Bituminous waterproofing mortar applied on masonry walls of 19th and 20th century buildings in Porto: an analytical study in the context of urban building rehabilitation

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Abstract

Bituminous mortars characterize the traditional buildings of the city of Porto and northern Portugal, especially those built between the end of the 19th century and the first decades of the 20th century. This article aims to determine the composition of this bituminous mortar through an analytical study by micro-FTIR and Py-GS/MS, and to assert experimentally its effectiveness as a waterproofing material though a series of tests including capillary tests, saturation content and water permeability. The present study is still a starting point for a more in-depth study on this subject, but the present investigation constitutes a further step in the characterisation of the constitution and function of bituminous mortar.

Keywords

Moisture, Bituminous Mortar, Oporto buildings, Water drainage, Chemical composition, Functional performance, Rehabilitation

1. Introduction

In buildings in the centre and north of Portugal dating from the 19th and early 20th century, a black coloured material (Figure 1) is commonly found under the final coating applied on stone masonry walls (Aguiar, 2002). This material had the function of improving the performance of walls against the action of water, under the liquid or vapour form, promoting horizontal and vertical water shedding (Mariz and Coroado 2004).

The use of waterproofing materials is a recurrent technique in civil construction to improve the performance of the building system against moisture (Lier-Klüge, 2018). The presence of water, whatever its physical state, is considered one of the main deterioration agents of building materials and elements (structural and decorative). On the other hand, it promotes the biological colonization of macro and microorganisms and the discomfort of its residents (Cóias, 2006). Thus, the presence of moisture, whether by capillary rise, condensation, infiltration or even by rupture of pipes, is one of the great problems of buildings (Freitas, 2002; Vasco, Torres and Guimarães, 2008).

In order to waterproof buildings between the 19th and 20th centuries in Portugal, it was common to use oil-impregnated limestone in the South of the country, and bituminous mortar in the granite-rich North region (Mariz, 2006). This black layer of a bituminous mortar was applied between the stone or brick wall and the plaster, which was then whitewashed, painted, or covered with tiles (Figure 2). For the preparation of this layer, a bituminous material of uncertain origin would be used, coming from tar or pitch (residual resin obtained in the distillation of vegetable resins or in the distillation of tar) (Brown, 2016)). Although there is some confusion in the designation of the binding material, its composition is known (Mariz, Coroado, 2004).

According to previous analyses (Mariz, Coroado 2004), the black mortar is composed of a binder and fillers. The fillers could include common earth or gravel, white sea or river sand, clay; and powder (probably the pulverized state of one of the aforementioned materials). Both earth and sand should be sieved to eliminate foreign materials and of a granulometry higher than desired.

At the time, and in subsequent documentation, the binder used to formulate the bituminous mortars was referred to by several names including "tar", "mineral tar", "pitch", "black pitch", "pitch", "coaltar pitch", "varnish" and "gas varnish". These designations were often used in a discretionary manner, without the rigour with which they are used today.

This study aims to determine the composition of this mortar and to experimentally verify its effectiveness as waterproofing material. For this purpose, 14 samples taken from 10 buildings in the city of Porto were used and subjected to micro-FTIR and Py-GS/MS analysis, and to water and thermal characterisation tests.

Figure 1 - Examples of buildings with bituminous mortar, visible after detachment of the final coating. Photos by Andrea Lier-Kluge.

