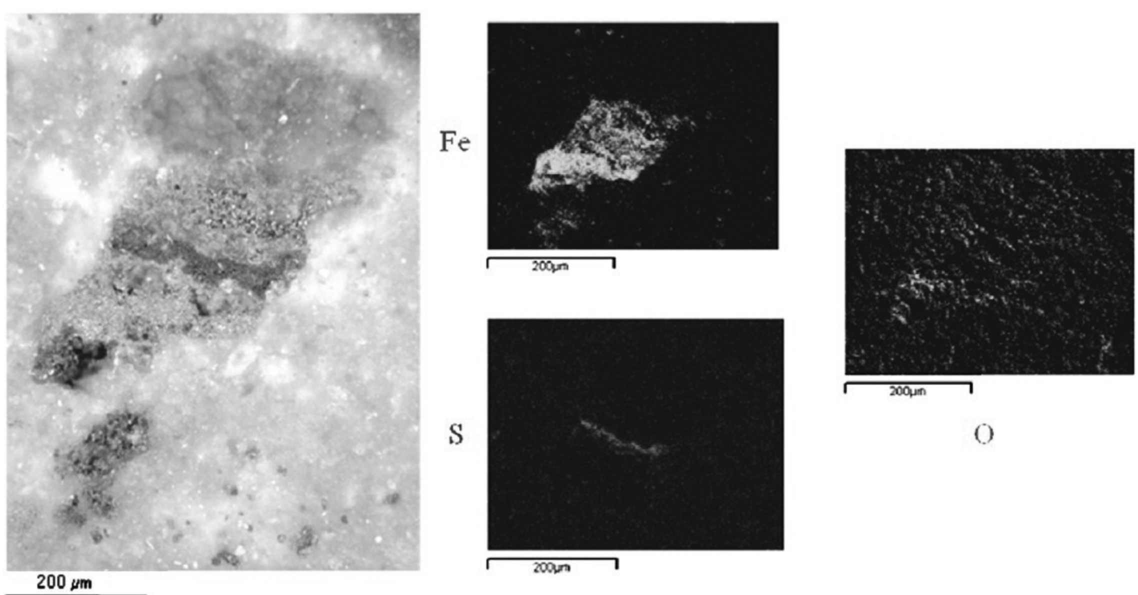


Fig. 6 Grain of oxidised pyrite: left/ reflected natural light OM exam to see rusty aspect of oxidised pyrite in cement – right/ X-ray mapping of the same grain to show repartition of iron, sulphur and oxygen.

Petrophysical and durability properties

Comparative petrophysical tests have been performed on cement mortars and limestone specimens to measure the porosity, the capillary sorption and evaluate the properties of fluid transfers within each material. This characterization evaluates the compatibility of natural cement applied as a repair material on a calcareous substrate. Table 4 gives results of cement and limestone samples presenting appropriated dimensions to prepare prismatic specimens.

The amount of water absorbed by a dried rectangular section of a sample in contact with a free water surface follows the relation $m= A.t^{1/2}$, where m is the amount of water ($g.cm^{-2}$) and t is time. The constant A is called water absorption coefficient ($kg.m^{-2}.h^{-1/2}$). Simultaneous measurement of the water level raised by capillarity is operated to determine the rate of water sorption following the relation $h=B.t^{1/2}$, where h is height of water uptake, B the water sorption rate ($g.cm^{-2}$) and t time. N_t and N_{48} are defined as the amount of water absorbed under vacuum respectively during 24 and 48 hours. The ratio between N_t and N_{48} is called Hirschwald coefficient S_{48}.

The solid density of all cement specimens is homogeneous, while the bulk density differs significantly. The fluctuations in the water/cement ratio used during the preparation of the cement paste are involved in the variation of the bulk density. Furthermore, the cement specimens present a higher total porosity (25-30 %) than the limestone (15-20 %) ones. Capillary sorption results reveal that the cement matrix presents a capacity (A) and kinetics (B) coefficients significantly higher than the calcareous substrate. This tendency is convenient in term of hygroscopic transfers between the two materials. High capillary sorption within the mortar avoids the water retention, especially at the stone/mortar interface and limits the risks of crystallisation of damaging salts.

Table 4 Capillary water uptake, water porosity, and density on limestone (†) and natural cement (*) specimens, complemented with compressive strength of cement sample 10.

Sample	Value A ($kg.m^{-2}.h^{-1/2}$) and *correlation* R^2	Value B ($cm.h^{-1/2}$) and *correlation* R^2	N_t (%)	N_{48} (%)	Hirschwald Coefficient (S_{48})	Bulk density ($g.cm^{-3}$)	Solid density ($g.cm^{-3}$)	Ultimate compressive strength (MPa)
1-1 *	-	-	28.62	20.10	0.70	1.80	2.52	-
2-a †	-	-	16.31	15.00	0.92	2.28	2.72	-
2-b †	1.07 (*R^2=0.9835*)	1.04 (*R^2=0.998*)	14.41	15.49	0.93	2.28	2.69	-
2-c †	0.45 (*R^2=0.9984*)	0.71 (*R^2=0.980*)	17.93	16.47	0.92	2.19	2.67	-
2-d †	0.78 (*R^2=0.9907*)	0.61 (*R^2=0.994*)	20.67	18.75	0.91	2.28	2.88	-
2 †	-	-	19.88	-	-	2.14	2.66	-
3 *	-	-	24.78	-	-	1.86	2.47	-
4-a *	-	-	27.24	-	-	1.80	2.48	-
4-b *		-	30.01	-	-	1.70	2.43	-
10-a *	8.56 (*R^2=0.999*)	2.32 (*R^2=0.989*)	49.86	49.10	0.96	1.32	2.63	15.34
10-b *	9.25 (*R^2=0.999*)	2.38 (*R^2=0.989*)	48.21	46.46	0.96	1.34	2.59	12.88
10-c *	9.03 (*R^2=0.998*)	2.50 (*R^2=0.994*)	47.24	45.36	0.96	1.39	2.63	15.50
10-d *	-	-	48.01	46.37	0.97	1.35	2.60	17.17
10-e *	-	-	49.23	47.20	0.96	1.33	2.63	-
11 *	-	-	27.46	-	-	1.78	2.46	-

Table 4 gives compressive strength results measured on the most porous sample of cement mortar (sample 10). The mean compressive strength is about 15 MPa with a low standard deviation (1.7 MPa). Comparatively, six months compressive strength has been evaluated in 1903 by the Laboratoire de la Ville de Paris [10] on different samples of Vassy cements coming from several plants (table 5). Those results were measured on neat cement paste (equal part of water and cement, without sand). By comparing the strength (17.6 MPa at 6 months and 15 MPa at around 160 years), we can conclude that the natural cement, mixed and applied in those conditions, would have passed through time without any loss of strength. This conclusion, based on petrophysical properties of a porous sample (50 %), could well be drawn to all natural cement specimens present on the façade of the edifice.

Table 5 Six months compressive strength results on Vassy cement paste coming from several plants, from [10].

Vassy cement Plant	Pure paste compressive strength (MPa)
Bougault	18.30
Dumarcet	20.50
Faure	17.58
Millot et Cie	16.58
Prévost	16.80
Rotton	17.00
Voyot	16.42
Mean	*17.60*

Discussion

At Bourges, the natural cement mortar is distinguished from traditional ones by the absence of mineral admixtures such as siliceous and carbonates aggregates. The insoluble residue size is too small for any addition of siliceous aggregates to be considered. This indication reveals a deficiency on the mix design. Indeed, the use of pure cement paste can promote successively thermal and mechanical shrinkage. This is confirmed by the archives mentioning a premature cracks network formed on the mortar surface, only two years after the end of the works [3].

The quartz and tridymite present in the insoluble residue led us to raise some questions on the temperature of marl stones calcination. During this process, α-quartz undergoes successive transitional phases: α-quartz at 573°C, (minor, short and unstable phase), tridymite (unstable phase between 867 and 1450°C) and cristobalite (stable phase above 1450°C) [27]. Moreover, according to literature, the natural cement process required marls firing at temperature estimated between 1000 and 1100°C [3]. Within this range of temperature, only tridymite would be present in the insoluble residue. The simultaneous presence of very fine grains of tridymite and quartz in the samples shows a heterogeneous firing of marl blocks. Consequently quartz originates from unfired marl fragments while tridymite is a residue of completely burnt stones. The presence of incompletely burnt marl stones reflects the heterogeneity of calcination temperatures, directly linked to weakly optimized process of firing. The use of shaft kiln was probably the main reason of such fluctuations in the cement production. From 1824, the development of Portland cements [28] has undergone similar defaults in the process of fabrication. Through the 19th Century, successive improvements in cement industry resulted, from the late 1870s, in the continuous production using rotary kiln and ball milling to grind the cement.

The main anhydrous phase of the cement is a bicalcium silicate C_2S, obtained for a temperature range of 900-1100°C. Above this temperature, tricalcium silicates C_3S would be the main reactant of the cement. The absence of C_3S confirms thus the data from the literature [3] on the cement calcination (1000 to 1100°C). At this range of temperature, the formation of C_4AF and C_3A depends on the sufficient amount of Al_2O_3 and Fe_2O_3 in marls before its calcination. According to the literature [4-11], the chemical composition of Vassy cement gives 9.20 % and 5.58 % respectively for Al_2O_3 and Fe_2O_3 amount, allowing the formation of significant amounts of C_4AF and C_3A in the clinker.

The determination of hydrated phases details the nature of raw marl used for the cement production. Vicat estimated a minimum clayey fraction of 27 to 30 % in marl stones destined to natural cement production [9]. In Bourges cement, highly argillaceous limestone is exhibited by a binder rich in calcium aluminates hydrates. C_4AH_{13} is clearly detected and partially converted into the stable form C_3AH_6. The binder rich in calcium aluminate confers to the material a high reactivity and rapid setting time. These properties are generally linked to a great heat of hydration and a consequent thermal shrinkage, as mentioned above. Additionally to calcium aluminate phases, poorly crystalline CSH implies the hydration of C_2S.

The chemical and mineralogical results retain attention on the high amount of sulphur in the cement samples. The influence of sulphur on Vassy cement has been notified from the earliest applied chemistry handbooks [4]. In 1885, Durand-Claye discussed the quality of those cements despite of their good hydraulic index – i.e. as defined by Vicat in 1856, the ratio between oxides from clay and lime fractions in raw marls, $i=(SiO_2+Al_2O_3)/CaO$. Earlier in 1857, Vicat [29] attributed the high sulphur content to accidental presences of calcium sulphate coming from sedimentary marls layers or combustibles used for cement manufacture. This initial sulphate content was qualified as a defect by Vicat and the concerned cements were avoided for marine structures, subject to aggressive saline environment.

In the present samples, the combination of sulphur and calcium on the subsurface results typically from a sulphation of hydrated phases (CH, C_4AH_{13}), anhydrous grains (C_3A), or calcium carbonate, by soluble atmospheric SO_2. The mechanisms of calcareous stones are well identified [23] but the atmospheric sulphation of mortars and concretes need more understanding. Recent studies [30] have explored the process of concrete sulphation by sulphur dioxide in urban and industrial sites. This type of sulphate attack in such conditions promotes mainly the formation of gypsum and ettringite in the porosity and the matrix of cementitious materials. Ettringite is thus a product of reaction between gypsum and anhydrous cement grains (such as C_3A and C_4AF) or hydrated calcium aluminates phases (C_4AH_{13}). Depending on the concentration of available sulphates and calcium aluminates, ettringite can expand with high pressure of crystallization generating internal mechanical stress. In particular conditions (location of ettringite growth, space availability), this pressure can lead to damage of the cement matrix.

Additionally to atmospheric soluble SO_2, plaster, applied to seal natural cement pieces [3, 12], is an external source of SO_3.

The oxidized pyrites implicate an internal source of sulphate, originating from raw marl to process the cement. Originally, the alteration of pyrites in marls can occur from a sedimentary process. This hypothesis would imply that goethite FeOOH, product of pyrite oxidation and identified in the acid insoluble fraction, should have been totally decomposed during the calcination stage and transformed into hematite Fe_2O_3 from a temperature of 250°C. Consequently, the presence of FeOOH corresponds to a secondary alteration occurring beyond the cement production or hydration. In this case, sulphuric acid released from pyrite alteration would react with calcium aluminates of the binder to produce gypsum and ettringite [31]. However, such reaction rims have not been observed in the surrounding of pyrite grains.

The two sources of sulphates (external from atmospheric SO_2 and internal from oxidized FeS_2) figured out in this study are potential sources of limestone pollution, when solubilised sulphates migrate to the calcareous matrix. Recent works [32], based on the dosage of sulphur and oxygen stable isotopes, are currently being carried out to characterize the different sources of sulphates in Bourges cathedral samples (healthy or decayed stone, plaster, roman cement). This isotopic approach aims at comparing the sulphatic signature of cement with the sulphates present in decayed stones, in order to evaluate the potential of pollution of natural cement on the edifice stones.

Although the natural cement used in Bourges constitutes a potential source of sulphates due to the high level of pyrites oxidation, its compatibility with limestone has been clearly demonstrated. The high porosity and the possibility of water evaporation characterize the natural cement as particularly adapted to substitute and repair the stones, in respect with proper fluids transfers and prevention of sulphate salts crystallisation.

Conclusions

This article gives new results on the characterization of natural cements produced in France during the 19th century. Their special application in monumental stones restoration is studied by means of mineralogical and petrophysical approaches. Through the determination of the cement composition and durability properties, the knowledge on natural cement process (nature of clayey marls stones, calcination temperature) and the state of art of stone restoration is enhanced. As the most of calcareous materials exposed in urban environment, natural cements undergo atmospheric sulphation leading to it superficial weathering (black crust). The several grains of oxidized pyrites have been identified as an internal source of sulphate. The soluble sulphates can react with calcium aluminate phases of the cement to form ettringite and gypsum. A secondary reaction involves the soluble sulphates migration through the cement to the calcareous substrate, leading to potential damage of the edifice stones. On one hand, the salt migration could be promoted by the high porosity of the cement matrix. But on the other hand, the high porosity and capillary transfer of the cement allow the evaporation of water and the crystallization of salt at the interface cement-stone. This property, complemented with consistent long term compressive strength, demonstrates the durability and the compatibility of natural cement to restore monumental stones.

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Development of lime based mortars for repairing glazed tile coatings of historic buildings in the city of Ovar, Portugal

Desenvolvimento de argamassas de cal para reparação de revestimentos azulejares de edifícios históricos da cidade de Ovar, Portugal

B. Teixeira
Civil Engineer, University of Aveiro, Aveiro, Portugal, miguelteixeira@ua.pt

C. Valente
Civil Engineer, University of Aveiro, Aveiro, Portugal, carla.valente@ua.pt

A. L. Velosa
PhD/Auxiliar Professor, University of Aveiro, GeoBioTech, Aveiro, Portugal, avelosa@ua.pt

M. R. Veiga
PhD/Main Investigator, National Laboratory of Civil Engineering, Lisboa, Portugal, rveiga@lnec.pt

I. M. Ferreira,
Msc/Archeologist, Atelier de Conservação e Restauro do Azulejo (ACRA), Porto, Portugal, isabel.feliz3@gmail.com

Abstract

Portugal is one of the European countries in which built heritage is a testimony of its history. In this context, the legacy of the decorative glazed tile coatings of facades must be preserved and restored.

This research is dedicated to the conservation of such facades in the city of Ovar, considered an example due to its rich heritage in glazed tiles, a high percentage of which requires a deep intervention. Therefore, this work is focused on the study of lime renders serving as a support for this type of tile facades. For this, samples were collected from several buildings in the city, targeting their mechanical and physical study with the aim of producing compatible mortars to be used for application of detached tiles in these buildings and generally for the repair of the facades with glazed tile coatings. For this purpose, four lime mortar formulations with different volumetric ratios were composed. The aggregates used were: ordinary river sand and local gravel. In three of the mixtures, metakaolin was added, with the intention of acting as an artificial pozzolan and thus improving the performance of these mortars. The use of a pozzolanic addition promotes hardening of lime mortars in cases when the ingression of carbon dioxide is low as is the case of mortars placed below glazed tile coatings.

These mortars were also tested in the laboratory taking into account their physical and mechanical characteristics. The mechanical characteristics determined were: modulus of elasticity by two different methods, compressive strength and flexural strength. In turn, the physical characteristics determined were: water vapour permeability and water absorption by total immersion and capillary action. The best mechanical behaviour (compressive and flexural) was observed in the mortar with pozzolanic additions. Similarly, the value of the modulus of elasticity was better in mortars with pozzolanic additions. The performance of these mortars was also adequate in terms of water behaviour.

The mortars revealed suitable characteristics for application in building conservation situations concerning coating with glazed tiles.

Keywords

Conservation; glazed tiles; lime mortars; pozzolanic additions.

Resumo

Portugal é um dos países europeus em que o património construído é um testemunho da sua história. Neste contexto, o legado relativo aos azulejos de revestimento de fachada necessita de ser preservado.

A investigação desenvolvida foi dedicada à conservação destas fachadas na cidade de Ovar, considerada exemplar devido à riqueza do seu património azulejar, do qual uma percentagem considerável necessita de acções de intervenção profundas. Por este motivo, o trabalho desenvolvido focou-se no estudo das argamassas de assentamento para este tipo de fachadas. De forma a permitir o estudo das argamassas antigas, foram retiradas várias amostras de argamassa de diversos edifícios da cidade. Estas amostras foram ensaiadas para determinar as suas características físicas e mecânicas com o objectivo de produzir argamassas compatíveis, passíveis de aplicação em casos de destacamento dos revestimentos azulejares nestes edifícios e, de uma forma geral, em fachadas azulejadas.

Com este objectivo, quatro formulações de argamassas de cal, com diferentes traços volumétricos, foram efectuadas. Os agregados utilizados foram areia de rio siliciosa e uma areia local (saibro). Em três destas composições foi adicionado metacaulino, para que actuasse como uma pozolana artificial, melhorando o desempenho destas argamassas. A utilização de adições pozolânicas promove o endurecimento de argamassas de cal nos casos em que o ingresso de dióxido de carbono é baixo como é o caso de argamassas de assentamento localizadas sob uma camada de azulejo. Relativamente a estas argamassas, foram efectuados ensaios laboratoriais para testar as suas características físicas e mecânicas. Como características mecânicas, foi determinado o módulo de elasticidade utilizando duas metodologias diferentes, assim como a resistência à flexão e compressão. A determinação de características físicas incidiu na determinação da permeabilidade ao vapor de água, na absorção de água por imersão e na absorção de água por capilaridade. Verificou-se uma melhoria do comportamento mecânico (resistência à flexão e compressão) de argamassas com adição de pozolana. De forma similar verificou-se um valor mais elevado para o módulo de elasticidade destas argamassas. O comportamento destas argamassas relativamente à absorção de água revelou-se adequado.

As argamassas testadas revelaram características apropriadas para a aplicação na conservação de edifícios no caso de utilização como argamassas de assentamento para revestimentos azulejares.

Palavras-chave

Conservação; azulejos; argamassas de cal; adições pozolânicas.

Introduction

Throughout its history, Portugal created a vast amount of heritage. The built heritage is a very important part of that collection, and also a visiting card of Portugal. In this context, the legacy of glazed tile facades marked an age in the country, so it must be respected and deserves to be taken care of. A fundamental aspect of the conservation and rehabilitation of glazed tile facades is the study of mortars used as a support for glazed tiles. These mortars are usually lime based mortars. In this context, Ovar is an exemplary case because it has an important glazed tile heritage, some of it very degraded and it is also considered the glazed tile museum city. Thus, the city of Ovar can be considered an "open sky laboratory" to be used for the preservation of its built heritage and to serve as an example for future studies and interventions.

This study focuses on execution, characterization and application of new compositions of lime based mortars with and without pozzolanic additions, compatible with the existing ones. Initially, in order to characterize the samples collected directly from the facades under study, several tests there were made to provide information about the desirable characteristics for the replacement mortars. So, old mortars were subject to mineralogical analysis by XRD (x-ray diffraction), and TGA analysis (thermo gravimetric analysis) to quantify the binder percentage in mortars. Concerning the mechanical characterization, the dynamic modulus of elasticity (E) by two different methods (ultrasound and by the resonance frequency) and compressive strength (Rc) were determined. Regarding the behaviour towards water, the following characteristics were determined: water absorption by capillary action and water vapour permeability.

Four mortar formulations were subsequently executed, three consisting of air lime and metakaolin (artificial pozzolan) and one consisting solely of air lime as binders. In these formulations two different sands were used. On these a series of mechanical tests were carried out in the laboratory: determination of the modulus of elasticity (E) by the method of the resonance frequency, determination of flexural strength (Rf), determination of compressive strength (Rc) and restrained shrinkage test. These mortars were also subject to tests such as capillary water absorption. In the end, "in situ" panels were executed in the facades of buildings targeted for intervention; these were subject to the determination of adhesive strength and water absorption tests using capillary Karsten tubes.