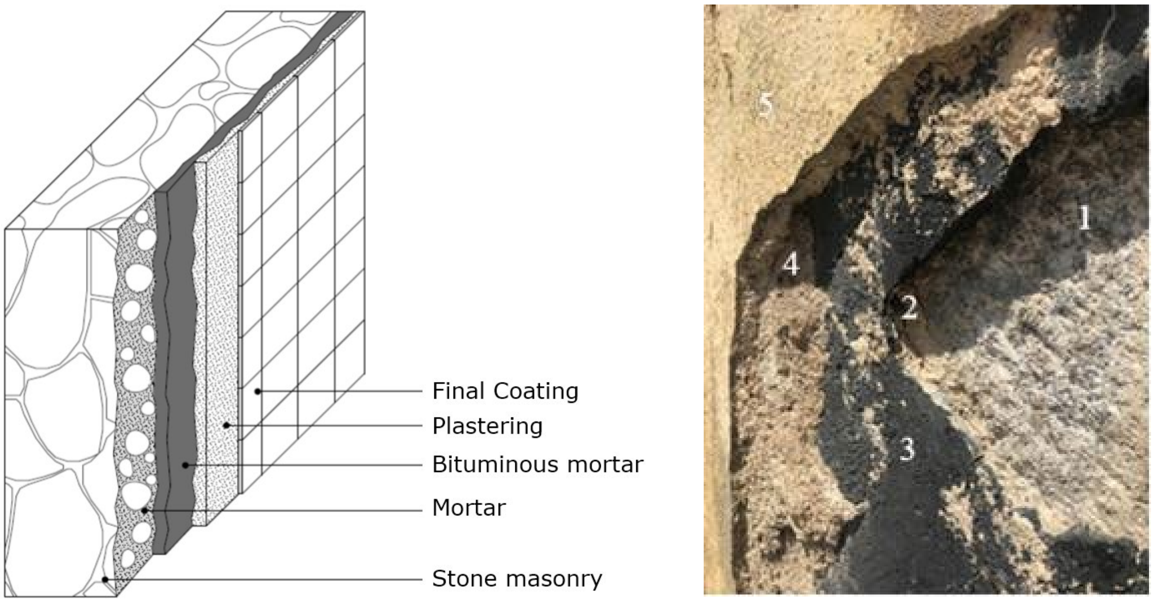

Figure 2 – Diagram (left) illustrating the successive main layers of a stone masonry wall cladding with bituminous mortar and example of the different layers from a typical wall (right): 1) stone masonry; 2) Grout for repointing joints; 3) Bituminous mortar; 4) Plastering; 5) Final coat (painting). Diagram and photo by Andrea Lier-Kluge.

3. Methodology

In order to study the bituminous mortar properties, several case studies were selected and samples were subjected to chemical and hydric analysis for characterization of the binding material. The chemical analysis was made by means of Fourier Transform Infrared spectroscopy (micro-FTIR) and pyrolysis coupled to gas chromatography and mass spectrometry (Py-GC/MS) techniques. The hydric characterisation was performed to verify the suitability of the materials regarding the liquid water transfer (permeability), saturation moisture content and capillary absorption coefficient, as well as thermal emissivity tests.

3.1. Case Studies

Forty buildings were initially selected as case studies but due to accessibility issues and owners' permission, the number was significantly reduced. Thus, a total of ten 19^{th} and 20^{th} century buildings were used as case studies from which 14 samples (Table 1) were extracted and tested in the laboratory.

Table 1 – Selected case studies and brief description of the collected samples.

Case Study #	Building Location	Sample #	Sample Wall
1	Rua do Passeio Alegre, 494	01	Side facade Exposed area*
2	Rua Senhora da Luz, 246/248	02	Wall share. Exposition due to the demolition of a building
3	Rua Francisco da Rocha Soares, 33	03	Frontal facade Exposed area
4	Rua das Taipas, 74	04	Frontal facade Exposed area
5	Largo do Moinho de Vento, 1	05	Inner Wall of the frontal facade Exposed area
		06	Inner wall: area of inspection window (intermediate level of the building)
		07	Lower floor (level below the ground floor)
6	Rua José Falcão, 144	08	Interior wall: inspection window open on the mezzanine
		12	Interior wall: opening of a new inspection window (next to the spiral staircase)
7	Rua João Grave, 74	09	Frontal facade Exposed area
8	Rua Padre Luís Cabral, 1016	10/11	Side facade Exposed area Inner Wall – exposed area
9	Rua da Torrinha, 64	13	Side facade Exposed area
10	Rua Fonte da Luz, 22	14	Frontal facade Exposed area

* Exposed area - the building was in poor condition with exposed bituminous mortar.

3.2. Chemical analysis

The micro-FTIR analyses were performed with a micro infrared spectrometer Bruker Hyperion 3000 equipped with a liquid nitrogen-cooled MCT detector and a 15x IR objective. The spectra were acquired in transmission mode in the spectral region 4000-600 cm^{-1}, with a spectral resolution of 4 cm^{-1} and an accumulation of 128 scans. For the analysis, a fragment smaller than 1 mm was taken from each bituminous mortar sample, which was then compressed in a diamond compression cell, so that the sample would be in the form of a thin film for analysis.

The whole set of samples was analysed by Py-GC/MS, with the exception of sample #4 because no organic material was detected according to the micro-FTIR results. The Py-GC/MS analyses were performed in an integrated system consisting of a CDS Pyroprobe 2000 filament pyrolyser, an Agilent gas chromatograph 6890N and an Agilent 5975N mass spectrometer. Pyrolysis of the samples was carried out at 610 °C for 10 s. Each sample was placed in a quartz holder and then introduced into the pyrolyser interface, maintained at a temperature of 250 °C. The chromatograph was operated in split injection mode (22:1), with a helium flow of 1.0 mL/min and the following furnace temperature program: initial temperature of 40 °C, maintained for 2 min; temperature increase at 10 °C/min up to 300 °C, followed by a isothermal period of 7 min. The mass spectrometer was operated under electron impact conditions, with an ionization energy of 70 eV, in continuous scanning mode in a mass/charge range between 45 m/z and 500 m/z. The interface between GC and MS, the MS source and the quadrupole type mass analyser were maintained at 280 °C, 230 °C and 150 °C, respectively. Data registration and instrumentation control were performed using the ChemStation program. For comparison purposes, a bituminous coal sample (sample #169) from the Ward's Natural Science Mineral Collection (Morgantown, West Virginia, USA) was also analysed (Figure 3). The identification of the pyrolysis products was performed by comparison with mass spectra libraries from NIST (National Institute of Standards and Technology) and Wiley (Silverstein *et al*., 1991).

Figure 3 - Bituminous coal. Sample 169 – Ward's Natural Science Mineral Collection, Morgantown, West Virginia, USA. © Chemistry and Biology laboratories of School of Arts/ Universidade Católica Portuguesa.

3.3. Water and Thermal characterization

To determine the capillary absorption coefficient, EN ISO 15148:2012 - *Hygrothermal performance of building materials and products - Determination of water absorption coefficient by partial immersion*, was used. This test consists in the partial immersion of the samples, obtaining, through weighing, the changes in mass as a function of the square root of time and, thus, the absorption coefficient (Aw). The test is exemplified in Figure 4. For this test, samples from three different buildings were used, containing three bituminous mortar samples per group.

To determine the saturation moisture content, no standards were followed since the samples did not comply with the parameters required by the standards. However, the following test procedure was followed: other samples collected from three different buildings, containing three bituminous mortar samples per group, were then used; the change in mass was measured after total immersion of the samples in water, and the saturation moisture contents were determined.

The mortar's water permeability was determined by examining its capacity to be permeated by water. For this evaluation, a test procedure not prescribed by any standard was followed but that allows to evaluate the samples' behaviour against the liquid water passage, 30 days after the beginning of the test. The test consisted of recording the progress of the water level contained in cups sealed around the samples and placed in an inverted position.

In addition to these water tests, a thermal characterisation test was also performed to measure the thermal emissivity of a sample of bituminous mortar from one of the selected buildings. The emissivity (ε) has a fundamental role in the phenomena of heat transfer by radiation of a material. radiation heat of a material and translates as the ability to emit energy by radiation through its surface. The test was performed only on one of the samples of bituminous mortar with lime-based mortar and an asphalt sheet in order to compare the results obtained. The thermal emissivity test was performed using ASTM C1371 - *Standard Test Method for Determination of Emittance of Materials Near Room Temperature*, using a portable emissometer model AE1 (*Devices & Services Company*), designed to measure emissivity on flat surfaces with good conductivity. The accuracy of the standards provided is of the order of ± 0.02.

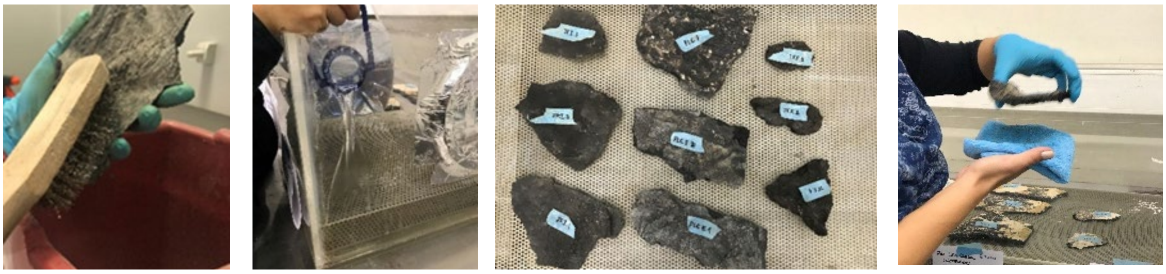

Figure 4 – Methodology steps for the determination of the coefficient of water capillarity absorption. From left to right: sample brushing to remove attached dirt; addition of water to the vat; partial immersion of the samples; and removal of excess water with the aid of an absorbent cloth, before weighing.