Requirements that repair mortars should fulfil

In an operation for conservation or in cases when it is necessary to replace the existing mortars the following must ensure mechanical, physical, chemical and aesthetic compatibility with the pre-existent materials. Functional characteristics and composition (constituents and type of mortar) of existing mortars should be reproduced as faithfully as possible, especially in cases of filling gaps or in historical monuments [1].

Air lime mortars usually ensure an adequate compatibility with old adjacent mortars. With the addition of metakaolin the general characteristics of mortars remain the same, following conservation practice requirements, but they attain higher mechanical strength, higher durability, higher resistance to salts and faster hardening even under wet environments or with low exposure to air.

Development of new lime based mortars

Table 1 shows the qualitative compositions of the developed mortars.

Table 1 Qualitative composition of the new mortars

Mortar	Materials			
	Lime	Metakaolin	Argillaceous sand	Ordinary river sand
COM	X	X	X	
CRM	X	X		X
CORM	X	X	X	X
COR	X		X	

Table 2 Water/binder ratio

Mortar	Water/binder
COR	1.38
CRM	1.21
CORM	1.21
COM	1.79

The volumetric ratio that was used in the studied mortars without metakaolin was 1:3, for mortars with metakaolin the ratio that was used was 1:0.5:2.5. The 1:3 ratio was chosen considering its current use, while the 1:0.5:2.5 ratio was chosen because of previous results obtained. It is important to refer that the amount of water used for mixing the mortar, shown in Table 2, was obtained in an experimental way assuming an adequate workability as the main goal.

Experimental procedure - Results and discussion

Historical mortars characterization

In this research, seven buildings were targeted for intervention, all of them located in the city of Ovar. The buildings object of intervention and the collected mortars are essentially from the nineteenth century [2].

Sampling

Samples were taken from the following buildings:

- Ovar Musuem (MO)
- Residential building at Rua Visconde de Ovar (VO)
- Residential building at Rua Dr. José Falcão (JF)
- Residential building at Rua Camilo Castelo Branco (CCB)
- Residential building at Rua Dr. Cunha (DC)
- Residential building at Rua Dr. António Sobreiro (DAS)

Sampling in each building was performed at various heights taking into account different conservation states of the facade tiles. Sample location is identified taking into account the building where it was collected, the area from which it was taken (Z1, Z2,…), as seen in Figure 1, and the sample number (A1, A2, A3…) in the form of : House_Zone_Sample.

Fig. 1 Mortar sampling, showing different sampling areas

Mechanical characterisation

Figure 2 reflects the determination of mechanical characteristics of the historical mortars.

The determination of the elastic modulus (E) was performed in two different conditions, the first considering the distance covered by the probe through the average thickness of the sample (cross measurement) and the second considering the distance travelled towards the average length of the sample (longitudinal measurement).

Figure 2 shows that the JF building mortars have a better performance in terms of compressive strength (1.67 MPa) and the second largest value of modulus of elasticity (1785 MPa); the highest elastic modulus concerns the VO building, however in this case it was only possible to test a single sample so the result may not reflect the real performance of the building. Since all the buildings are located in the same geographic place, the average value of compressive strength of all samples is 1.38 MPa. In this way the MO building assumes the most distanced value from the average.

In the definition of the elastic modulus, the longitudinal measurement of samples led to values substantially higher (about twice) than the cross measurement. This discrepancy may be due to the fact that, in cross measurements, the heterogeneity of material that the probe crosses is greater than that in longitudinal measurements. The values for the longitudinal measurement are more reliable and consistent for this type of mortars.

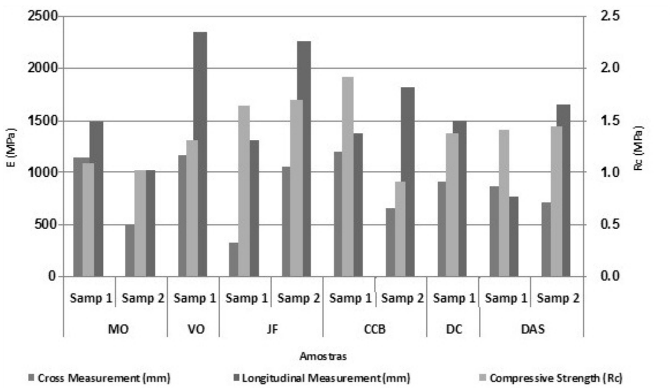

Fig. 2 Mechanical characterisation of the historic mortars

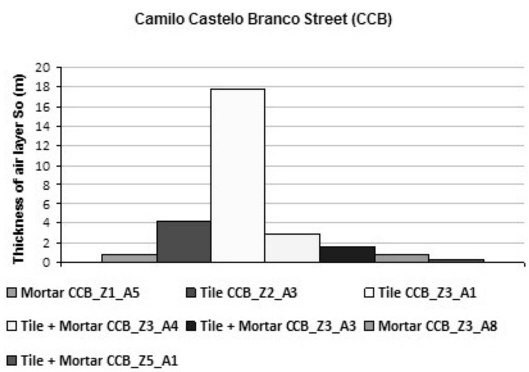

Fig. 3 Thickness of the equivalent air layers of mortar samples

Still, within the same building, the samples with lower compressive strength also have a low modulus of elasticity, with the exception of the CCB building.

It is possible to conclude that the JF building has the best average for mechanical behaviour [3]. On the other hand, the results of lime based mortars from the MO building show that the building has the worst mechanical behaviour. The discrepancy between the values of the elastic modulus and the compressive strength of samples collected in the same building is also relevant, leading to the conclusion that the area of sampling may greatly influence the mechanical characteristics probably due to changes in state of repair or due to the different degrees of exposure to aggressive actions.

Physical characterisation

Absorption of capillary water

Water absorption by capillary action test results revealed that the capillary coefficient of the two old tiles that were tested is very similar (MO_Z3_A9 - 4.69 kg/m^2.h$^{1/2}$ and CCB_Z3_A3 - 5.89 kg/m^2.h$^{1/2}$), however the capillary coefficient of the Camilo Castelo Branco building sample (CCB) is slightly higher.

Water vapour permeability of tiles, mortars and tile/mortar set

The determination of water vapour permeability was made according to EN 1015-19 [4].

Table 3 Characteristics of the developed mortars

Characteristics			Samples			
			COM	CRM	CORM	COR
Average consistence (mm)			133.25	124.50	124.00	119.50
Water/binder			1.38	1.21	1.21	1.79
Density (g/dm^3)			1969.15	2001.65	2007.10	2037.60
E (MPa)	Resonance frequency	28d	3810	3300	2905	2970
		90d	2944	2448	1673	2995
	Normals	28d	3775	3639	3390	3092
		90d	2602	2280	1617	2570
	Exponentials	28d	2011	2495	2145	2226
		90d	2482	2035	1410	2482
Compressive Strength (MPa)		28d	2.78	2.63	2.18	0.62
		90d	3.15	1.82	1.66	0.65
Flexural Strength (MPa)		28d	0.52	0.42	0.37	0.20
		90d	0.79	0.41	0.26	0.20
Restrained Shrinkage		CSAF	-	-	3.70	1.60
		CREF	-	-	0.80	0.80
Capillary Coefficient (kg/m^2.h$^{1/2}$)		28d	8.01	8.71	8.64	10.94
		90d	12.93	13.04	13.73	10.15

Samples of several buildings were tested, with the following variations: only tile, only mortar and tile/mortar specimens.

From the analysis of Figure 3, it appears that the thicknesses of the equivalent air layers of mortar samples are similar, whilst tile samples have a greater difference of values. This variability may be due to the area of tile location. The sample of tile CCB_Z3_A1 which has the lower permeability was taken from an area without much effect from water action. Although the sample CCB_Z2_A3 is also from a tile, this already presents a much higher permeability than the sample above, but still lower than that of all other samples containing mortar. This sample is an area of deployment, as seen in Figure 2.

Relatively to samples of tile with mortar (2 layers), samples CCB_Z3_A2 and CCB_Z3_A4, they have a similar behaviour. Comparing them with the sample CCB_Z5_A1 of tile with mortar, but with a single layer, they have lower permeability. It should be noted that the sample containing only one layer of mortar had higher deterioration and it no longer had glaze. During the collection of samples it was found that in the area (Z5) the mortar was wet.

Characterisation of the new lime based mortars

Table 3 summarizes all determined characteristics of the developed mortars.

Concerning the determination of fresh mortars characteristics (consistence, water/binder ratio and density), it appears that COR is the mortar that has the lowest consistence of the studied mortars despite its higher water/binder ratio and density.

In fact, the characteristics of hardened mortars shown in Table 3 reveal that the mortar without metakaolin (COR) is the one that presents the worst results at all levels (modulus of elasticity, flexural and compressive strength).

It is still possible to see that the results for the modulus of elasticity determined by normal transducers are always higher than those determined by exponential transducers and look more alike the results for the modulus of elasticity by the resonance frequency.

In general the modulus of elasticity between 28 days and 90 days decreased in value. It is interesting to note that the variation in the mortars with metakaolin (CRM, COM, CORM) was higher than in the mortar without metakaolin (COR). The same is true in the case of compression and bending. Through the compression/flexural ratio, it can be seen that the mortars with metakaolin have higher relations than the mortar without metakaolin. Ideally, this ratio should be close to 1, indicating a lower cracking susceptibility, but this is not the case in the studied mortars [3]. Although relations are high, in CRM and COM mortars the same fell about 1.5 times with ageing, unlike the mortars CORM and COR that have a slight increase.

In short, mortars with metakaolin, in a 60 days period, reduced their mechanical properties in a relevant way. The air lime mortar without metakaolin had a behaviour without large fluctuations, as the values determined at 28 days were very similar to those obtained after 90 days. It is also clear that the mortar with a mixture of sands (CORM) has a worse mechanical behaviour than the mortars with only one sand type.

Regarding the susceptibility to cracking, according to Rosário Veiga [5] the COR and CORM mortars belong to the same class (average) of susceptibility to cracking. This test also indicates that the maximum force developed by restrained shrinkage is similar and relatively low in the two tested mortars, indicating good compatibility of these mortars with old supports and with old glazed tiles [6].

The adhesion test was not possible to execute as during the initial drilling procedure glazed tiles came off. Therefore, the test was performed without drilling. However, in this variant of the test, all the strength of the glazed tile was mobilized. The new tiles tested higher than the old.

From this experience it is possible to see that there is a fragility of the system, in which there is a low adhesion between the tile and mortar.

Table 3 also indicates that the mortar with the largest spread is CRM (lime, river sand and metakaolin) although the water/binder relationship, in weight, is the lowest, due to lower absorption of river sand when compared with sand of Ovar, which has more clay. The mortar COR (lime, sand of Ovar and river sand) is the mortar with less spread, higher density and greater water/binder ratio. This presents the higher capillary coefficient at 28 days, when compared with the other mortars and at 90 days it shows a lower capillary coefficient.

By comparison, it is evident that the mortars with metakaolin at age 90 days increased their capillary absorption rate and those containing no metakaolin slightly decreased the value obtained at 28 days. This decrease can be explained by the influence of carbonation, because there is a differentiation of the porous structure. The mortars with metakaolin have fibrous CSH and do not have pores filled with calcite crystals, whilst mortars with no metakaolin have carbonated producing calcite crystal and thus filled the porous structure.

Compatibility between historical and new mortars

Table 4 has the purpose of allowing the comparison between the several determined mechanical characteristics (E and Rc) common to both historical and new mortars. Regarding the comparison between the modulus of elasticity (E) of old and new mortars at 90 days it is evident, in general, that in the new formulations the value is higher than in the old mortars (about 1.5 times higher). However, it is pointed out that the mortar CORM assumes values similar to those of ancient mortars and mortar of VO building takes values similar to the new mortars.

Regarding compressive strength of old and new mortar, the obtained values are similar. Despite this similarity, it is interesting to note that the COR mortar, without metakaolin, has about half the value obtained in the old mortars. This may be due to the fact that the carbonation process of the lime has already been completed in the old mortars while in the new mortars the same process is still under way. It also possible to raise the issue if in the old mortar there is some material that works as a pozzolanic binder, such as metakaolin, because CRM and CORM mortars contain metakaolin and have very similar values to Rc.

Regarding COM mortar, its compressive strength is about twice that of old mortars. This would be expected because the mortar contains argillaceous sand and metakaolin. Thus, the last mortar can be little compatible

Table 4 Mechanical behaviour of mortars (historical and new)

Mortars		E (MPa)		E (MPa) 90d			Rc (MPa)
				Ultrasounds		Renossance Frequency	
		Long. M.	Cross M.	Normal	Exponencial		
Historical mortars	CCB	1593	917	-	-	-	1.41
	MO	1250	818	-	-	-	1.05
	JF	1785	684	-	-	-	1.67
	DC	1489	907	-	-	-	1.37
	VO	2338	1159	-	-	-	1.30
	DAS	1206	858	-	-	-	1.42
New Mortars	COM	-	-	2602	2482	2944	3.15
	CRM	-	-	2280	2035	2448	1.82
	CORM	-	-	1617	1410	1673	1.66
	COR	-	-	2570	2482	2995	0.65

with other materials from the old buildings studied. However, the moderate modulus of elasticity of this mortar shows that it has a deformation capacity closer to that of other compositions which reduces the risk of incompatibility.

From the analysis of figure 4, the difference between old and new tiles is evident in terms of capillary absorption but not in terms of the capillary coefficient. New tiles have a lower absorption of water in comparison with the old, while the value of the capillary coefficient is similar between tiles 5 and 6 and the old tiles. The new tile 4 shows the greatest difference in capillary coefficient, with lower water absorption.

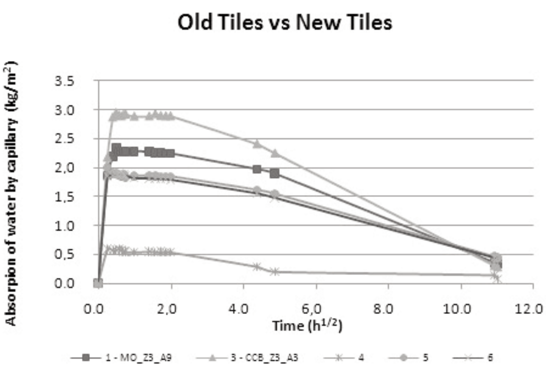

Fig. 4 Water absorption by capillary action (kg/m^2)

Through the figure above it is evident that the old tiles behave better in terms of drying. The tile CCB_Z3_A3 is the one with a greater absorption of capillary water with a maximum absorption of 2.95kg/m^2, for 15 minutes. The new tile 4 has the lowest capillary absorption values, and its maximum of 2.00 kg/m^2 is, immediately, reached at 4 minutes after the start of the test.

Conclusions

Historical mortars characterization

The mechanical characteristics analysis may indicate that the area where the mortars were sampled can influence their mechanical behaviour; as an example, in one of the analyzed buildings, the samples analyzed provided the lowest and highest compressive strength values. This may be due to differences in the state of conservation, related to greater or lesser exposure to aggressive actions (water pollution).

It is noted that the modulus of elasticity determined by measuring the longitudinal length of the sample is more consistent with the values generally obtained for this type of mortar instead of the modulus of elasticity determined by measuring the cross length of the sample.

Likewise the compressive strength values are congruent with the values normally obtained for this type of mortars.

Water absorption by capillary action tests showed that the absorption of water from old tiles was higher than that of new tiles, but they also had a better drying ability.

The results obtained with water vapour permeability tests of old mortars, showed huge variations depending on various sets (tile, tile + mortar, mortar) and the different areas where they were sampled.

Results of water vapour permeability for mortars were, however, similar. There is a high influence of tiles in this parameter, and variability factors of the composition and conservation status of the tiles.

New mortars characterization

The mechanical characterization of mortars developed allowed the conclusion that the mortars with pozzolanic additions (metakaolin) have a better mechanical behaviour in comparison to the mortars without metakaolin for the ages of 28 and 90 days.

Despite the best behaviour of these lime/metakaolin mortars it should be enhanced that from 28 days to 90 days of age, in general, the mortars with metakaolin present a relevant decrease in their mechanical behaviour.

It may also be concluded that the studied mortars formed only with a single type of sand have better mechanical behaviour than the mortars studied with two types of sand (gravel and ordinary river sand), probably due to the chemical and mineralogical nature of the gravel.

Regarding the "in situ" tests it is possible to infer that there is a problem to be solved concerning the mortar / tile interface.

Compatibility between the new and historic mortars

After a comparative examination of historic and developed mortars, concerning their mechanical behaviour it may be concluded that the determined characteristics are similar.

Regarding the results for the modulus of elasticity, the new studied mortars tend to converge to the values obtained for the old mortars.

Concerning the compressive strength there is also a similarity of values, with the exception of COM mortar.

Consequently there is compatibility between the old and new mortars regarding the type of binder and mechanical behaviour. It is estimated that there is chemical compatibility between them, given the similarity of compositions.

After the development of new mortars, they were subjected to tests to characterize their behaviour towards water.

Applications like salt analysis, water absorption and adhesion were performed.

The results obtained in this study suggest that the lime based mortars with metakaolin might be good solutions for use as tile support mortars in the building facades of Ovar in conservation interventions.

It will be important to continue with this work in order to carry out more detailed characterization of old mortars of glazed tiles facades of the city of Ovar (Tile Museum City), because of the wide range of the heritage that exists.

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Study of materials and technology of ancient floor mosaics' substrate

Estudo dos materiais e da tecnologia dos substratos de mosaicos de pavimento antigos

Vincenzo Starinieri

PhD student, Laboratory of Building Materials, Department of Civil Engineering, Aristotle University of Thessaloniki, Thessaloniki, Greece,
starvinc1@inwind.it

Ioanna Papayianni

Professor, Laboratory of Building Materials, Department of Civil Engineering, Aristotle University of Thessaloniki, Thessaloniki, Greece,
papayian@civil,auth.gr

Maria Stefanidou

Lecturer, Laboratory of Building Materials, Department of Civil Engineering, Aristotle University of Thessaloniki, Thessaloniki, Greece,
stefan@civil.auth.gr

Abstract

A floor mosaic's substrate is composed of a variety of preparatory layers of mortar built on natural levelled ground or on top of a previous pavement. Mosaics' substrates differ one from the other in number, thickness and nature of the mortar layers. In this sense, it has been considered relevant to state how these differences are related with historical period, geographical position, function of the pavements within the building, technology of the substrates. A number of floor mosaics' substrates of Hellenistic and Roman period from archaeological sites in Greece and Italy are under study. The stratigraphy of substrates is recorded in situ, and samples from each mortar layer are analysed in the laboratory by means of different techniques. Results obtained so far indicate that characteristics of the Roman substrates mortar layers are clearly dependant on their position in the substrate stratigraphy, whereas in the case of the Hellenistic substrates, characteristics of the mortar layers are less varying with the stratigraphic position. Furthermore results show that floor mosaics' substrates are different according to the function of the pavement in the building.

Keywords

Mortar; mosaic; substrate.

Resumo

O substrato de um mosaico de pavimento é composto por uma variedade de camadas preparatórias assentes sobre um terreno nivelado ou sobre um pavimento previamente existente. Os substratos de mosaicos podem diferir entre si em número, espessura e natureza das camadas de argamassa. Neste sentido, foi considerado relevante registar de que forma se relacionam estas diferenças com o período histórico, posição geográfica, função dos pavimentos no edifício, tecnologia dos substratos. Um grupo de substratos de mosaicos dos períodos Helenístico e Romano, provenientes de sítios arqueológicos localizados na Grécia e em Itália, estão correntemente em estudo. A estratigrafia dos substratos é registada in-situ, e são recolhidas amostras de cada camada de argamassa para análise em laboratório através de diferentes técnicas. Os resultados até agora obtidos indicam que as características das camadas de argamassa dos substratos romanos dependem claramente da sua posição na estratigrafia do substrato, enquanto que, no caso dos substratos helenísticos, as características das camadas de argamassa variam comparativamente menos com a posição estratigráfica. Os resultados mostram ainda que os substratos de mosaicos de pavimento diferem consoante a função deste último no edifício.

Palavras-chave

Argamassa; mosaico; substrato.