4. Results and Discussion

4.1. Chemical analysis

Two analytical techniques - micro FTIR and Py-GC-MS - were used for the chemical analysis for the characterisation of the organic material used as binder in the bituminous mortar samples.

With the exception of sample 04, the IR spectra obtained from all the samples reveal the presence of silicates and of an organic material consisting mainly of a complex mixture of aromatic hydrocarbons (Beychok, 1974; Coates, 2000). As an example of the 14 samples, Figure 5 presents the FTIR spectrum of sample #1. The spectrum of most samples presents a series of bands that are due the presence of aluminosilicates, namely clay minerals of the kaolinite group ($Al_2Si_2O_5(OH)_4$). Besides kaolinite, other silicates are present but whose identification is not possible due to the overlapping of their bands with those of other materials in the samples or by the lack of characteristic bands that allow their identification by FTIR (Bellamy, 1975; Derrick *et al*., 1999; Casadio, Toniolo, 2001). It is also possible that the samples contain quartz (SiO_2), a silicate that presents an intense and wide band around 1080 cm^{-1}, weak bands at about 795, 780 cm^{-1} and at about 795, 780 and 695 cm^{-1} (Marel, Beutelspacher,1976). However, it is not possible to verify its presence in the samples due to the overlapping of its absorption bands with those of the with those of the kaolinite group minerals.

The inorganic fillers detected in the samples were introduced during their preparation. Detection of minerals from the kaolinite group was within the expected range. Kaolinite is a clay and is found in significant quantities in the materials mentioned as fillers for bituminous mortar. Sand, soil and gravel are materials made up of quartz and silicates, among other components such as iron oxides.

All the acquired infrared spectra were very similar, indicating that it was either a bitumen or asphalt of natural origin, but it did not differentiate the reference of coal or bituminous coal in the studied samples. For this reason, analyses were performed by Py-GC/MS, where the acquired spectra allowed the identification of some of the compounds.

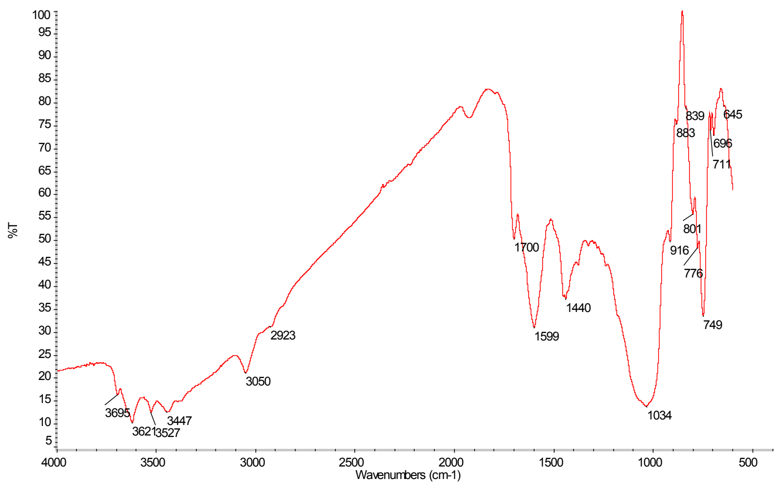

Figure 5 - IR spectrum of sample #1. Spectrum by José Carlos Frade.

As an example of the 14 analysis, Figure 6 presents the pyrogram comparison between sample #1 and reference sample #169. The main compounds detected by Py-GC/MS in the bituminous mortar samples are shown in Table 2. The pyrolysis products that appear in larger quantities in the bituminous coal reference are only compounds with 5 or 6 aromatic rings, while in the studied mortar samples relevant quantities of aromatic compounds with two, three, four and five rings are found. Some of these compounds with fewer aromatic rings, such as naphthalene, anthracene or phenanthrene, were also detected in the bituminous coal reference, although in very low or trace amounts.