So far, the substrates of sixteen floor mosaics located in five archaeological sites, two of which are in Greece and three in Italy, have been included in this research. Two of these substrates belong to floor mosaics placed in two rooms at the west side of the Palace of Aegae, in Vergina (Greece). The Palace of Aegae was the earliest royal residence in late Classical Greece, dated to the second half of the 4th century B.C.. Five more of the studied substrates belong to mosaics situated in Roman buildings inside the ancient city of Dion (Greece). Dion, located in the foothills of Mount Olympos, was the sacred city of the Macedonians, inhabited continuously from the Classical period to Early Christian times. Two more substrates belong to the remains of a Roman building, dated to the 1st century A.D. situated at a level below the S. Severo's Basilica in Classe, Ravenna (Italy). In the area of the S. Severo's Basilica, the last of Ravenna's great churches to be completed, dated to the end of the 6th century A.D, an archaeological excavation is actually in progress. Two more of the investigated substrates are those of the floor mosaics located in two rooms of the "Villa Romana delle Muracche" in Tortoreto (Italy), a Roman Villa dated to the 1st century B.C.. The last five substrates under study belong to floor mosaics that had been lifted during past excavations in different areas of the city of Florence (Italy) and are currently stored in the courtyard of the Archaeological Museum of Florence, the so called "Cortile Romano".

Analytical methodology

In situ analyses

A characterization of the mosaics' substrates, including recording of layers thickness, description of mortars cohesion and layers mutual adhesion is carried out in situ. The stratigraphy of the substrates is reproduced schematically in order to compare substrates characteristics, especially number and thickness of the mortar layers, among the floor mosaics under study (Fig. 1). Furthermore, information about the sites are collected, especially those regarding the ancient use of the rooms where pavements investigated are located, and an effort to connect these information with substrates characteristics is made [1].

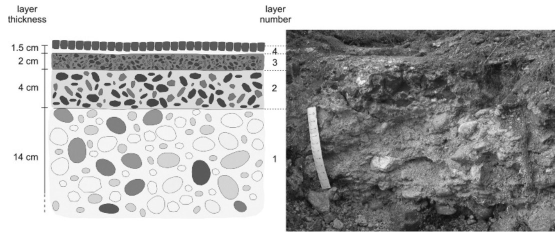

Fig. 1 Roman floor mosaic, ancient city of Dion (Greece), stratigraphy of substrate.

Laboratory analyses

According to sampling conditions in situ, collected samples are made up of one or more layers of mortar. In the latter case the different layers of mortar are separated in laboratory by means of scalpel, chisel and rock saw when necessary. Analyses are then performed on each mortar layer of the substrates' stratigraphy. Layers are indicated with increasing numbers starting from the deepest. The mortars are studied by reflected and transmitted light optical microscopy to investigate presence of enclosures, shape and position of pores and cracks, morphology and mineralogy of the aggregate. Observations are carried out on cross sections using a Leica Wild M10 stereomicroscope and on thin sections using a Leitz Laborlux 12 Pol S polarizing microscope. Grain size distribution of the mortar layers is determined by mechanical sieving (ISO 3310 series sieves). The sample, about 80 g, is disaggregated manually, paying attention to not breaking the aggregates, and fractionated in a column of sieves stirred for 10 min, and then the masses of the collected fractions are determined. The granulometric fraction of particle size < 75 μm, containing the binder, is analysed by AAS and HPLC in order to determine the chemical composition and the content of soluble salts. AAS analyses are performed on a Perkin Elmer 3110. For the preparation of samples, 0.25 g of the fraction < 75 μm are treated by wet ashing with $HClO_4$, HF and HCl 6N for the determination of the total oxides content, and 0.25 g are used for the determination of the oxides soluble in HCl 0.1N. HPLC is performed with an Alltech 330 column. Furthermore, a loss on ignition test is carried out on the fine particle size < 75 μm in order to estimate carbonate, organic and elemental carbon content.

The open porosity of the samples is measured following a method based on RILEM CPC 11.3 [2]. Compressive strength of some of the mortars is estimated on samples of approximately cubic shape immerged in moulded gypsum. Before performing the test, gypsum in excess is removed from the four vertical sides of the sample while upper and lower horizontal gypsum surfaces are kept for the application of the compressive stress.

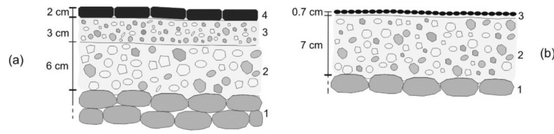

Fig. 3 Hellenistic floor mosaic, Palace of Aegae, Vergina (Greece), stratigraphy of the substrate in the inner area (a) and in the outer frame (b).

Results and discussion

Results obtained so far indicate that mosaics' substrates of a defined historical period located in the same building, either Roman Villa or Roman Domus or Hellenistic Palace, differ one from the other according to the type of room where they are placed. An example is provided by mosaics in the Roman buildings of the ancient city of Dion. The pavements situated in the atrium of a Roman Domus and in the atrium of a Roman Villa have substrates composed of two layers of mortar and a layer of tesserae (Fig. 2 d and c), while those covering the floor of a different room, probably a dining room, in the same Domus, and the floor of a Roman Temple adjacent to the Villa, have substrates composed respectively of three and four layers of mortar plus a layer of tesserae (Fig. 2 b and a).

Furthermore, the analysis of the substrates stratigraphy in different areas of a same mosaic revealed that, in the case of mosaics having an outer frame, the substrate in the area of the frame is different from that in the inner area of the pavement. In Fig. 3 the schematic reproduction of the substrate of a mosaic located in a room serving as banquet hall in the Palace of Aegae, illustrates this case.

Optical microscopy observations indicated for both Roman and Hellenistic mortars the use of materials of local origin. Fluvial sand and pebbles are the main constituents of the aggregates. The use of brick fragments (cocciopesto) as part of the aggregate is recurrent in the mortars of the Roman substrates (Figs. 4 a and 5 a). Brick fragments are always present in the mortars of the 2nd and 3rd layers of the stratigraphy of substrates composed of three or four layers as well as in the mortars of the 1st layer of substrates composed of two layers. Furthermore, in the case of Classe and Tortoreto, brick fragments with dimensions of several centimeters are present also in the 1st layer of substrates composed of three or four layers. Marble dust and fragments were also used for the 4th layer (bedding layer) of the Roman substrates from the ancient City of Dion and the Roman Villa in Tortoreto. In the Hellenistic mosaics' substrates from the Palace of Aegae, pozzolanic sand has been recognized.

In Fig. 6 a and b, the results of the fractionation and sieving of the Roman mortar samples from the ancient City of Dion are represented as the wt.% of each particle-size range against particle-size range. Three types of grain size distribution have been observed in the granulometric analysis:

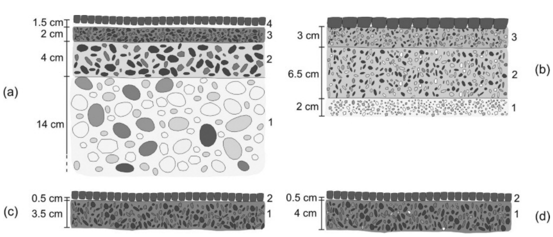

Fig. 2 Roman floor mosaics, ancient city of Dion (Greece), stratigraphy of substrate in a Roman temple (a); dining room in a Roman Domus (b); atrium in a Roman Villa (c); atrium in a Roman Domus (d).

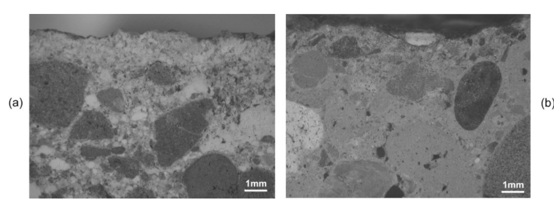

Fig. 4 Cross sections under reflected light microscopy, 3rd and 4th layer of a Roman substrate from the ancient city of Dion (a) and of a Hellenistic substrate from the Palace of Aegae (b).

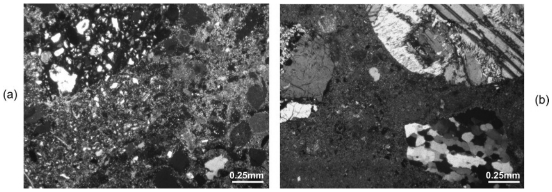

Fig. 5 Thin sections under transmitted polarized light microscopy, 3rd layer of a Roman substrate from the ancient city of Dion (a) and of a Hellenistic substrate from the Palace of Aegae (b).

descending, bimodal and unimodal. In general, substrates composed of at least three layers, besides the bedding layer, showed descending distribution for mortars of the first and the second layer of the stratigraphy and bimodal distribution for mortar of the third layer (Fig. 6 a). On the other hand substrates composed of only one layer of mortar, beside the bedding layer, showed unimodal distribution (Fig. 6 b).

The AAS results for both total and soluble in HCl 0.1 N oxides content show that the chemical characteristics of the Roman mortar layers, with the only exception of those from the mosaics of the Roman Villa in Tortoreto, are related with their position in the substrate stratigraphy. In fig. 7, the total content in CaO + MgO of the mortars is plotted versus the $SiO_2 + Al_2O_3 + Fe_2O_3$ total content. Considering the Roman mortars, those of the 1st layer have low percentages of $SiO_2 + Al_2O_3 + Fe_2O_3$ (11-21%) and high percentages of CaO + MgO (46-51%). Mortars of the 2nd layer are characterised by a $SiO_2 + Al_2O_3 + Fe_2O_3$ content between 19% and 42% and a CaO + MgO content between 23% and 37%. Most of the mortars of the 3rd layer have high percentages of $SiO_2 + Al_2O_3 + Fe_2O_3$ (43-55%) and low percentages of CaO + MgO (18-22%). The homogeneity in the chemical characteristics of the mortar layers of the Roman Villa of Tortoreto can be explained considering that an argillaceous limestone was used to make mosaics' tesserae. The same limestone was maybe used also for the lime production process, which would explain why the mortar of the 1st layer, containing neither natural nor artificial pozzolanic materials, showed the same composition as mortars of the 2nd and 3rd layer. Mortar layers of the Hellenistic substrates also showed a substantial uniformity in the chemical characteristics, being characterised by a $SiO_2 + Al_2O_3 + Fe_2O_3$ content between 27% and 32% and a CaO + MgO content between 35% and 40%.

In fig. 8, the loss on ignition (L.O.I.) is plotted versus the CaO + MgO total content in the mortars fraction of particle size < 75 μm. Most of the mortars of the 2nd and the 3rd layer of the substrates stratigraphy showed significant L.O.I. percentages and low CaO + MgO percentages, indicating a possible organic and elemental carbon content.

HPLC registered no relevant concentrations of Cl^-, NO_3^-, SO_4^{2-} in the mortar layers of all the substrates under study except for those of the mosaics of the area of Classe, in Ravenna. In this case, chloride salts have

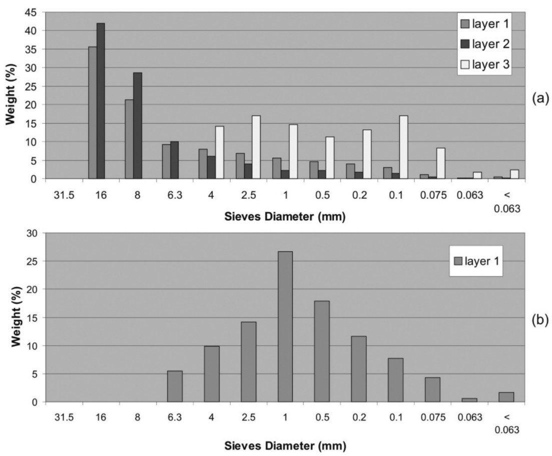

Fig. 6 Grain size distribution of mortar layers of mosaics' substrates in a Roman temple (a) and in the atrium of a Roman Villa (b) in the ancient city of Dion.

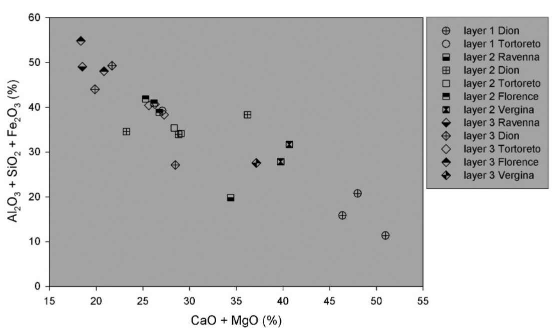

Fig. 7 CaO + MgO total content versus $SiO_2 + Al_2O_3 + Fe_2O_3$ total content of the fraction of particle size < 75 μm of the mortar layers.

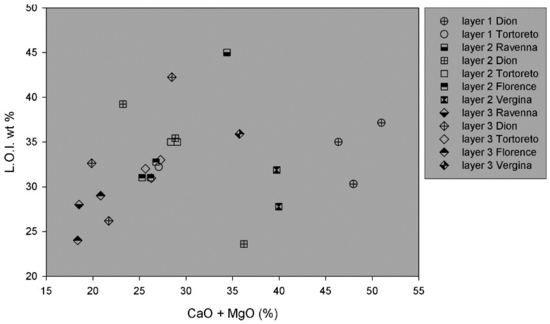

Fig. 8 CaO + MgO total content versus loss on ignition for the fraction of particle size < 75 μm of the mortar layers.

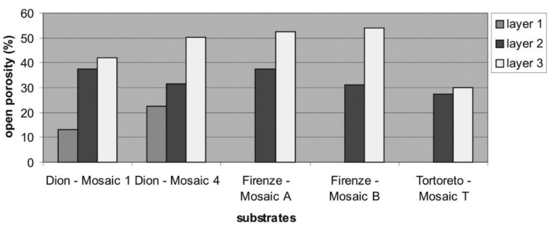

Fig. 9 Open porosity trend for Roman floor mosaics' substrates.

been found, with higher concentrations in the lower part of the substrates stratigraphy. Presence of chlorides in buried structures is probably related, in the area of Ravenna, to underground sea water upraise phenomena.

The results of the open porosity test show that, in most of the Roman substrates, open porosity of the mortars increases from the 1st to the 3rd layer of the stratigraphy (Fig. 9).

So far, results of the compressive strength tests have not shown evident trends in the mechanical characteristics of the mortar layers. Anyway it is noticeable that, in the Roman substrates, layers characterised by higher values of compressive strength are the 2nd and the 3rd. These mortar layers are those containing brick fragments of granulometric size < 1.5 - 2 cm and showing higher content of $SiO_2 + Al_2O_3 + Fe_2O_3$, as stated by chemical analyses. Furthermore, between the 2nd and the 3rd layer, it is the former that in most of the examined substrates is characterised by a higher compressive strength. This is probably related to the fact that its open porosity is generally lower than that of the 3rd layer [3-4].

Conclusions

The systematic study of floor mosaics' substrate, by means of in situ and laboratory analyses, leads to the understanding of technological aspects involved in their manufacture. The description of the stratigraphy of the studied substrates shows the existence of a correlation between substrates characteristics and ancient usage of the rooms where pavements are located within the building. Furthermore, the same pavement, if composed of more structural elements such as an external frame and an inner area, can have two different substrates below the two elements, in this case the frame and the inner area.

The microscopic, chemical, physical and mechanical characterization of the studied mortars indicates that, in the case of Roman substrates, characteristics of the mortar layers are distinctly related to their stratigraphic position in the substrate [5]. Properties mostly distinguishing mortars of the different layers include granulometric distribution of the aggregates, open porosity and chemical composition of the fraction of particle size < 75 μm. On the other hand, results obtained so far for the Hellenistic substrates from the Palace of Aegae indicate that, although mortar layers differ one from the other in terms of granulometric distribution of the aggregates, other properties, such as open porosity and chemical composition of the fraction of particle size < 75 μm, that demonstrated to be distinctive of the various mortar layers in the Roman substrates, are in this case less varying with the stratigraphic position.

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Reconstruction of the Chalet of the Countess of Edla: preliminary evaluation of the effects of fire in the architectural surfaces

Reconstrução do Chalet da Condessa de Edla: avaliação preliminar dos efeitos do fogo nas superfícies arquitectónicas

Graça Horta

Bachelor in Conservation, Conservator-Restorer, Instituto dos Museus e da Conservação, Lisbon, Portugal, hortae@gmail.com

José Maria Lobo de Carvalho

MA Cons.(York)/Architect, Parques de Sintra – Monte da Lua, S.A., Sintra, Portugal, jose.lobodecarvalho@parquesdesintra.pt

V. M. Rato

Ph.D./Assistant Professor, Fac. Ciências e Tecnologia – Univ. Nova de Lisboa, Lisbon, Portugal, vnmr@fct.unl.pt

Abstract

This paper addresses the effects of fire on historic surfaces through the scientific analysis of the original mortars and pigments of the Chalet of the Countess of Edla, located in the Park of Pena (Sintra, Portugal). It is intended as a first approach to the study and characterization of the historical fabric in the context of the ongoing restoration project, promoted by Parques de Sintra-Monte da Lua, SA.

Keywords

Pena; Countess of Edla; fire; mortars; decorative render.

Resumo

O presente trabalho aborda os efeitos do fogo em superfícies históricas através da análise científica das argamassas e dos pigmentos originais do Chalet da Condessa de Edla, localizado no Parque da Pena em Sintra. Tem como objectivo constituir-se como uma primeira abordagem ao estudo e caracterização deste património histórico no contexto do projecto de restauro em curso, promovido pela empresa Parques de Sintra-Monte da Lua, SA.

Palavras-chave

Pena; Condessa de Edla; fogo; argamassas; reboco decorativo.

Introduction

The Chalet is a romantic cottage built between 1864-69 by the King–consort Dom Fernando II and his second wife, the Countess of Edla, within the Park of Pena, surrounding the Royal Palace (figure 1), part of the wider area listed by UNESCO as Cultural Landscape of Sintra, in 1995. This building is a remarkable example of romantic architecture with significant influences of the Northern European and American mountain cottages, following the Countess's original countries of birth and adoption (Germany and USA). Being a small scale structure, it was built as a personal retreat, away from the court life, with a particular decorative scheme that includes painted exterior renders simulating wood planks and the extensive use of cork covering doors and window frames, the balconies and the roof decoration elements. In the interior, the building exhibits an interesting decorative scheme of different painted architectural surfaces with coloured patterns and geometric designs.

After the end of the Monarchy in 1910, the building was occasionally used and remained neglected for decades, until a fire of unknown causes destroyed it severely in July 1999. It is believed that the fire started over night and was detected in the following morning, resulting in a slow combustion of the wooden elements of the roof and upper pavements, including the majority of the internal partition walls. The solid limestone masonry structure resisted well, the fire consumed the wooden structure. It also had a destructive effect on the stone slabs from the cantilevered veranda (figure 2).

Following the successful bidding for financial support form the EEA Grants, a partnership was created between Parques de Sintra-Monte da Lua and several national and international partners, with particular mention to the Norwegian Institute for Cultural Heritage (NIKU) and the cleaning operation and salvage selection was carried between September and October 2007.

In order to study the execution of new internal renders compatible with the existing ones (both for partition and masonry walls), the Department of Civil Engineering, from Universidade Nova de Lisboa (a national partner in the project) was responsible for scientific analysis of mortar samples, in order to characterize the properties that will determine the new formulations to be applied, like capillary water absorption, drying and water vapour permeability and the microstructural properties. Additionally, the internal decorative renders recovered from the fire (salvage) were also the object of careful removal, selection and analysis regarding its composition, layering, original colours and pigments used. This analysis was conducted by the Instituto Português de Conservação e Restauro (IPCR - IMC), another national partner of the project. This paper describes the preliminary evaluations.

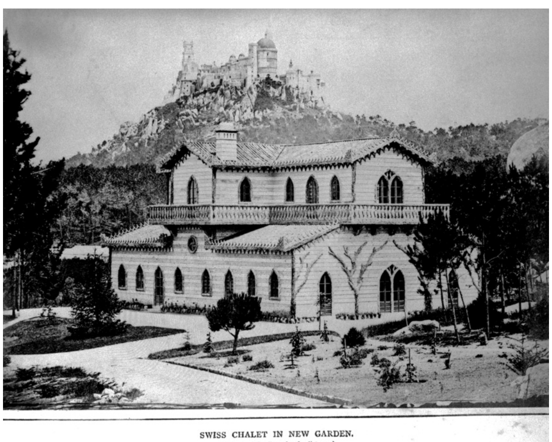

Fig. 1 External view of the Chalet shortly after its construction, with the Palace of Pena in the back.

Fig. 2 Current condition after the fire in 1999 (September 2007).

Experimental campaign and results

The internal renders are made of two mortar layers and one final plaster layer onto which the decorative surfaces were apposed. One of the mortar layers was applied directly on the wooden base (layer A) and the other, an intermediate mortar (layer B) intended to be the substrate for the plaster. In this preliminary study the plaster layer was not analysed. A set of seven fragments was collected from which the samples for testing were taken to evaluate the following materials:

- Pigments of the pictoric layer;
- Mortar of layer A (open porosity and mercury intrusion porosimetry). Samples used are from room 6 and the staircase (figure 3); in both cases, non-affected mortars (samples R6.1 and St.1) and fire-affected mortars (samples R6.1f and St.1f) were analysed;
- Mortars of layers A and B (binder:aggregate ratio). Samples used are also from room 6 and the staircase. The sample of layer A from the staircase was fire-affected whilst the other ones were non-affected by fire.