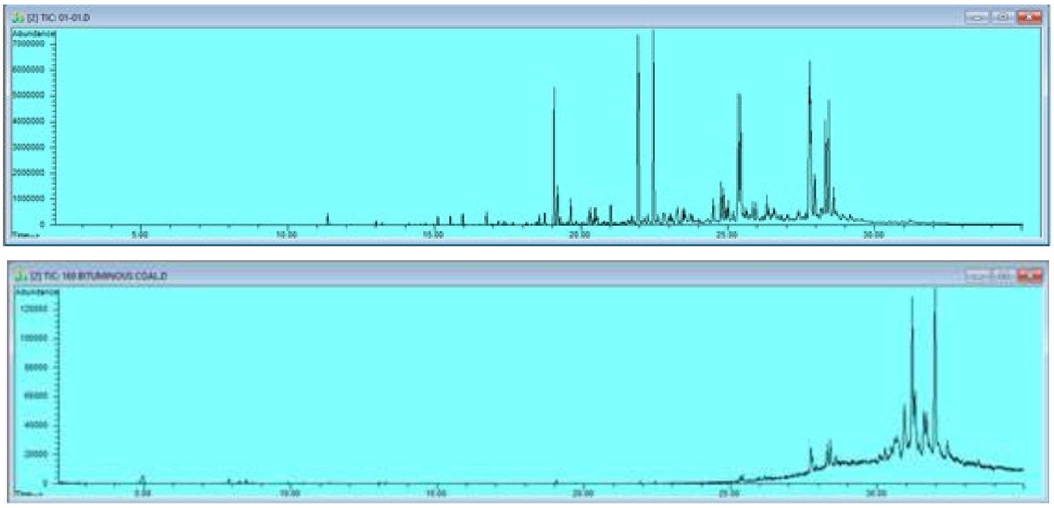

Figure 6 – Pyrogram comparison of sample #1 (above) and reference (below) from a bituminous coal (sample #169). Pyrograms by José Carlos Frade.

Table 2 – Compounds identified in the bituminous mortar samples, analyzed by Py-GC/MS.

Tr (min)	**Compound**	**Molecular Mass**
11.38	Naphthlene	128
11.48	Benzothiophene	134
13.01	1-methyl-naphthalene	142
13.23	2-methyl-naphthalene	142
15.12	Biphenylene	152
15.54	Acenaphthene	154
15.97	Dibenzofuran	168
16.79	Fluorene	166
18.76	Dibenzothiophene	184
19.11	Phenanthrene or Anthracene	178
19.21	Phenanthrene or Anthracene	178
21.96	Fluoranthene or Pyrene	202
22.49	Fluoranthene or Pyrene	202
25.36	Benzanthracene or Triphenylene or Chrysene	228
25.44	Benzanthracene or Triphenylene or Chrysene	228
25.60	Benzanthracene or Triphenylene or Chrysene	228

Tr (min)	Compound	Molecular Mass
27.78	Perylene or Benzopyrene or Benzofluoranthene or other isomer of these compounds	252
27.86	Isomer of compound with RT*= 27.78 min	252
27.96	Isomer of compound with RT= 27.78 min	252
28.32	Isomer of compound with RT= 27.78 min	252
27.44	Isomer of compound with RT = 27.78 min	252
28.88	Derived from Hopan: 22,29,30-Trisnor-(17.alpha.H)-hopane	370
29.97	Derived from Hopan: 28-Nor-17.beta.(H)-hopane (or isomer of this compound)	398
30.47	Isomer of 28-Nor-17.beta.(H)-hopane	398
30.79	Hopane (also referred to as A'-neogammaceran) or Gammacerane	412
31.25	Hopane (also referred to as A'-neogammaceran) or Gammacerane	412

* RT- retention time

The pyrograms of the various bituminous mortar samples show some differences between them, particularly in the case of a few samples (#5 to #9, and #12 to #14), where a lower quantity of the compounds that resulted of the chromatographic column between 23 min and 28 min of analysis (polyaromatic compounds with 4 or 5 rings) was detected, when compared with the other samples. This fact may be related to the state of conservation of these mortar samples: the exposure to the various environmental factors to which they were subjected, over the years, may have contributed to the degradation of the compounds belonging to this higher molecular weight fraction, which were converted into lower molecular weight compounds.