Pigments

X-Ray diffraction

The composition of the pictoric layer was identified by X-Ray diffraction with a Bruker AXS equipment, model D8 Discover with Cu Kα radiation, GADDS detector and using ICDD (International Centre for Diffraction Data) cards; the collimator diameter has 1 mm (the diameter of the beam on the sample).

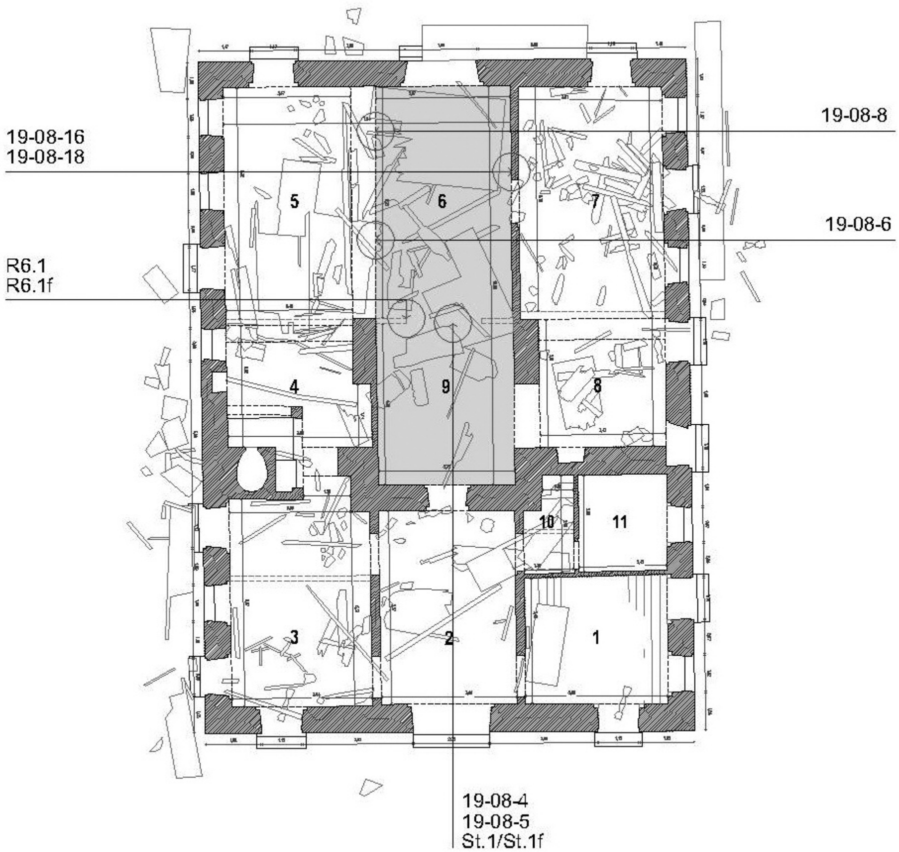

Fig. 2 Plan of the building (ground floor) with the locations of sample collection.

Table 1 Results of X-Ray diffraction of pictorial layers of samples 19-08-1 to 19-08-9.

Samples		Identified Phases
References	Layer/ Colour	
19-08-1 1st fl., Broderies Room	Dark blue	Calcite, Quartz, Barite, Lazurite
19-08-2 1st fl., Broderies Room	Light blue	Calcite, Quartz, Lazurite
19-08-3 1st fl., Broderies Room	White	Calcite
19-08-4 Staircase hall	Red (decoration)	Calcite, Quartz, Hematite, Kaolinite, Cinnabar
19-08-5 Staircase hall	Red (background)	Calcite, Quartz, Kaolinite, Cinnabar
19-08-6 Ground fl., Living Room	Red (decoration)	Calcite, Quartz, Kaolinite, Hematite
19-08-7 Ground fl., Living Room	Red (background)	Calcite, Quartz
19-08-8 Ground fl., Living Room	Yellow (decoration)	Calcite, Quartz, Kaolinite, Goethite
19-08-9 Ground fl., Living Room	Yellow (background)	Calcite, Quartz

Table 2 Results of X-Ray diffraction of pictorial layers of samples 19-08-10 to 19-08-19.

Samples		Identified Phases
References	Layer/ Colour	
19-08-10 1st fl., Cork R.	White	Calcite, Quartz
19-08-11 1st fl., Cork R.	Red (decoration)	Calcite, Quartz, Hematite
19-08-12 1st fl., Cork R.	Black (alteration)	Calcite, Quartz
19-08-13 Ground fl., Living Room, Upper frise	Red (decoration)	Cinnabar, Hematite, Calcite
19-08-14 Ground fl., Living Room, Upper frise	Gray (decoration)	Calcite, Quartz, Barite, Lazurite
19-08-15 Ground fl., Living Room, Upper frise	Red (Background)	Calcite, Quartz, Hematite, Kaolinite
19-08-16 Ground fl., Living Room, Upper frise	Red (decorative motif)	Calcite, Quartz, Hematite, Gypsum
19-08-17 Ground fl., Living Room, Upper frise	Brown (colour alteration of the decorative motif)	Calcite, Quartz, Hematite, Gypsum
19-08-18 Ground fl., Living Room, Upper frise	Black (colour and chemical alteration of the decorative motif)	Calcite, Quartz, Magnetite, Gypsum
19-08-19 Ground fl., Living Room, Upper frise	White	Calcite, Quartz

Table 1 and table 2 show the compounds identified in the samples 19-08-1 to 19-08-19.

Figures 4 to 9 show the fragments from which samples were collected. In some of these, it was possible to analyse the pigment in its original phase and also in its altered phase (darkened areas).

The samples 19-08-6 and 19-08-8 (figure 6) are both of the living-room and concerning the same decoration pattern, with decorative motives painted on the same substrate. The colour of this substrate is a rose tone in most areas, where the identified pigment is hematite. In some small areas, it has a light yellow tone where the identified pigment is goethite. It is not likely that these differences in colour were intentional in a decorative substrate with symmetrical patterns. Therefore, the effects of fire or the action of the water used by firemen have most probably caused a phase change in the original pigments. In order to explain this, two approaches are possible:

- The original pigment was goethite and the decorative substrate was yellow. The heat of fire has dehydrated the goethite with the consequent phase change to hematite that originates the rose tone. In some residual areas the temperature may not have been high enough for this phase change to occur and therefore some scattered yellow areas remained corresponding to the original goethite;

- The original pigment was hematite and the decorative substrate was of a rose tone. In this case, water absorption on some areas of the surface, together with the very humid local climate, have caused the hydration of the hematite with the consequent phase change to goethite which in turn caused the colour change to yellow.

The second approach seems more plausible because the uniformity of the rose colour in most of the painted areas in this room is more in agreement with an original application of a homogeneous colour. In this case the yellow areas should therefore correspond to affected and altered material.

Also, the results of the samples 19-08-16 and 19-08-18 of the living room (figure 7), concerning a decoration near the ceiling, show the change of hematite to magnetite, a different phase of oxidation, probably caused by fire, and giving rise to a colour change from the original reddish to black.

The graphic of figure 10 shows the results of sample 19-08-16 (hematite) and the graphic of figure 11 shows the results of sample 19-08-18 (magnetite).

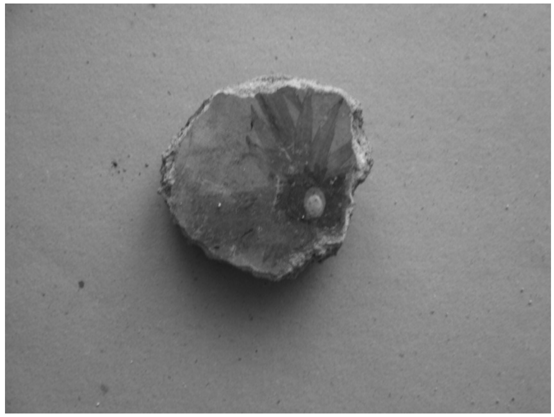

Fig. 4 Samples 19-08-10 to 19-08-12.

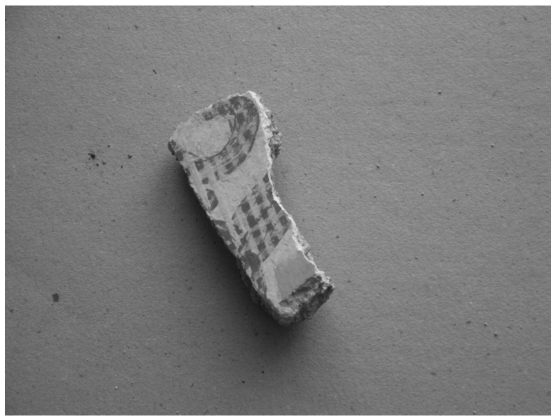

Fig. 5 Samples 19-08-01.

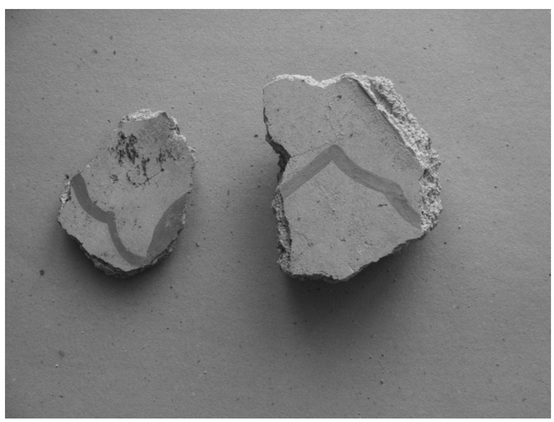

Fig. 6 Samples 19-08-6 to 19-08-9.

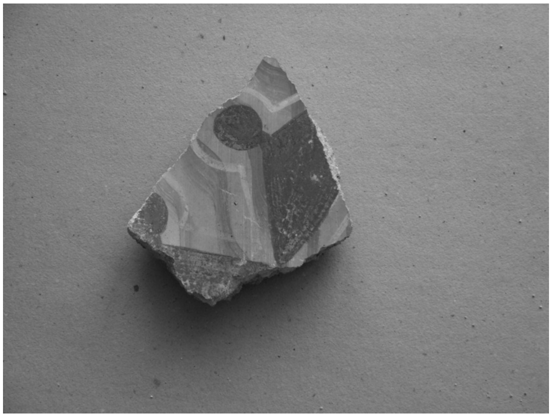

Fig. 7 Samples 19-08-16 to 19-08-18.

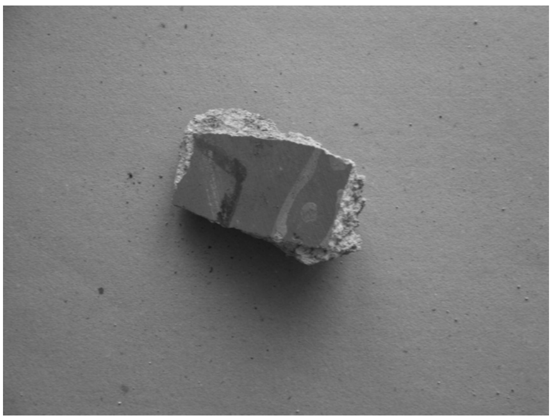

Fig. 8 Samples 19-08-13 to 19-08-15.

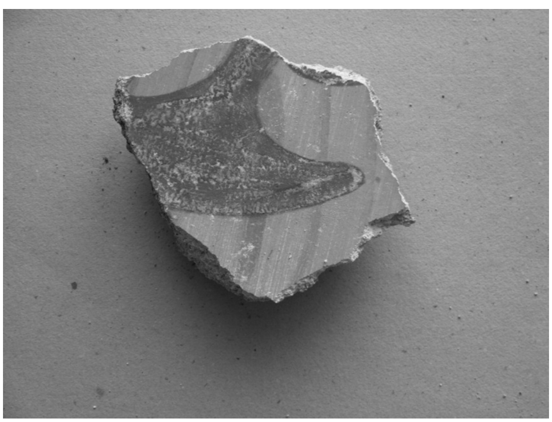

Fig. 9 Samples 19-08-4 to 19-08-5.

Mortars

Density and open porosity

The four collected samples were analysed for the determination of the apparent density and the open porosity. Before testing, samples were dried at 60 °C to constant mass. The test procedure was based on the European Norm EN 1936 [1], by total saturation with water under vacuum and hydrostatic weight. Results are presented in figure 12.

It may be observed that the mortars have very similar apparent densities and open porosities. Nevertheless, comparing mortars R6.1 and R6.1f and mortars St.1 and St.1f, there is a slight trend that may indicate that mortars subjected to high temperatures were altered in such a way that open porosity is slightly increased. This trend shall be further investigated with new collected samples both affected and non-affected by fire. On the other hand, in order to better evaluate this possible effect, non-affected mortars will be submitted to high temperatures in the laboratory and then tested.

Mercury porosimetry

Pore-size distribution was performed with a mercury porosimeter on each collected mortar. Samples were dried to constant mass at 60ºC. Two equivalent penetrometers were used with a 5 cm^3 bulb and a total intrusion capacity of 1.716 cm^3. Low pressure testing ranged from 0.0138 MPa (2 Psi) to 0.1979 MPa (28.7 Psi) and high pressure analysis from 0.2124 MPa (30.8 Psi) to 206.4063 MPa (29936.7 Psi). Equilibration times were 10 seconds for both low and high pressures. As mercury parameters, the following were used: advancing and receding contact angle = 140º; surface tension = 0.485 N/m; density = 13.5335.

Cumulative and incremental curves are plotted in figures 13 and 14. These plots represent the pore size diameter in microns and each step of mercury intrusion in percentage of total intrusion.

These bimodal pore-size distribution curves are typical of non-hydraulic lime mortars [2, 3]. This type of distribution is the result of a well interconnected macropore network that is formed as a result of the drying

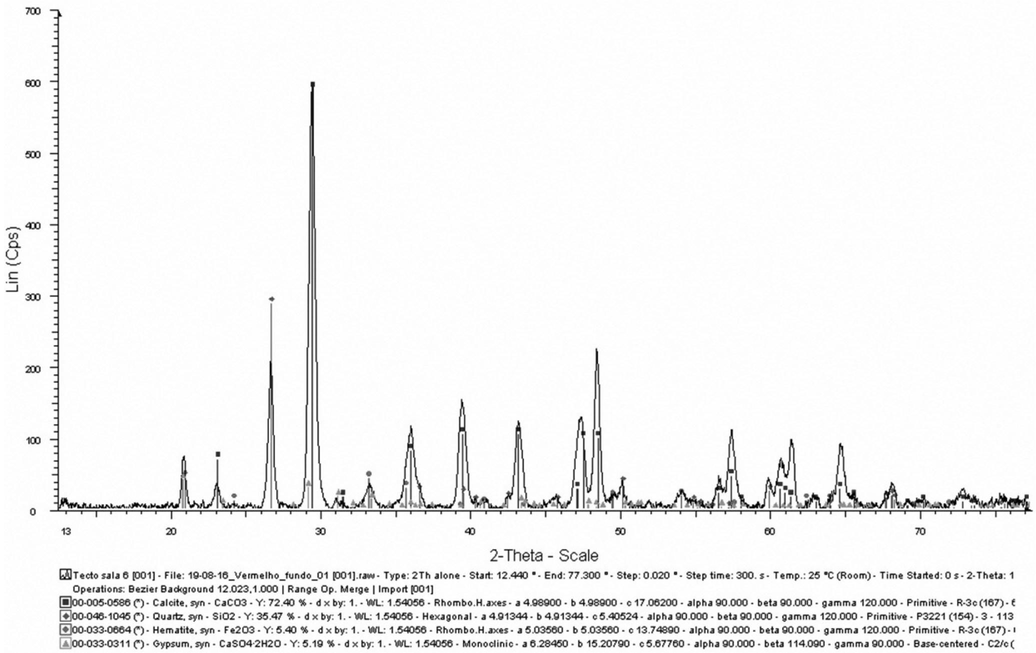

Fig. 10 X-Ray diffraction for specimen 19-08-16.

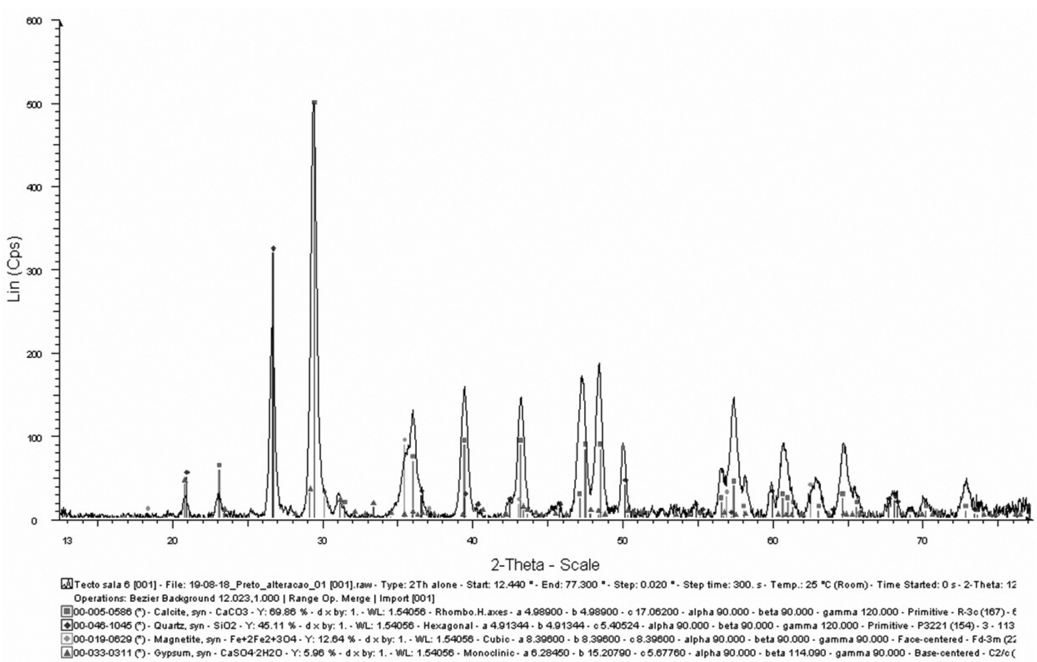

Fig. 11 X-Ray diffraction for specimen 19-08-18.

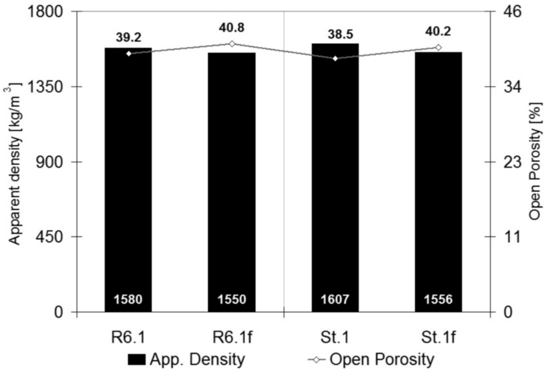

Fig. 12 Apparent density and open porosity of the mortars.

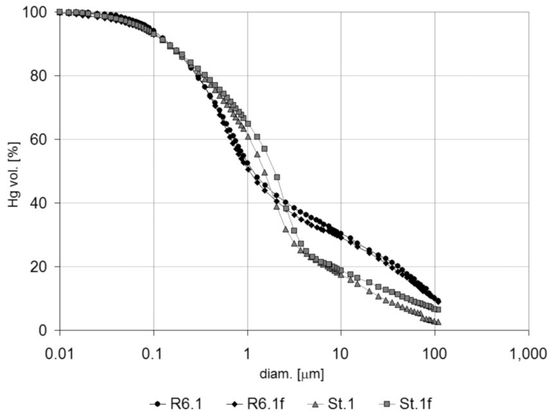

Fig. 13 Cumulative mercury intrusion curves.

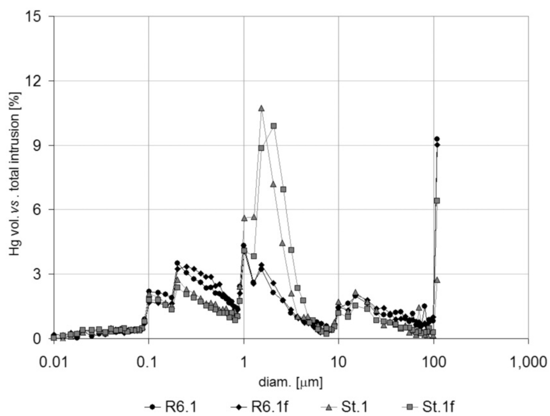

Fig. 14 Incremental mercury intrusion curves.

shrinkage of lime mortars [4]. This macropore network is more significant in mortars R6.1 and R6.1f. This can be confirmed by higher values in the first intrusion [5], corresponding, in this case, to the accessible and interconnected pores with diameters above 108 mm. In the range of the smaller pores, all of the mortars have important threshold diameters of 0.1 mm, 0.2 mm, 1.0 mm and 1.5 mm.

In a first approach, it can be said that the main differences in pore-size distribution are not related to the effect of fire. Mortars R6.1 and R6.1f have very similar intrusion curves and the same can be observed with mortars St.1 and St.1f. Therefore, these differences appear to be related to the area where samples were collected. It would not be surprising if mortars applied on different rooms of the building would have dissimilarities in their internal structures. As it is well known, this could arise from different manpower, curing conditions, amount of mixing water, etc.

One fact deserves however a specific reference: mortars St.1 and St.1f were collected from the area of the building that was the most affected by the fire. The significant distinction on the respective intrusion curves in the range of the 1.0 to 1.5 mm diameters is therefore a feature that will deserve a more detailed analysis together with the future tests of open porosity previously mentioned.

Binder : aggregate ratio

The identification of the binder : aggregate ratio was obtained by the method of dissolution of the carbonates with hydrochloric acid and weighting (table 3). Although there is no information about the origin of the sands used in the construction of the chalet, most probably local siliceous sands were used. Therefore the results of this particular test should not be affected because the sand has no calcareous elements in its composition.

Results for the two layers of the two samples that were analysed are very similar (1:4 and 1:5 by weight); it was not therefore possible to establish any distinction related to the effects of fire in this particular feature.

Table 3 Composition and binder : aggregate ratio of mortars.

Compounds	Room 6 Layer A	Room 6 Layer B	Staircase Layer A	Staircase Layer B
Calcium carbonate binder (%)	18.80	14.26	16.71	18.81
Aggregates (%)	74.67	77.45	73.48	72.58
SiO2 soluble (%)	3.95	3.01	4.46	3.35
Organic matter + H2O (Renderings) (%)	2.12	2.72	4.05	3.28
Total (%)	99.55	97.44	98.72	98.02
Indeterminated (%)	0.45	2.56	1.28	1.98
Binder : aggregate ratio (weight)	1:4	1:5	1:4	1:4

Conclusions

In what concerns the microstructural properties of the mortars, it seems in a first approach that there was not significant damage caused by fire. The effects of fire temperatures on the superficial plaster layer have probably caused a dehydration of the gypsum which, in turn, would lower the thermal conductivity. In this way, the mortar substrate would be less affected. On the other hand, it can be observed on the samples that the carbonation of the inner wooden structure of the walls has caused some superficial changes on the subsequent mortar layers. Nevertheless, the results obtained in this preliminary study show that these effects were not as serious as to cause important alterations in the material.

The effects of fire in the Chalet's fabric are not yet fully clear; however there are some interesting findings. Evidence gathered so far suggests that the fire has not reached enough temperature in order to alter significantly the physical properties of the mortars, but it caused interesting alterations to the integral decorated surfaces. Pigments analysed showed colour changes indicating slow combustion of the fabric, eventually up to 400 °C, judging by the phase change of the hematite to magnetite. Additionally, the relative stability of mortars indicates that temperatures were not high enough to alter the calcium carbonate, otherwise there would probably be some cracking in the internal renders and detectable changes in the properties of their microstructure.

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Methodology for diagnosis of rendering anomalies due to moisture in walls

Metodologia de diagnóstico de anomalias de rebocos exteriores devidas à humidade

Ana Cristian Magalhães
Civil Engineer/ Laboratório Nacional de Engenharia Civil, Portugal,
acristian@lnec.pt

Maria do Rosário Veiga
Civil Engineer/Laboratório Nacional de Engenharia Civil, Portugal,
rveiga@lnec.pt

C. Pina Santos
Civil Engineer/Laboratório Nacional de Engenharia Civil, Portugal,
pina.santos@lnec.pt

Luís Matias
Physicist/Laboratório Nacional de Engenharia Civil, Portugal,
lmatias@lnec.pt

António Vilhena
Civil Engineer/Laboratório Nacional de Engenharia Civil, Portugal,
avilhena@lnec.pt

Abstract

Moisture is one of the most powerful causes of decay of historic buildings. A lack of understanding concerning the action of moisture and the resulting degradation mechanisms are often preventing efficient repair of ancient walls. Thus, there is a need to develop and systematise a methodology of diagnosis of defects due to moisture, taking into account the characteristics and properties of materials used on those walls. The use of in situ test methods, especially non-*destructive*invasive methods, is an important tool, but those methods must be adapted to this particular use, calibrated and interrelated in a global approach. As a result of a Research Project developed at LNEC a methodology involving several phases is proposed, based on tests applied to several case studies and on the analysis of their results. This work synthesises the proposed methodology presenting examples of data combinations for the suggested techniques and its interpretation in order to characterise the state of conservation of renderings and to estimate their performance.

Keywords

Renderings; ancient walls; anomalies; diagnosis; moisture.

Resumo

A humidade é uma das mais poderosas causas de degradação de edifícios históricos. O desconhecimento da acção da humidade e dos mecanismos de degradação dela resultantes impedem frequentemente uma reparação eficaz de paredes antigas. Assim, é necessário desenvolver e sistematizar uma metodologia de diagnóstico das anomalias devidas à humidade, tendo em conta as características e propriedades dos materiais usados nessas paredes. O uso de ensaios in situ, especialmente de métodos não-destrutivosinvasivos, é uma ferramenta importante, mas tais métodos devem ser adaptados a esta utilização, calibrados e inter-relacionados numa abordagem global. Como resultado de um Projecto de Investigação desenvolvido no LNEC, propõe-se uma metodologia envolvendo várias fases, baseada em ensaios aplicados a vários casos de estudo e na análise dos respectivos resultados. Este trabalho sintetiza a metodologia proposta, apresentando exemplos de combinações de resultados para as técnicas sugeridas e para a sua interpretação, de forma caracterizar o estado de conservação dos revestimentos e a avaliar o seu desempenho.

Palavras-chave

Rebocos; paredes antigas; anomalias; diagnóstico; humidade

Introduction

Exterior renderings of ancient buildings often present several defects which have moisture as main origin. In fact, water is a primary cause of many anomalies and secondary cause of many others. Moisture in walls of ancient buildings can have diversified causes: groundwater, rain, inner condensation, presence of soluble hygroscopic salts, rupture of piping, obstruction of gutters or of fall pipes, etc.

The knowledge of the origin of anomalies due to moisture and of the resulting mechanisms of degradation of affected renders is essential to provide a correct diagnosis. In this sense, the detection and the quantification of anomalies resulting from moisture in walls of ancient buildings must be carried out using a set of different techniques, whose results, considered together, supply important data regarding the state of conservation of renderings [1, 2].

The characterization of walls' materials is essential to understand the degradation mechanisms. The literature has presented several studies in this field [3-9].

A study aiming to systematize and to apply a set of available characterization techniques of materials was developed in order to integrate them on a reliable methodology for diagnosis of damage of ancient walls, in particular those defects directly associated with moisture [10].

The objective of the diagnostic is the gathering of information allowing the elimination or minimization of the damage mechanisms and, in particular, the definition of a repair strategy for the wall renderings.

The techniques were selected based on the analysis of results obtained on several case studies (Santa Marta Fortess, Oitavos Fortress, Évora Cathedral, Inglesinhos Convent, described in previous work [4, 10-15]), taking into account criteria related with complementarity – evaluation as complete as possible of mechanical, physical and chemical aspects – limitation of destruction – and practicality – easy and quick application, low cost, low specialization of operators (as much as feasible).

The proposed methodology involves the following phases:

- general observation of the building and registration of the main anomalies;
- detection of problematic zones using non-destructive *in situ* test methods of global analysis;
- quantification of damage of mechanical, physical and chemical nature, using non-destructive test methods of localized application;
- whenever necessary use of destructive test methods to complement the quantification of degradation;
- classification of the state of conservation of renderings;
- establishment of a diagnosis;
- definition of a repair strategy.

Methodology

The methodology, synthesized on figure 1, concerns a set of available techniques and their interpretation and correlation of results.

General observation of the building and its renderings

A general observation of the building and its renderings, carried out by an expert observer, allows to identify and to register the symptoms and their severity, establishing a map of anomalies with a classification by type.

The visual observation must always be complemented by photographic records. It is important to include data concerning extrinsic conditions to the building (orientation, solar radiation incidence, predominant wind, rain intensity, medium and extreme values of temperature and relative humidity) in order to predict how they affect the materials and how they contribute to the degradation process.

The type of rendering must be identified in this process, as well as its composition and technique of production and application – coats, thickness, texture and colour – implying a first selection of low deteriorated zones for this observation. Rendering from high degraded zones must also be observed in order to collect information for the evaluation of the degradation mechanisms. In subsequent phases, those zones, identified as low deterioration and high deterioration zones, can be used for the application of *in situ* tests and, if necessary, to collect samples for laboratory analysis, allowing for characterisation of the material – low deterioration zone – and for deterioration study – high deterioration zone.

A preliminary damage diagnosis based only on the observed symptoms may be possible at the first phase. If

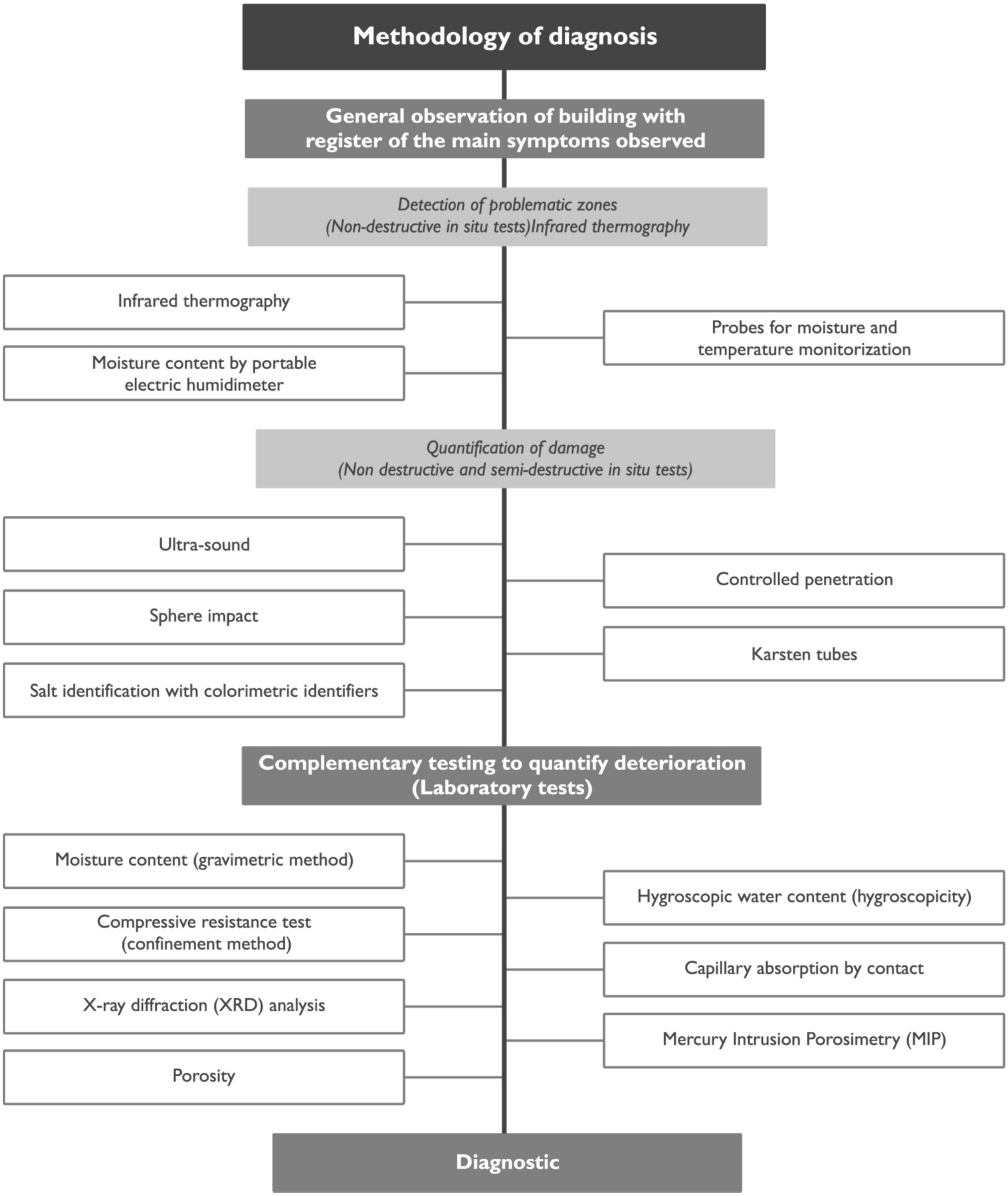


Fig. 1 Proposed methodology for diagnosis of wall renderings' defects due to moisture.

this is not possible, the diagnostic should be substantiated by an extended study, using experimental characterization techniques [16].

Detection of problematic zones using methods of global analysis

After the visual analysis, a study orientated for the location of problematic zones is carried out by resource to test methods allowing a global analysis of the wall. *In situ* wide spectre non-destructive techniques are used, aiming to detect anomalies and to trace the degradation symptoms.

The global analysis test methods referred in this paper – infrared thermography, monitorization of moisture and temperature with miniature probes and with a portable electric humidimeter – aim essentially at the identification and the quantification of areas with high water content, the assessment of the distribution of water inside the wall and the follow up of the evolution of water content along time.

Quantification of damage using methods of localized application

The detected anomalies of mechanical, physical or chemical origin can be quantified through methods of local application, which supply information concerning intensity and level of degradation.

In general, previously to the tests, it is advisable to systematize information concerning renderings characteristics and their degradation level based on visual analysis, in order to improve the interpretation of the results and their representativity. In fact, even if results are quantified, they are always comparative values and they must be evaluated considering the type of mortar.

As much as possible, test areas must be selected taking into account the diversity of characteristics and the existence of sufficiently regular areas to perform the tests in the best possible conditions.

The most relevant anomalies of the renderings must be selected in each zone and classified according to the degree of degradation produced. Loss of adhesion to the support or loss of cohesion are often the predominant anomalies, because they are particularly difficult to repair. Tests should be carried out in zones with different degradation types and levels in order to compare results.

For the quantified evaluation of damage related to mechanical characteristics some *in situ* tests can be used: ultrasounds, sphere impact and controlled penetration. For the evaluation of the water behaviour the Karsten tubes method is proposed. Colorimetric identifiers can be used for the detection of soluble salts.

Complementary quantification of degradation

For clarification of some aspects concerning characterisation of materials, study of degradation products and quantification of damage, the use of complementary techniques of diagnosis can be necessary, requiring the extraction of samples of ancient renderings. In general, these techniques are carried out in laboratory, allowing rigorous determinations.

The set of proposed techniques are: moisture content by gravimetric method, hygroscopic water content (hygroscopicity), compressive resistance test (confinement method), capillary absorption by contact, mercury intrusion porosimetry (MIP), porosity (hydrostatic pressure method), X-ray diffraction (XRD) analysis.

The majority of these tests is currently applied in characterisation or in damage diagnosis of ancient buildings, so their description is available in the literature. However, some of the tests were specially developed or adapted at LNEC to be used with mortar samples extracted from old buildings; they were calibrated and described in previous works [6, 7, 11].

Evaluation of test results

In general the application of the referred techniques to ancient mortars is relatively complex due to different factors like typology and heterogeneity of the materials. Therefore the data obtained in each test must be used as comparative and not absolute values.

Some tests used in the methodology are illustrated on figures 2 to 4.

In table 1 the main limitations and advantages of the techniques are presented.

The interpretation of the results of the described test program, applied in a systematic way, by a specialized team, enables a safe classification of the state of conservation of ancient renderings, supporting eventual interventions to be carried out.

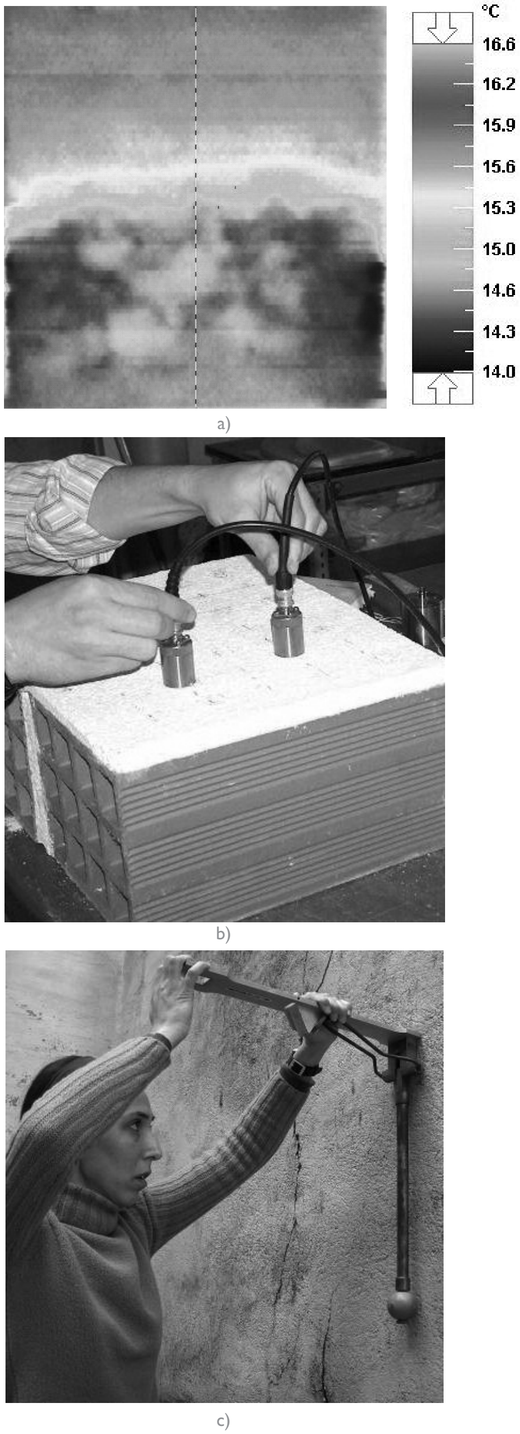


Fig. 2 a) infrared thermography; b) ultra-sound; c) sphere impact.

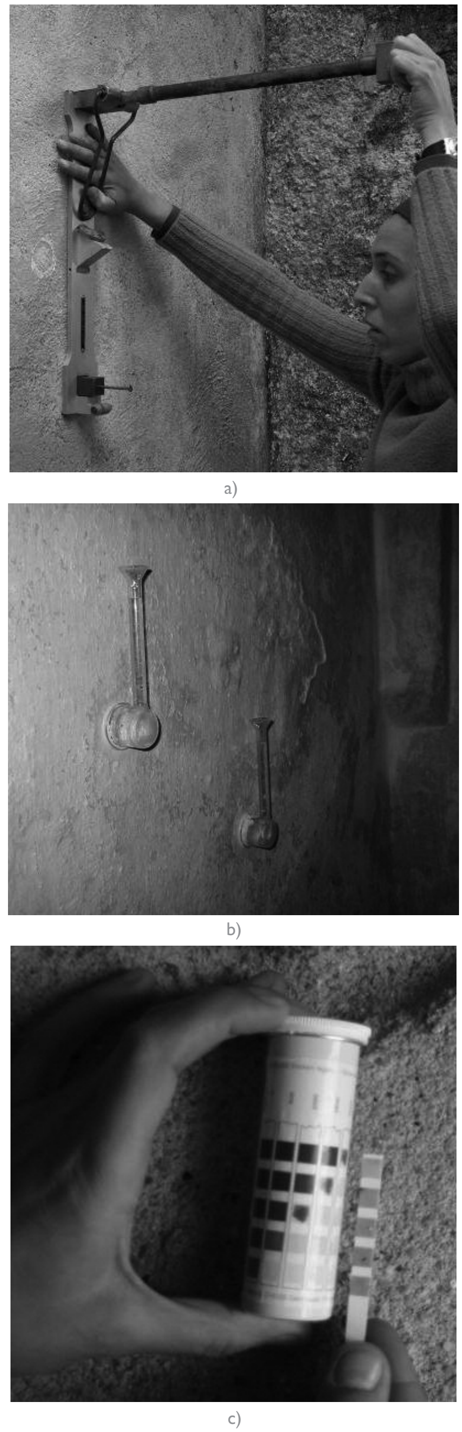
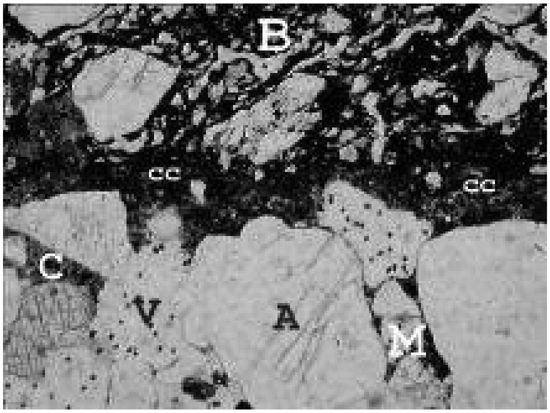
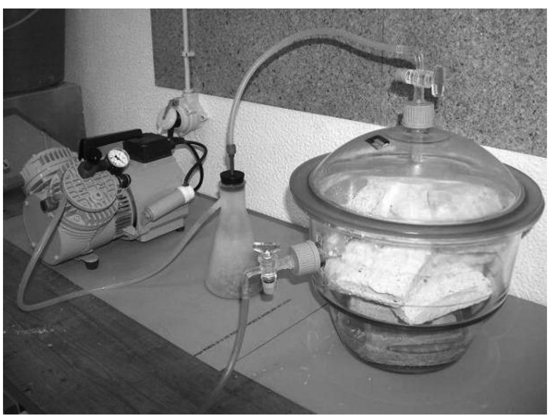


Fig. 3 a) controlled penetration; b) Karsten tubes; c) Salt identification with colorimetric identifiers.



a)

b)



c)

Fig. 4 a) compressive resistance test (confinement method); b) capillary absorption by contact; c) determination of open porosity.