In the case study samples, besides the polyaromatics already mentioned (compounds at retention times lower than 27.44 min), some pentacyclic triterpenes, such as hopane, gammacerane and hopane derivatives, were also detected. These compounds appear in the pyrograms at retention times greater than 28 min and are characteristic of bitumens of natural origin, together with benzothiophene and dibenzothiophene, which are compounds containing sulfur (S) in their molecular structure (Languri, 2004).

We also highlight the fact that in some of the samples (#1 to #3, #10 to #13) the pentacyclic triterpenes detected were found in trace amounts. This smaller quantity may be due to the degradation processes that may have occurred. However, accelerated ageing tests with natural bitumen would be necessary to simulate the conditions to which these samples were submitted over time in order to understand if there is a significant variation in the quantity of these compounds due to the influence of temperature, humidity, atmospheric pollutants, or if there is another cause that explains those differences.

Considering the case study #6, a sample from an inspection window intentionally opened for the collection (#12), and hence deemed a healthy area, is the one that presents the smallest amount of hopanes and gamma-kerane, it is probable that there is another explanation for the different amounts of these compounds among the several samples analysed. Thus, it is hypothesized that there is some influence on the chemical composition of the bitumen introduced by the procedures taken in the preparation and application of the bituminous mortar in the building.

4.2. Water and thermal characterization tests

After characterisation of the constituents of the mortars under study, tests were performed to determine the capillary absorption coefficient, the saturation moisture content and to evaluate the water permeability of the mortar: the capillary absorption coefficient was determined based on the measurement of the variation of mass over time; for the moisture content, the variation in mass was measured after the total immersion; permeability of the mortars was based on their hydrophobic capacity.

The capillary absorption coefficient (Aw) test concluded that the bituminous mortars present an Aw values of about 3E-04 kg/(m^2.$^{s0.5}$), while the saturation moisture contents ranged between 0.48% and 3.54%, which are values considered as very low (Table 3).

Table 3 – Weight variation during the capillary absorption coefficient test.

Capillarity											
Date	23 Oct	24 Oct	25 Oct	25 Oct	25 Oct	25 Oct	25 Oct	25 Oct	25 Oct	26 Oct	27 Oct
Schedule	11h	11h	9h40	5 min	20 min	1h	2h	4h	8h	24h	48h
	m0 (g)	m1 (g)	mi(g)	300s	1200s	3600s	7200s	1400s	28800s	86400s	172800s
Group I											
JF.I. 1	204,961	204,650	204,940	205,533	205,505	205,391	205,435	205,436	205,475	205,570	205,565
JF.I. 2	180,356	180,345	180,337	180,651	180,697	180,808	180,664	180,668	180,765	180,766	180,616
JF.I. 3	130,251	130,242	130,239	130,563	130,530	130,603	130,571	130,571	130,602	130,581	130,511
Group II											
P CL. E. 1	283,068	283,056	283,042	283,480	283,828	283,722	283,813	283,764	283,900	284,129	284,184
P CL. E. 2	320,940	320,910	320,904	321,930	322,615	321,873	321,678	321,608	321,765	321,796	321,830
P CL. E. 3	577,658	577,631	577,620	578,979	875,741	578,128	578,224	578,279	578,200	578,796	578,191
Group III											
JF.F. 1	140,998	140,785	140,614	140,670	140,861	140,876	140,937	141,047	141,222	141,615	141,969
JF.F. 2	55,559	55,312	55,246	55,368	55,531	55,562	55,664	55,810	55,920	56,288	56,617
JF.F. 3	31,644	31,604	31,502	31,556	31,739	31,773	31,805	31,874	31,986	32,090	32,305

The various tests have shown that bituminous mortars hardly absorb any water by capillarity or saturation. These results, in themselves, suggest that bituminous mortar is not perfect but it is well suited for its function as waterproofing layer, which was later further confirmed by the water permeability tests.

During the water permeability test it was seen that there was no water absorption in any of the three samples, concluding that this bituminous mortar has a high waterproofing capacity on the surface. The three tests described revealed that the bituminous mortar has hydrophobic properties, hardly absorbs water by capillarity and has very low saturation moisture levels, indicating that this coating material fulfils its waterproofing function.