Establishment of a diagnosis

The location of damaged zones and the identification and quantification of zones with high water contents by non-destructive techniques of wide spectre - infrared thermography or techniques of moisture monitorization - allow to detect and to follow the evolution of water presence in the interior of the walls during long periods. It also permits to identify with some confidence the water's origin.

Very wet zones of renderings can present high levels of degradation, namely with loss of cohesion and adhesion and increase of the porosity. The tests of sphere impact, controlled penetration, ultra-sounds and Karsten tubes allow to establish a classification of the state of conservation, related with reduction of mechanical characteristics and increment of porosity. The existence of soluble salts such as sulphates is a cause of expansions in the renderings, producing powdering, loss of adhesion or detachment. The colorimetric identifiers (strips) allow to detect the presence of ions in the mortar and to determine their concentration in a semi-quantitative way. Through the correlation of the test results it is possible to confirm if there is a real correspondence between renderings with high water content and high degradation level (generalized loss of cohesion, bad adhesion, significant increase of water permeability, very low strength, etc.).

In the next phase, constituted by laboratory tests, the rendering materials characteristics are carefully quantified and the reaction products are identified in order to make possible the understanding of the degradation mechanisms.

The gravimetric method for moisture content determination complements the portable electric humidimeter measurements, allowing to quantify moisture inside the mortar and making possible the establishment of moisture profiles.

The values of hygroscopic moisture can reveal the existence of contents of soluble hygroscopic salts inside the mortar, and to check if these salts are the main cause of the moisture levels. Reduced values of the hygroscopic moisture are not due to the presence of hygroscopic salts, and point out to moisture originated by infiltration through the roof, through wall cracks or by rising capillarity through the interior of the wall.

Table 1 Main advantages and limitations of techniques.

Technique	Objective(s)	Advantages	Limitations
Infrared thermography	Detection of wet surface and internal zones through the evaluation of the distribution of temperatures in the wall surface	Non-invasive *in situ* test; evaluation of the states of wetness and of conservation of large areas of rendering; quick use; possibility of use in places of difficult access	Need of water evaporation or imposed heating to produce temperature differentiation, therefore, in practice the technique gives best results in dry, or sunny periods and in walls with solar significant exposition; alternatively, it needs artificial heating; method is sensitive to the presence of soluble salts, which can influence evaporation and the capacity of moisture detection [17]
Moisture and temperature monitorization with probes	Continuous measurement of moisture and of temperature near the surface or in the interior of the wall (probes inserted in drilled holes)	Possibility of assessment of evolution of the rendering hygrotermal state; complementary to thermography	Invasive test; imprecise measurements when the holes' air is saturated
Moisture content measurements with portable electric humidimeter	Evaluation of water contents on the mortars' surface	Non-invasive *in situ* test; quick and practical use for the detection of zones with high and medium moisture content	Need of a great number of careful and systematized measurements; sensitive to the presence of salts in the mortar, which can modify the results
Ultra-sonic velocity measurements	Location of possible damaged zones through reduction of ultra-sound waves velocity; complementary to other data concerning the materials mechanical resistance and stiffness	Non-invasive *in situ* test; quick and practical use; useful for assessment of the state of conservation and also for the evaluation of compatibility of substitution renderings	Interpretation of the results requires particular care and experience; the technique requires previous calibration for each type of mortar
Sphere impact	Information regarding rendering's deformability and mechanical resistance	Quick *in situ* tests of easy interpretation; no demand of specialized technicians	Invasive tests; the obtained information is localized, requiring a series of measurements in different zones
Controlled penetration	Evaluation of mechanical resistance of internal coats		
Karsten tubes	Evaluation of water permeability. Qualitative information of the rendering's state of conservation	Non-invasive *in situ* test; quick technique, of use *in situ* and in laboratory	Possible limitations of representativeness of *in situ* test due to the presence of paintings or thin organic finishing or due to high water content of the mortar, preventing further water absorption.
Salt identification with colorimetric identifiers	Identification of a specific ion	Non-invasive *in situ* test; very simple test, of easy and quick use both *in situ* or in laboratory	Very localized test; need of several determinations in the same zone
Moisture content by gravimetric method	Determination of the mortar water content	Possible use of samples of any shape and dimensions	Destructive laboratory test; it requires special care in gathering and storage of sample in order to avoid losing moisture before the first weighing

Technique	Objective(s)	Advantages	Limitations
Hygroscopic water content	Determination of the hygroscopic mortar water content and estimation of hygroscopic soluble salts content	Possible use of samples of any shape and dimensions	Destructive laboratory test; the method requires a systematized gathering of samples, from different heights and depths of the wall; results can be masked by the impossibility of complete extraction of the finishing coats
Compressive resistance test (confinement method)	Information concerning the mechanical resistance of mortars	Possible use of small irregular samples	Destructive laboratory test; the confinement mortar must be stronger than the sample to test in order to obtain rupture by sample but nevertheless there is a possibility of some influence of the confinement mortar on the results
Capillary absorption by contact	Determination of the coefficient of capillarity by contact (Ccc)	Possible use of irregular samples	Destructive laboratory test; the test requires relatively large quantity of sample; the results can be affected by the shape of the sample, especially in the case of very thin specimens
Porosity	Information about open porosity of the mortar	Possible use of small samples without special shape demands	Destructive laboratory test; porosity value does not characterize by itself the material porous system; it requires to be complemented by other techniques, such as MIP
Mercury intrusion porosimetry (MIP)	Estimated quantification of the size distribution and the volume of pores	Possible use of small irregular samples	Destructive laboratory test; high cost of the equipment; demand of specialized technicians; health risk for operators due to the possibility of contamination with mercury; interpretation requires knowledge concerning porous structure
X-ray diffraction analysis (XRD)	Information regarding the type of binder. Detection of the presence of pozzolanic constituents and of alteration products	Possibility to carry out qualitative and crystallographic analyses of any crystalline components; requires a small quantity of sample reduced to powder or in rough state.	The technique does not allow to observe the inter-space relation of the mortars components; quantitative information is of limited precision; the equipment is expensive and requires a specialized operator

The analysis of the results of compressive resistance tests using the method adapted for irregular mortar samples (confinement method) allows to compare the strength of ancient samples to mortars of known behaviour; low resistance points out to poor cohesion but it may be a characteristic of the type of mortar or a symptom of a degradation process.

Coefficients of capillarity determined by the test of capillary absorption by contact allow to compare the speed of absorption of the existent mortars to new mortars of known behaviour. Together with the values of mechanical resistance they allow a picture of the state of conservation: reduced coefficients of capillarity and relatively high mechanical resistance indicate mortars in good condition, cohesion and adhesion. On the other side, absorption and drying curves allow an analysis of how the absorption and drying of water take place in the interior of the mortar, complementing the water permeability data obtained with the Karsten tubes.

The application of XRD on mortars allows the identification of main mortar constituents and supplies information on the presence of products related with the development of mechanisms of degradation [14]. This information, combined with the results of compressive resistance and of capillary absorption, contributes to the identification of degradation mechanisms, complementing the data regarding the state of conservation of renderings.

The pore size distribution obtained through the mercury intrusion porosimetry and the values of open porosity, in combination with the results of compressive strength, Karsten tubes, capillary absorption and hygroscopicity complement the evaluation of the mechanical behaviour and water behaviour of the mortar. The relation between the composition – determined by XRD analysis – and mercury intrusion porosimetry allows to appraise the degradation process of the mortars generated by the presence of salts. Higher porosity increases the total absorption and larger pore dimensions induce higher water absorption coefficients. However the degradation can be more significant in mortars with a larger volume of smaller pores, since higher pressures are produced in these pores during the process of salts crystallization, which takes place with volume increase. Smaller pores are also in the origin of higher rising capillarity. On the other hand, mortars with larger pores are weaker and they have higher total absorption. So, when repair mortars are formulated, a good balance between these characteristics is not easy to achieve.

Definition of a repair strategy

The strategy to adopt to solve problems related to moisture in renderings of ancient buildings with historical value must give priority to the preservation of the existent renderings (instead of removing and replacing them), with resource to conservation strategies supported by *maintenance plans*, *local repair* and *consolidation techniques*.

However, in the case of high severity anomalies, reduced value of the building and insufficient means, the strategy of intervention can pass by the option of partial or total *substitution of renderings*, imposing the previous elimination of the causes of anomalies, or at least their minimization, made possible by the first phase of the methodology of diagnosis applied: detection of problematic zones. Hence, the strategy of removal and replacement of historical mortars must be the last hypothesis to consider, only when the preservation strategies are not possible, and supported by the results of the applied methodology, namely by its second and, if necessary, third phases: quantification of degradation.

Maintenance includes the following operations: cleaning, treatment with biocides, correction of situations causing water infiltration, repair of finishing coats and *fulfilment* of superficial cracks.

Localized repair comprehends operations of treatment of cracks, salts elimination and filling out of lacunae, using materials similar to the existent ones.

Consolidation consists on the use of groutings in renderings with bad adhesion and cohesion consolidants when cohesion is insufficient.

Partial or total substitution of renderings implies the use of substitution materials compatible with the existent ones to avoid the risk of degradation increase.

The diagnosis of anomalies and the characterisation of the state of conservation of the renderings are important tools in the definition of strategies of intervention to adopt in ancient buildings with historical value.

Clear criteria must be defined to decide between i) repair of existent renderings, ii) partial or total substitution with compatible materials or iii) more onerous and complex options, involving consolidation (restitution of lost adhesion or of lost cohesion). The decision must be based on the following factors: value of the building and of the rendering; state of conservation of the rendering; availability of means in terms of technology, workmanship, time and budget allowances.

Conclusions

The described methodology proposes the evaluation of the renderings anomalies due to moisture in ancient walls by the simultaneous application of different techniques, whose connection of results supplies important data regarding the state of conservation of a rendering.

According to the proposed methodology, the investigation of the building must begin with a *general observation* and with the *register of the main anomalies and their severity*. After this first observation, a *global analysis* of the wall through *in situ* techniques of evaluation, allowing a more precise detection of the zones with problems, is proposed. Next, it is proposed a *localized analysis* of the anomalies using techniques of quantification and finally, for the establishment of the diagnosis, the use of laboratory techniques to explain some aspects and *complement the quantification* of the identified anomalies.

The experience shows that the systematized application of the proposed methodology makes possible the most rigorous determination of the qualitative and quantitative characterization of the renderings anomalies and their state of conservation.

The correlation of results obtained with the different techniques must lead to conclusions concerning the degradation mechanisms caused by moisture and their evolution allowing a classification of the state of conservation [4, 11, 18] of the rendering.

Considering the detection of causes and the identification, localization and quantification of anomalies, permitted by the methodology of diagnosis, an intervention methodology can be established based on conservation strategies including maintenance plans, techniques of localized repair and consolidation, preceded by the correction of causes and elimination of degradation mechanisms.

Repair and substitution materials must be selected fulfilling compatibility criteria with the pre-existent components, established taking into account both ethical conservation principles and the knowledge of composition, characteristics and behaviour of pre-existent materials provided by the described methodology.

Acknowledgment

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Cause of decay and intervention on external mortars of the S. Vittore Church - Stresa (Italy)

Causas de decaimento e intervenção nas argamassas exteriores da Igreja de S. Vittore - Stresa (Itália)

Massimo Valentini
Engineer, Politecnico di Milano - Department of Energy, Milan, Italy, massimo.valentini@polimi.it

Raffaelo Visentini
Architect Freelance, Lesa (NO), Italy, r.visentini@tiscali.it

Abstract

The ecclesiastical complex entitled to S.Vittore rises on the Pescatori Island in the Lake Maggiore in the northern Italy. The building was constructed in the X century and was afterwards widened in the XVII century. Inside the several steps in which the total plan of restoration of the church is divided (the diagnosis and the elimination of the causes of decay, the restoration of the external mortars and of the internal frescoes, the control of indoor environmental conditions), this work concerns the preliminary diagnostic and cognitive investigations and the subsequent intervention on the external mortars. The initial investigations have been addressed to understanding the causes of decay of the building and to the characterization of the external mortars. It was so verified that the main cause of decay is the water present in masonries coming from the meteoric water rather than from rising damp through the ground. The characterizations of the external mortars have shown that visible differences between the ancient (X century) and the more recent masonry parts result from the different binder--aggregate ratio, rather than from the mineralogical-petrographic composition. Following the results of this first step of the investigations, the rain water-drainage of the ground around the church has been carried out in order to collect and to remove the meteoric waters; the collection and removal system of the rain-waters has also been arranged. With regards to the external plasters, to ensure the visual, material and functional compatibility with the existing mortars it has been carried out the sealing and integration of existing mortars and plaster with compounds chosen on the basis of the analysis results and of the local constructive traditions. "Protected" in this way the external covering, the plan will continue with the internal restorations.

Keywords

Diagnostic investigations; external mortar; rising damp; drainage.

Resumo

O complexo eclesiástico consagrado a S.Vittore eleva-se na ilha dei Pescatori, situada no Lago Maior, no Norte da Itália. O edifício foi construído no século X e posteriormente ampliado no século XVII. No âmbito dos diversos passos nos quais se divide o plano global de restauro da igreja (diagnóstico e eliminação das causas de degradação, restauro das argamassas de exterior e dos frescos no interior, controle das condições ambientais no interior), este trabalho diz respeito ao diagnóstico preliminar e investigações cognitivas e à subsequente intervenção sobre as argamassas de exterior. As investigações iniciais foram dedicadas a compreender as causas de degradação do edifício e à caracterização das argamassas de exterior. Foi, deste modo, verificado que a principal causa de degradação se deve à presença nas alvenarias de água sobretudo proveniente da chuva (água meteórica), mais do que da humidade ascensional a partir do solo. A caracterização das argamassas de exterior mostrou que as diferenças visíveis entre as alvenarias mais antigas (século X) e as mais recentes resultam de rácios ligante-agregado diferentes, mais do que da composição mineralógico-petrográfica. De acordo com os resultados deste primeiro passo da investigação, foi efectuada a drenagem da água da chuva no solo que circunda a igreja, com o objectivo de recolher e remover as águas meteóricas; foi adicionalmente colocado um sistema de recolha e remoção das águas pluviais. No que diz respeito aos rebocos exteriores, para assegurar a compatibilidade visual, material e funcional com as argamassas existentes, foram efectuadas a selagem e integração das argamassas e rebocos existentes com compostos escolhidos com base nos resultados das análises e nas tradições de construção locais. Com o revestimento exterior "protegido" desta forma, o plano prosseguirá agora com os restauros no interior.

Palavras-chave

Investigações de diagnóstico; argamassa de exterior; humidade ascensional; drenagem.

Introduction

The restoration of an historical building must always be preceded by a careful and complete phase of instrumental diagnostic and historical-bibliographical search to estimate the state of decay of the building, to know the decay mechanisms and the materials and techniques used for its construction. To this first phase of knowledge, it will follow the elimination (or reduction) of the causes of decay of the building, the restoration of the building and, where it is possible or necessary, the indoor conservation conditions improvement of the building and of its contained works of art.

All these steps have been followed in the plan of restoration of the S. Vittore Church. The article describes the phases finished up to now: the research of the causes of decay, the intervention on main of these and the restoration of the external plasters of the church.

The intervention plan

The building and its state of conservation

The S. Vittore Church is a small dimensions building (width 10.8 m - length 16.2 m) that rises on the rocks bench that forms the small Pescatori Island in Lake Maggiore, always crowded by many visitors.

Of the first single nave Romanesque oratory of the last quarter of X century, originally dedicated to S. Gandolfo, remain the apse and parts of the boundary walls, pointed out towards the north-west and the south-west, then absorbed in the successive widening of the church.

The cover of the Romanesque portion had to be constituted from a hut roof of tile, supported by a wood truss (some tiles of remarkable dimensions are still visible on the fronton, absorbed in the more recent masonry).

Beginning from the 1500's, the Romanesque church was entitled to S. Vittore and underwent a series of transformations that reduced the apse to one of the lateral naves of the new church constructed on it, partially using its masonries. Between the 1600's and the 1700's, the church underwent further enlargements.

The external masonries of the church are plastered, except for a portion of the Romanesque part exposed to the north-west with cutstones at sight and only some traces of original plaster (Figure 1).

The church contains a series of frescoes, valuable marble altars, a pulpit in inlaid wood of walnut and four busts of Saints of the Church.

One of the main aim of the restoration is to bring back to light a series of frescoes, at the moment covered by a white washing layer, discovered in a small room on the south-west side of the Romanesque portion; the room (on whose external wall is recognizable the ancient opening of approximately 2 m overhang from an arc delimited by "roman bricks" emerging from plaster) has been up to now used as a warehouse. The plan previews the partial demolition of the dividing wall in order to allow the full fruition of frescoes as it had been thought to the age of their realization.

Fig. 1 The north-west side of the Romanesque part of the church.

The visual investigations and the observation of the macroscopic state of conservation of the building and its surfaces allow two reflections that have partially directed the following successive cognitive investigations. On one side, it is obvious the distinction between the more ancient part and the following interventions: testified by the comprehensive view of the whole building and the different renders of the external and internal walls. On the other side, the internal surfaces of the Romanesque part of the building show the greater decay, with plaster lacunae, rising damp with white efflorescence and the erosion of the columns of the two lateral altars and of the baptistery plaster. The external plasters show many concrete patches and a kind of decay due to the atmospheric and structural conditions: lacunae, small cracks, biological growth.

The plan themes

The aim of the plan is the conservative restoration of the monument. The interventions have been lead in the respect of the restoration guidelines, with special attention to the following disciplinary principles:

- respect of the existing for its documentary value;
- least intervention: the removal of every element of the building has been limited to the minimum indispensable;
- chemical-physical compatibility of all products used for the interventions;
- reversibility: the materials and the procedures have previewed, within feasible limits, the possibility to return to the status-quo preceding the intervention.

Diagnostic investigations: methodology of the measurements

Diagnostic instrumental investigations have followed three parallel paths: the measure of the hygrometric state of masonries, the monitoring of indoor microclimatic conditions and the characterization of the external plasters.

The hygrometric state of the masonries is characterized from the values (and their variations in time) of water content in walls and of the soluble salts content. The water content has been measured with the gravimetric method [1] applied to the samples extracted from masonries at two different depths from the surface and at various quotas from the floor level (sampling from the internal surfaces has been limited to the single quota of 10 cm). The measure, slightly invasive, has been carried out in 9 Points of measure on the internal walls and in 11 Points of measure on the external walls (Figure 2) and repeated in four days chosen like representative of the various seasonal periods: 29 September 2003 - 5 February 2004 - 28 May 2004 - 10 September 2004.

Between two subsequent water content measures, the Permanent Points of Measure (PPM) method has been applied; by a brick cylinder put in the hole made in the first day of measures with the gravimetric method, PPM method allows the qualitative estimation of the water content in masonries, avoiding to remove other material from the building. [2].

The contents in soluble salts with ionic chromatography were also analyzed [3].

Indoor microclimatic conditions monitoring has been carried out to estimate both the eventual negative winter heating effects and to collect reference data to check if the future interventions already planned (the installation of a walls dehumidification system and the elimination of the wall of the frescoed room of the

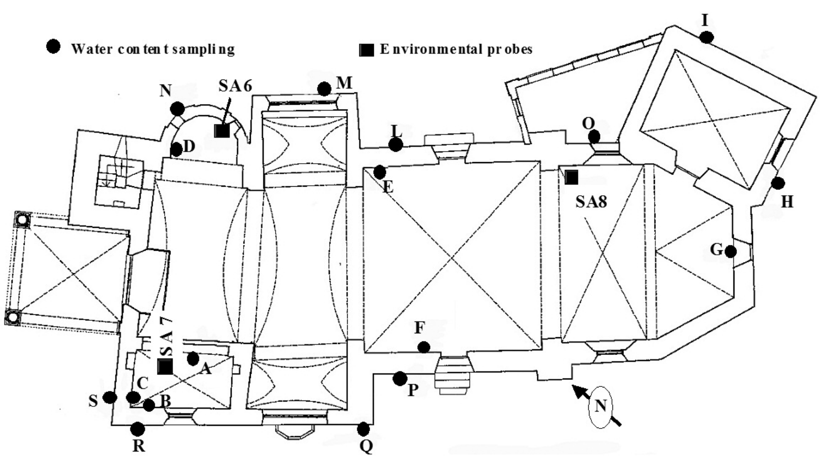

Fig. 2 Position of the zones of masonries water content measures (in some zones sampling has been carried out at various quotas from the floor plane) and of the probes for the indoor conditions monitoring.

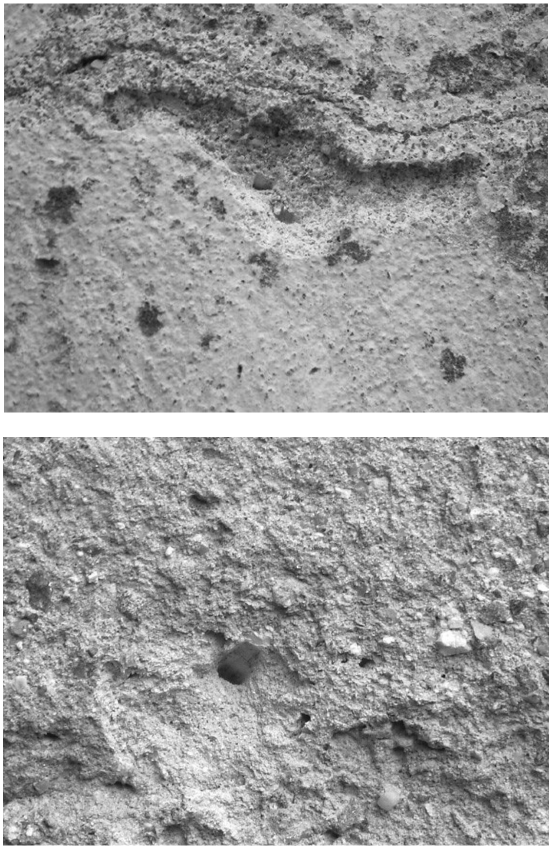

Fig. 3 The different appearance of the older plasters (above) and recent one (below).

Romanesque portion) will have effects on the environmental air conditions.

The analyses on the external plasters had analytically to explain the visible difference between the older plaster (in the Romanesque part of the church) and those most recent in the rest of the building (Figure 3) and to characterize the bedding mortars of the Romanesque part. Sampling was carried out taking into account minor damage to the walls; for this reason, rendering pieces were obtained at edges where detachment was initiating on three sides of the church (excluding the facade). In order to obtain as complete as possible a characterization so as to enable later execution of similar mortars based on the information gathered, X-Ray diffraction (to determine the mineralogy) and observation of thin section by optical microscopy with transmitted and polarized light (to check mortar's microstructure) were carried out.

Results

Water content in the walls

The water is distributed in a non uniform way in the various inquired walls. The average water content values (measured in all points of measure of the Figure 2 at 10 cm height from the floor plane) are shown in Figure 4.

The Romanesque portion of the church shows the greater water contents in masonries (Figure 5) without

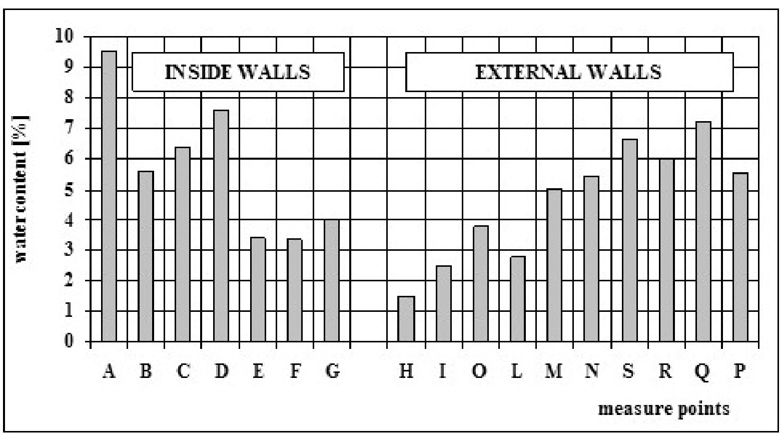

Fig. 4 Average water content.

difference between the different sides of the building. The result is in accordance with the greater decay of this part of the building drawn above.

PPM method has allowed to verify the variations in time of the values up to here indicated in absolute terms. The Romanesque part of the church has also the greatest water content values: the water in the walls keeps high and almost constant for great part of the year. In the rest of the church, on the contrary, the water contents are low. The outdoor walls show a greater variability in accordance with the unsteady evaporation caused by air movement (Figure 6).

The geologic inspections leave out the hypothesis of water stagnations or phenomena of capillarity in the cliff below the church. The water distribution in the masonries and its variations in time can then be explained by the different hygrometric behaviour of the materials of the Romanesque part regarding the rest of the building and by the presence of an "external source" of water fed by meteoric water that falls in the strip of land that runs along the building.

The "external source" is also deducible by salts contents analysis. The eighteenth-century portion of the church shows high nitrate contents that, by way of the water transport, come from the outside cemetery that completely encircles the apse.

Also in this case, a difference between the two constructive ages of the building is found, since the walls of the Romanesque portion have a higher content of sulphates: the efflorescences taken from the surface of the

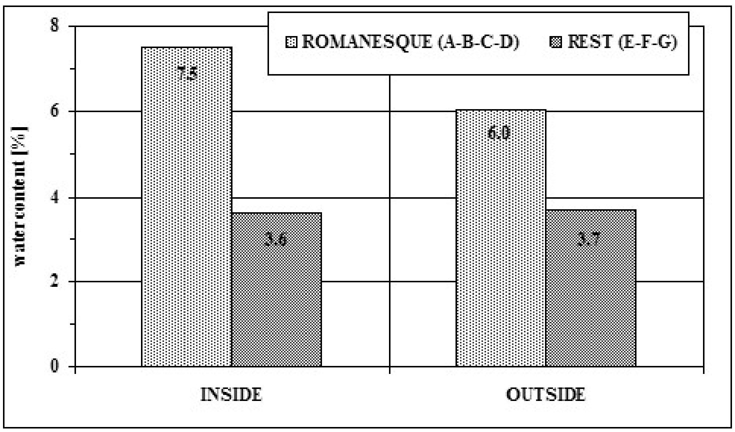

Fig. 5 Comparison between the average values of water content of masonries measured in the Romanesque part and in the rest of the building.

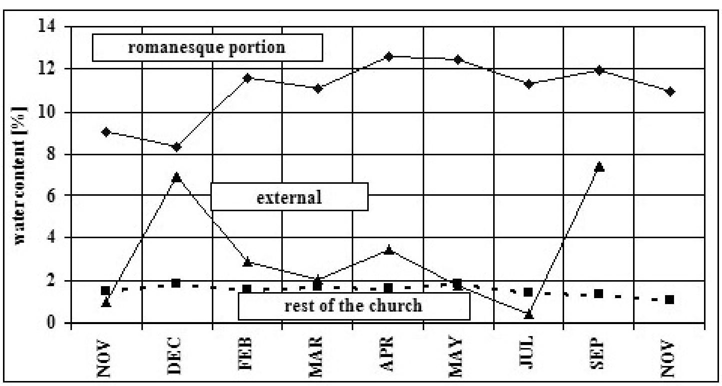

Fig. 6 Water content vs. time.

Romanesque part are in fact constituted by calcium sulphate bi-hydrate (gypsum - $CaSO_4 \cdot 2H_2O$) and magnesium (hexaedrite - $MgSO_4 \cdot 6H_2O$ - and epsonite - $MgSO_4 \cdot 7H_2O$).

The results of the diagnostic measurements and their analysis have therefore addressed the plan towards a first outdoor intervention of water-drainage of the strips of land that encircle the foundations of the building (upon the cliff).

The characterization of the external plaster

The sampling and the analyses have been lead in order to estimate the visible difference between the plasters of the two parts of the Church, the "ancient" Romanesque part and the eighteenth-century "recent" part.

Samples indicated as "ancient" are characterized by mortars with weakly magnesian lime binder with micritic texture and with irregular shaped cavities. The aggregate is made of silicate sand with angular quartz rounded metamorphic rock The aggregate show a variable granulometry from 0.2 to 1.0 mm (cliff fragments have greater dimensions that can arrive to 3-4 mm). The binder-aggregate ratio is approximately 3: 2.

The "recent" plasters have the same mineralogicalpetrographic nature of the "ancient" one, but show a minor percentage of the aggregate, with a binder-aggregate ratio 1:1 (Figure 7).

The samples of bedding mortar show the same composition and binder-aggregate ratio of the samples of the "recent" plasters group.

The analytical analyses therefore have indicated that the plasters settled on the external walls of the church (surely during the subsequent maintenance of the building) have been obtained with the same materials, probably coming from nearby quarries (the near Valley of Ossola has certainly been an historically testified source of supplying for the buildings of the lake Maggiore) mixed in different ways.

Subsequent stratigraphical-archaeological plasters sampling in the Romanesque apse have allowed to find a lime coming from furnaces from the neighbouring Caldè, recognizable for the light rose coloration. The presence of the furnace for the lime production from local stones is testified beginning from the XIII century from annals of the "Fabbrica del Duomo" (Milan Dome Factory) with

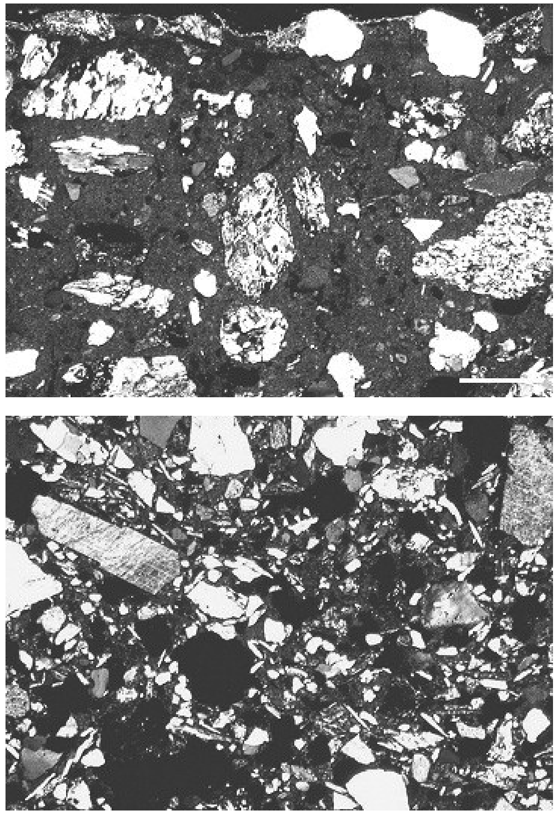

Fig. 7 Thin sections of a sample of "ancient" (above) and "recent" (below) plaster observed with the optical microscope in polarized light (bar = 100 μm).

a seasonal production alternated with fields working. The production became stable in the 1800's. The furnace has been then stopped after the Second World War; currently the rests of the furnace make a little port for the navigation on the Lake Maggiore.

The interventions

The interventions against water

Following the indications of the diagnostic investigations discussed in chapter 4.1, the first outside intervention on the building had the aim of removing from the church the meteoric water that falls in the proximity of it. In an excavation carried out around the building, a drainage

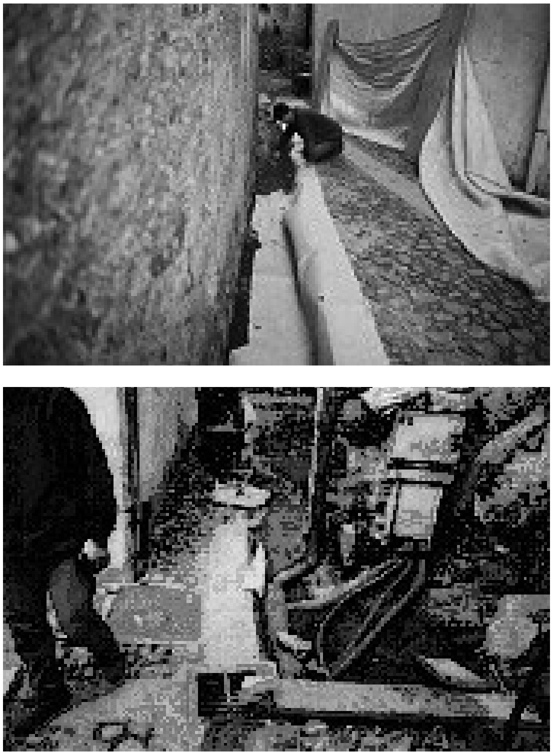

Fig. 8 The excavation and the drain strip around the church.

pipe has been put down and connected to the public water discharging; all the down-pipes of the meteoric waters collection plan have been connected to the same canalisation. The excavation has been then covered with a gravel layer of various diameters in order to obtain an effective water-drainage (Figure 8).

The interventions on external plaster

After the first phase of cleaning and consolidation (with injections of hydraulic lime and micronized binder) of the plasters, the phase of integration of gaps has been carried out following the diagnostic indications: using therefore the same compounds (seasoned air lime and different granulometry aggregates: fine sand, sand with diameter 1 mm, gravel from 5 to 9 mm, quartz crystals with size from 3 to 7 mm, micronized "cocciopesto" with diameter from 1 to 6 mm) for every single integration their ratio has been varied so as to integrate it with the existing plaster to match its granulometric and chromatic appearance (Figure 9). When necessary, a chromatic veiling with watercolour has been executed to better meet the original plaster colour.

Fig. 9 An example of integration of gaps of the external plaster (above) and of the bedding mortar (below).

The same procedure and materials have been adopted for the integration of the bedding mortar of the Romanesque portion of the church.

Conclusions

Because the described diagnostic analyses and the intervention on the external mortars are a part of a diffuse procedure of restoration, the authors wish to emphasize the integration of these procedures within a wider plan that has placed diagnostic investigations as an essential step to define every intervention of restoration of an historical building. After the described intervention, other works are previewed for the church indoors: the installation of a masonry dehumidification system (whose effectiveness will be measured in time), to bring back to their original context the frescoes contained in the warehouse of the Romanesque portion after their restoration, to remove soluble salts from walls. All these interventions will be controlled and supported by new measurements carried out to control the effectiveness of the masonry dehumidification, to help the restorer in the choice of the better salts removal technique and to check the new microclimatic indoor conditions in order to verify the possible necessity of a further plant intervention on the thermo-hygrometric air conditions.

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Materials Considerations Regarding Rain Penetration in Historic Fired Clay Brick Masonry

Considerações sobre materiais relativas à penetração de chuva em alvenarias históricas de tijolo de barro cozido

Caspar Groot
Delft University of Technology, Faculty of Civil Eng and Geosciences, 2628 CN-4, NL,
C.J.W.P.Groot@tudelft.nl

Jos Gunneweg
Delft University of Technology, Faculty of Civil Eng and Geosciences, 2628 CN-4, NL,
J.T.M.Gunneweg@tudelft.nl

Abstract

Moisture is a major source of damage in historic massive masonry. Therefore control of moisture movement in masonry is instrumental to the durability of masonry buildings. From research and practical experience it is known that a series of causes may play a role regarding permeability problems in masonry. This paper is focused on materials aspects regarding water penetration in historic fired clay masonry walls, constructed with moderate-to-high absorption bricks and lime mortars; the occurrence and influence of parameters such as brick porosity, interface leakage and mortar joint resistance are discussed. Subsequently, quantitative tests results are given on the effects of these parameters on the leakage of massive walls of different thicknesses. The results of the investigations lead to a number of recommendations to be used in case of repair of historic massive masonry. Finally, attention is paid to the influence of workmanship on the permeability behaviour of historic massive walls.

Keywords

Rain penetration; historic masonry; brick; mortar choice.

Resumo

A humidade é uma das principais causas de ocorrência de anomalias em alvenarias históricas. Deste modo, o controle dos movimentos da humidade em paredes maciças de alvenaria é um instrumento fundamental para a melhoria da durabilidade destas. A investigação e a experiência prática identificaram já várias das causas que podem influenciar os problemas de permeabilidade em alvenarias. O presente artigo é dedicado aos aspectos materiais relacionados com a penetração de água em paredes históricas em alvenaria de barro cozido, construídas com tijolos de absorção moderada a alta, assentes com argamassas de cal; discutem-se, nomeadamente, a ocorrência e influência de parâmetros como a porosidade dos tijolos, infiltrações nas interfaces e resistências das juntas de argamassa. Subsequentemente, são apresentados resultados quantitativos de testes que avaliam os efeitos destes parâmetros na ocorrência de infiltrações em paredes maciças de diferentes espessuras. Os resultados das investigações permitem apontar algumas recomendações às quais recorrer no caso de reparações em alvenarias históricas maciças. Finalmente, é ainda dedicada alguma atenção à influência da mão-de-obra na permeabilidade deste tipo de paredes.

Palavras-chave

Penetração da água da chuva; alvenaria histórica; tijolos; escolha de argamassas.

Introduction

Water leakage in historic massive masonry regularly occurs and is a major source of damage: in masonry, frost and salt damage; in timber, rot. Moreover, humidity may have negative effects on the living conditions in historic buildings.

From the literature [1-2] and practical experience, a number of causes for moisture problems like leaking can be deduced:

- inadequate material properties of the applied fired clay brick and masonry mortar; incompatibility between brick and mortar properties;
- cracks in masonry;
- inadequate design (e.g. lack of protection measures);
- poor ventilation;
- negative effects of a number of restoration interventions [like application of water repellents, application of dense plasters (prevention of drying) etc.];
- inadequate craftsmanship of the builders during construction and/or restoration.

The amount of possible causes of moisture problems in historic masonry underlines the complexity of this phenomenon [3]. Additionally, this complexity is enlarged by the often difficult to predict effects of an inadequate execution. However, it is quite clear that the influence of workmanship on the occurrence or effective cure of moisture problems is underestimated.

This paper is primarily focused on aspects dealing with an adequate choice of mortar and brick for water tight massive masonry.

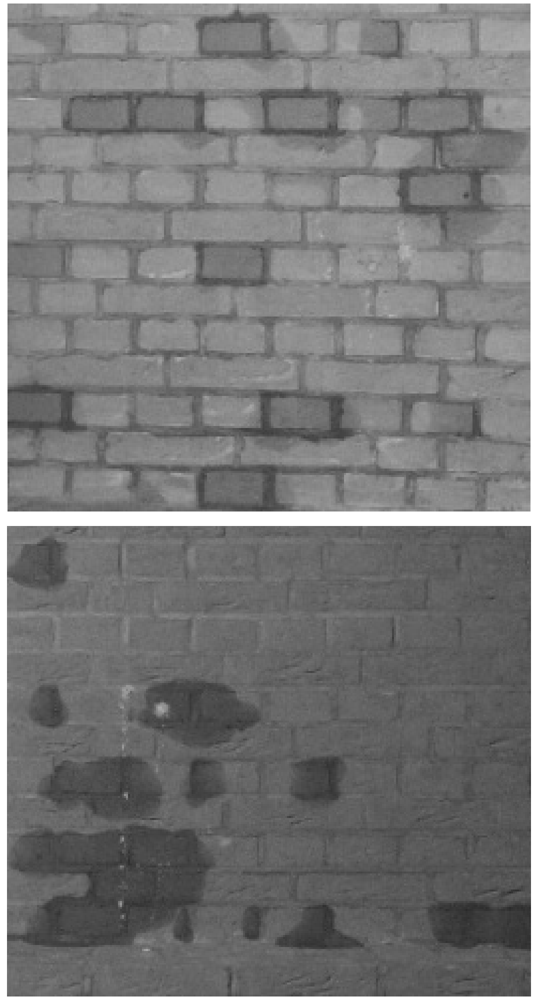


Fig. 1 Above: brick leakage in a 1 brick thick wall, caused by a high porosity of the applied brick (IRA brick 5.5 kg m^{-2} min^{-1}); Below: leakage in a 1 brick thick wall caused by mortar-brick interface leakage (IRA brick 1.5 kg m^{-2} min^{-1}) .

Rain penetration in walls of 1/2 and 1 brick length thick

Introduction

Focusing on materials behaviour in masonry walls of 1/2 and 1 brick length thick two main causes of leakage can be observed:

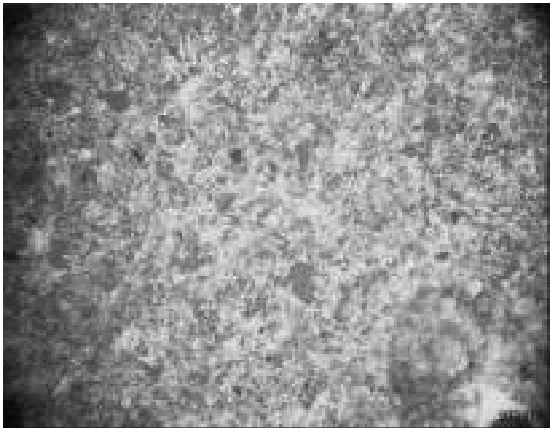
(i) leakage through the brick

(ii) leakage through the interface brick-mortar joint

the first cause being a pure materials characteristic and the latter mainly a hygric compatibility problem between brick and mortar [4]. These two types of leakage are shown in Fig. 1, being results of rain tests on two 1 brick length thick walls.

Brick Porosity

Essential to moisture transport in materials such as bricks and mortars is the pore system, as moisture absorption is a function of the capillary action of the



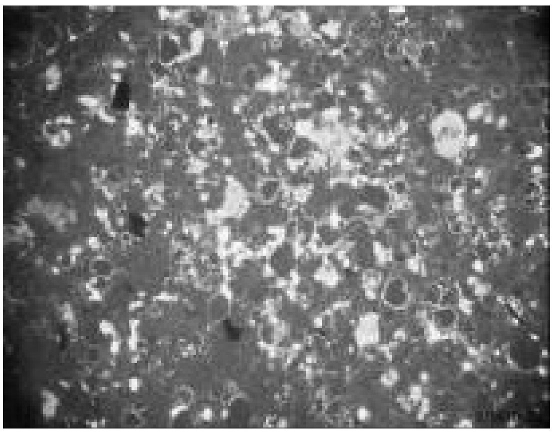


Fig. 2 Two fuorescence-microphotos of fired clay bricks: white indicating the porosity and dark is solid matter. Above: the strongly interconnected capillary network of a high absorption brick with a high absorption capacity. Below: a low absorption brick with isolated pores and a low absorption capacity (Photos Rockview, Amsterdam).

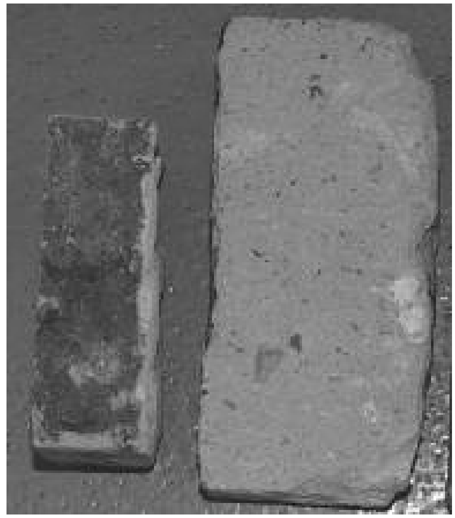


Fig. 3 Above, outside weathered side face of brick and inside (not weathered) original bed face of brick; below, outside weathered side of a wall.

pores and drying is determined by the evaporation rate. Although capillary absorption is a much quicker process than drying through evaporation, both depend on the pore size (distribution) of the materials.

Apart from clay type and the manufacturing process, the porosity of bricks is to a high degree determined by the firing process. In this process the final stage of "sintering" (melting of the clay) has a significant effect on the porosity: with a higher degree of melting the total porosity decreases (causing shrinkage) and coarser isolated pores are formed; the permeability of this type of bricks is low. With a lower degree of melting the total porosity is higher and pores form an interconnected network, enhancing the permeability of the brick (see Fig. 2).

Brick characterization

Basic aspects for moisture uptake in bricks are the "ease" of water absorption and the water storage capacity. The "ease" of water absorption maybe be characterized for instance by the Initial Rate of Absorption (IRA), that is, the water absorption per surface unit in 1 minute, or, when measuring the water uptake over longer periods of time, by the absorption coefficient.

The water storage capacity may be characterized by the free or vacuum water absorption.

Often bricks with a strongly interconnected capillary network show a high (initial) water absorption combined with a high water storage capacity. However, from tests on various brick types it was concluded that for a comparable water storage capacity the IRA may significantly vary. This means in case of equal water storage capacity a brick type with a higher IRA will be saturated in a shorter time than a brick type with a lower IRA.

Another aspect is that the IRA may vary with time as a function of the weathering conditions of the brick (see Fig. 3). In many buildings it can be observed that the IRA of the weathered side of the masonry significantly differs from that of the original material: the initial rate of absorption of the weathered outside is often less than half of the original material (the unchanged inside faces of the brick). The weathered side of brick is usually at the outer face of the masonry.

Consequently, with time the moisture uptake (rain absorption) of a wall will diminish; it is even conceivable (and observed in reality) that a leaking wall will stop leaking with time as a result of weathering.

Weathering of high absorption bricks of an exterior face of a wall results in a slower absorption of rain water; however, and this is an advantage, water storage capacity of the wall remains equally high.

Cleaning of weathered walls may result in an increase of the water absorption rate of the masonry, making it prone to leakage (especially sand blasting).

Brick – mortar interface

An important parameter for leakage is the quality of the interface layer between mortar and brick. With quality it is meant the porosity / density of the interface. The porosity of the interface is largely influenced by moisture transport from mortar to brick, directly upon brick laying. A dense interface may be formed if the brick exerts enough suction so that fine particles like cement or lime are transported to the interface and compaction at the interface occurs [5]. An open porous interface is created if the moisture of the mortar is not absorbed by the brick; this may easily occur using very low absorption bricks.

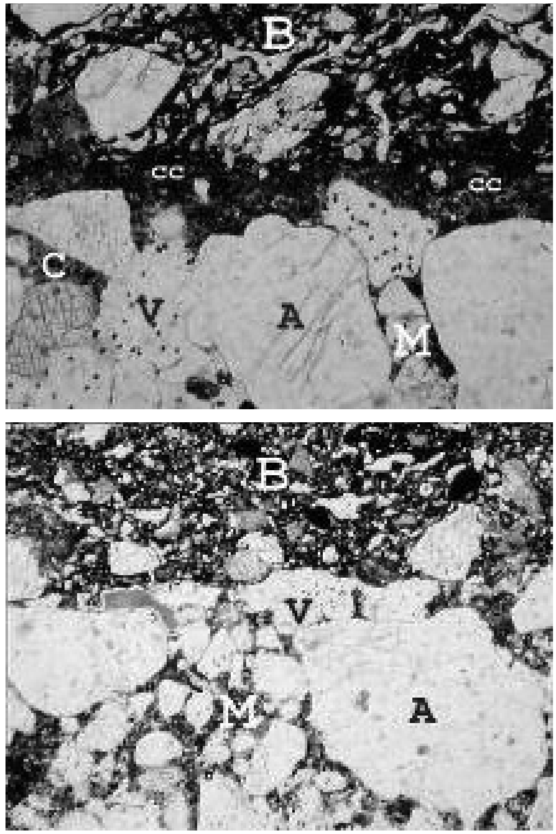


Fig. 4 Two examples of Brick-Mortar interfaces. Above: High void content at the interface; B: brick (IRA=0.29); M: mortar; A: aggregate; V: void; C: hydrated cement. Below; Layer of cement (cc) at the interface; B: brick (IRA=3.34); M: mortar; A: aggregate; V: void; cc: partially hydrated cement layer (Photos Joe Larbi, TNO-Built Environment and Geosciences, the Netherlands).

In figure 4, examples of an open and of a dense interface are shown [6]. Two types of bricks were used, with free water absorption values of 2.5 % and 19.5 % respectively, and an IRA of 0.29 kg m^{-2} min^{-1} and 3.34 kg m^{-2} min^{-1} respectively; and one type of mortar: a cement mortar (cement/sand ratio 1:4.5 (v/v) and water/cement ratio 1.03) was applied.

In order to obtain a good water tightness of the interface, the mortar composition should be compatible to the absorption properties of the brick. Fig. 4 (left) shows an incompatible combination: low IRA brick (IRA=0.29) combined with a mortar with a relatively high water/cement ratio (w/c ratio=1.03). This results in concentration of water at the interface, which cannot be absorbed by the brick (resulting in porosity after drying).

So, adaptation of the mortar composition to the brick properties is needed to assure a good interface. Basically, this means that the mortar composition, and in particular the moisture content of the mortar is adjusted to the absorption properties of the brick. Experience and trial-and-error are often the tools to find compatible brick-mortar combinations, as hygric characterization of the separate materials (mortar and brick) may not sufficiently predict the hygric behaviour of the mortar-brick combination.

In building practice some simple site tests can be applied to test the brick-mortar bond on site (the one-minute test and the 10 minutes test).

Rain penetration in walls > 1.5 brick length thick

Rain water that penetrates in walls with thicknesses > 1.5 brick length has to travel through a brick as well as crossing a mortar layer. So, water transport through a wall may be influenced by a moisture transport resistance exerted by (a) mortar layer(s).

Tests

A test program was set-up to study the effect of the mortar layer on the moisture transport in masonry test specimens. Starting point for the mortars was the lime mortar, as lime was generally used as binder in historic masonry with leakage problems. Test specimens were designed such that they can be considered as part of a wall, see figure 5. The test specimen consists of 3 courses of brick, 1½ bricks long and 1½ bricks thick (3 layers) put together using a bedding mortar.

During testing the uncovered face of the test specimen was immersed in a few centimetres of water. After a period of water absorption (24 h) the test specimen was removed and placed face up to let it dry (36 days).

Looking at the test specimens it is clear that during the absorption test for 2/3 of the cross section water has to travel through the brick *as well as* crossing a mortar layer.

For the tests two types of bricks were used: a red brick with a moderate IRA of 2.3 kg m^{-2} min^{-1} and a yellow brick with a high IRA of 3.5 kg m^{-2} min^{-1}. The bedding mortars applied were: two lime mortars: A and C; a weakly natural hydraulic lime mortar: B; and a strongly hydraulic mortar: X. The binder-to-sand proportions were always 2:1. No special allowance was made for the difference in brick IRA during specimen construction. Curing procedure: 1 week protected then in open air at 20 °C and 50-60 % RH.

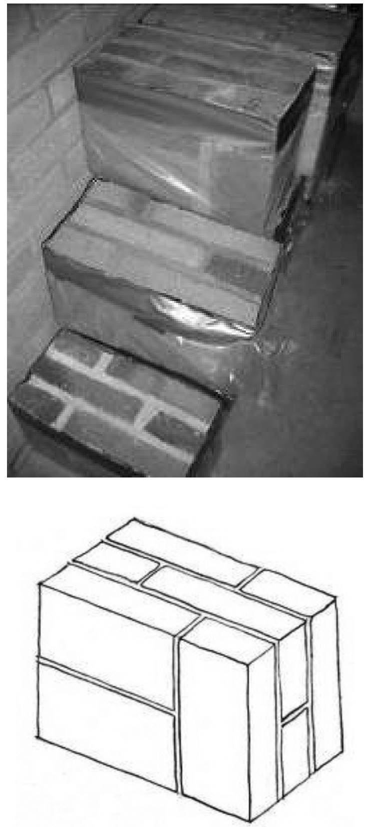
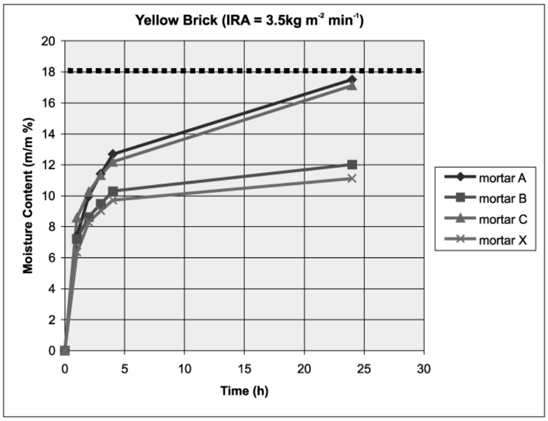


Fig. 5 View of test specimens. During the test, the uncovered face (the outer side of the wall) is exposed to absorption of water ("rain") or drying ; the other 5 sides are protected by plastic to allow drying from the uncovered face only (unidirectional drying as in a wall). Tests were performed by TNO-Built Environment and Geosciences, the Netherlands.

In order to indicate the effects of the different mortars on the moisture transport in the specimens some of the water absorption test results are presented in Figure 6.



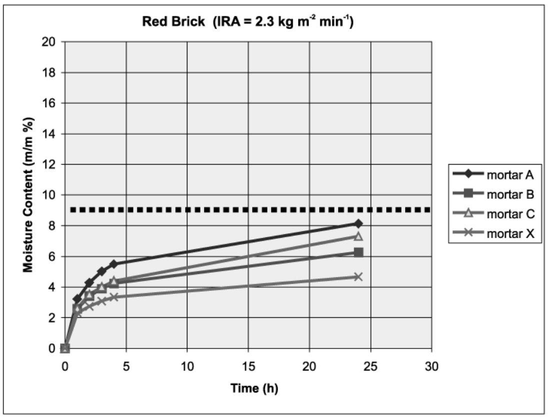


Fig. 6 Water absorption tests on various brick-mortar combinations tested; mortar A and C are lime mortars, mortar B and X are hydraulic mortars; the horizontal dotted lines in the figures indicate the values of the free water absorption by weight of the bricks (by capillary absorption from one face, over a 24 hour period) for the yellow bricks ~18 (m/m %); for the red bricks 9 (m/m %).

Barrier effect

The moisture absorption of the high-absorption brick specimens (left in Fig. 6), with the lime mortars (A) and (C), is significantly higher than that of the specimens with the weakly natural hydraulic lime mortar (B) and the strongly hydraulic masonry cement mortar (X).

After 24 hours of water absorption, test specimens A and C are almost saturated (up to their free water absorption capacity: horizontal dotted line). This is not the case for the test specimens containing the two hydraulic mortars (B and X); here, the water absorption is about 2/3 of the free water absorption capacity (11-12 m/m %). Apparently in the latter case the mortar acts as a barrier, restricting the rise of water to the top of the test specimen.

The distinction in water uptake of test specimens made with lime and hydraulic mortars, and low-absorption bricks is much less (right in Fig. 6). However, the order in the quantity of water uptake of the test specimens is the same for the four different mortars.

Rain leakage tests

Rain leakage of fired clay masonry walls was studied in walls with thicknesses of half, one and two brick length. The test walls consisted of high IRA bricks (5.5 kg m^{-2} min^{-1}) and moderate IRA bricks (1.5 kg m^{-2} min^{-1}). They were built with a weakly-hydraulic lime mortar.

After a rain test of 90 hours, performed according to NEN 2778, leakage of the walls was tested using a water supply of 2 L m^{-2} min^{-1} and an overpressure of 400 Pa (these are extreme conditions). Water leaking through the walls was collected and weighed.

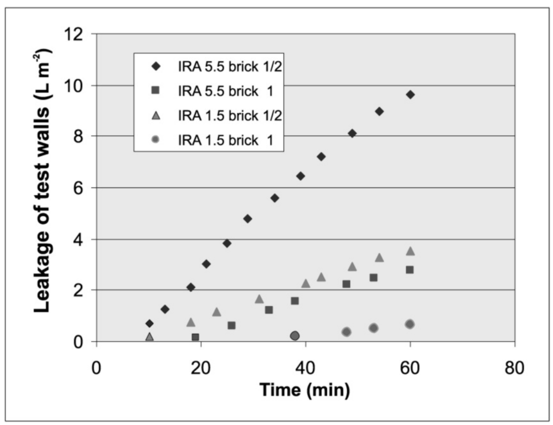


Fig. 7 Leakage of walls with thicknesses of half and one brick lengths. Two types of bricks have been used: a high absorption brick (IRA=5.5 kg m^{-2} min^{-1}) and a moderate absorption brick (IRA=1.5 kg m^{-2} min^{-1}); the walls were put together with a weakly hydraulic natural lime mortar.

As from Fig. 7 can be deduced, the leakage of the ½ brick thick wall with high absorption bricks (IRA=5.5) is significant: after 1 h is about 10 L m^{-2}. The water in this case mainly travels through the interconnected pores from one side of the brick to the other side (see as well Fig. 1, left). The leakage of the one brick thick wall (IRA=5.5) is more than 50 % lower, which mainly can be attributed to the barrier effect of the mortar joints in 50 % of the masonry (see Fig. 1, left).

The leakage of the moderate absorption (IRA=1.5) brick walls appears to be mainly caused by open brick-mortar interfaces and apparently is substantially less than for the high absorption walls. However, the leakage of the moderate absorption brick walls could still have been diminished if the bedding mortar composition would have been more compatible to the applied bricks.

With the two different brick types also walls with thicknesses of 2 brick lengths have been constructed. The same weakly hydraulic natural lime mortar was used. Applying the same "rain intensity" (120 L m^{-2} h^{-1}) and overpressure (400 Pa) no leakage occurred, neither in the high absorption masonry (brick IRA=5.5), nor in the moderate absorption masonry (brick IRA=1.5).

Apparently, the barrier effect of mortar layers is sufficient to prevent leakage in both mortar-brick combinations.

Materials choices

The investigations regarding rain penetration in massive fired clay masonry was triggered by leakage problems in historic windmills in the west of the Netherlands. These age-old mills were usually built with high absorption bricks and lime mortars. Unsatisfactory permeability resulting in leakage was not exceptional. The test results show that this is mainly caused by a poor barrier effect of lime mortars. The situation may be improved by weathering of the bricks.

In the case of windmills, the use of lime mortars is not only a disadvantage as the heavy dynamic solicitations of the sails on the masonry require a high deformation capacity of the mortar; and this is provided by a lime mortar.

In the case of repair, it is recommended to use bricks with similar hygric properties as the weathered old bricks (in practice 1.5< IRA< 3.0); for the mortars a weakly hydraulic mortar (with a hydraulicity index of 0.3-0.5, according to [7]) may be used in order to maintain as much as possible the deformation capacity of the masonry and to prevent compatibility problems with the old mortar.

If in historic massive masonry pozzolanic binders (for instance trass) were used the permeability problems are usually less severe, as a better barrier action is secured. Also these mortars show a satisfactory deformation capacity, important in masonry with few or lacking dilation joints. Choosing a repair mortar this deformation capacity should be maintained.

In cases where little deformation capacity is required, water tightness can relatively easy be achieved since the introduction of modern binders at the nineteenth century. Many churches, towers and factories, built from the 1880's on, show a very good water tightness. This was achieved by using moderate to low absorption bricks (IRA=1-2 kg m^{-2} min^{-1}) and hydraulic (shell lime, that is, lime obtained from burned sea shells, which may be non--hydraulic to feebly hydraulic) lime-cement mortars (for instance in a binder-rich composition of 10 shell lime, 3 cement and 10 sand).

Workmanship

Studying a number of problem cases in practice the influence of workmanship on permeability problems in massive masonry is unmistakable. This aspect is often underestimated.

A basic requirement for water tightness is that during execution no voids are left; to avoid this every brick should be fully surrounded by mortar. This is only possible if the brick laying is done brick-by-brick.

Skilful laying of bricks is a relatively slow process. As in practice economic considerations may prevail and quicker brick laying methods may be applied with negative effects on the water tightness (whereas voids are introduced as a result of the applied brick laying technique)

In practice it is observed that voids, apart from being water reservoirs in the wall, as well may promote leaching of soluble material (such as calcium hydroxide).

The filling up of voids by (mineral) grouts, using injection techniques, may significantly improve the water tightness of massive masonry.

Acknowledgments

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Material não publicado:

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Exemplo: Varley, A.J., 'Statistical image analysis methods for line detection', tese de doutoramento, University of Cambridge (1999).

Internet:

Autor, *Título do site ou do documento*, url (data de acesso).

Exemplo: IIC, *Author's guide: Studies in Conservation*, http://www.iiconservation.org/publications/scguide.php (acesso em 15-2-2004).

Submissão das colaborações

Os manuscritos devem ser enviados à Comissão Editorial através de *e-mail* ou através de disquete ou CD. No primeiro caso o envio deve ser feito para o endereço ajcruz@ipt.pt e no segundo para Francisca Figueira, Instituto Português de Conservação e Restauro, Rua das Janelas Verdes, 37, 1249-018 Lisboa. Em qualquer um dos casos, deve ser utilizado um ficheiro com um dos seguintes formatos: Microsoft Word (extensão .doc) ou *Rich Text Format* (.rtf). As figuras, se existentes, podem estar inseridas nesse documento ou ser fornecidas num formato gráfico (jpeg, gif, bmp, psd, wmf, emf ou cdr, entre outros).

Embora não seja obrigatório, é vivamente recomendado a utilização de um modelo de documento do Microsoft Word que pode ser obtido no *web site* da ARP (http://www.arp.org.pt).

Normas e instruções revistas em 18 de Dezembro de 2007.