However, it should be noted that some water may eventually be carried to the interior of the wall through the bituminous mortar and be retained in the interface stone-mortar, causing the degradation of this interface and the disintegration of the stone. This phenomenon is believed to be the major promoter of detachment of bituminous mortar, often observed in old buildings.

The thermal emissivity test showed that the bituminous mortar has a value of 0.93. When compared with asphalt blanket (0.87), it can be seen that both thermal emissivity values are considered high, indicating that the material has a low thermal performance and is therefore not very refractory. This test showed that the bituminous mortar has a high value of thermal emissivity, and hence a low thermal performance, being a poorly refractory material. Although mortars can be considered good "hydric insulators", due to their poor thermal insulation properties, their capacity to conduct heat may have an influence on the problem of condensation inside buildings.

Conclusions

Most materials used in construction present a certain degree of porosity that allows water, whatever its origin or state, to migrate through the pores and to moisten them. In the case of building walls made of stone masonry and their materials, this phenomenon, associated with lack of maintenance, causes anomalies in the elements, contributing to the detachment of coatings such as plaster, tile or paint. The use of a waterproofing material applied on the walls, such as the bituminous mortar in study here, enables the counteracting of this effect. However, it is possible to verify that, in some cases, occasional detachments occur, resulting in degradation of this layer or of the interface, namely of the material supporting it or of the materials that are in contact with it.

The constitution of this material is not very precise, considering the few historical records that exist. There is some confusion regarding the nomenclature used at the time to designate the material used as binder (binder) in the preparation of bituminous mortar. Also, no distinction was made between the type of sand, from the sea or river, used as filler in the preparation of the mortar.

The chemical characterisation of the mortar samples studied allowed us to verify that the material used as a binder is a bitumen or asphalt of natural origin. The micro-FTIR analyses showed that the binder had similar characteristics, pointing to the possibility that it is a material composed of aromatic compounds. The Py-GC/MS analysis detected compounds, namely gammacerane, specific to natural bitumen.

On the other hand, the water tests carried out demonstrated that the material fulfils the function of waterproofing material, preventing water migration, independently of its state, liquid or gaseous. This result allowed us to estimate that the factor which most influences the detachment of this mortar is the retention of the water transported to the interior of the wall, either by capillarity or infiltration, at the interface with this material. The permanence of this water causes the disintegration of the stone material and the rendering mortar and, consequently, the loss of cohesion, causing the detachment of the bituminous mortar and/or the layers adhering to it. On the other hand, it was also observed that, when subjected to temperature, bituminous mortar becomes malleable, making it essential to have a good external coating or protection to avoid the direct action of the heat that can modify its properties and function.

Finally, the thermal characterisation test shows that the material is a poor thermal insulator and that this characteristic may enhance the occurrence of condensation phenomena inside the buildings, although in a dispersed way, since the entire area of the masonry wall, and not only circumscribed zones, are in the same insulation conditions. It is most likely that all these factors contribute to the detachment of the bituminous mortar, often observed in old buildings.

The decision to keep the mortar, or not, should be made by analysing each case individually. If the mortar is in a good state of conservation and presents good adherence to the support, it should be maintained since it fulfils the waterproofing function. Besides, it is a material and a historical technique with heritage value that is disappearing with the rehabilitation of old buildings that do not contemplate the maintenance of the original coatings, thus losing materials, construction techniques and decorative elements characteristic of its period.

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Technical Standards

ASTM C1371 - *Standard Test Method for Determination of Emittance of Materials Near Room Temperature. Using Portable Emissometers*

Desempenho térmico de edificações Parte 2: Métodos de cálculo da transmitância térmica, da capacidade térmica, do atraso térmico e do fator solar de elementos e componentes de edificações. ABNT novembro de 2004 Projeto 02:135.07-001/2. Universidade Federal de Santa Catarina Ctc - Departamento de Engenharia Civil - Laboratório de Eficiência Energética em Edificações.

EN ISO 12570:2000 - *Hygrohermal performance of building materials and products – Determination of moisture content by drying at elevated temperature*.

EN ISO 15148:2012 - *Hygrothermal performance of building materials and products – Determination of water absorption coefficient by partial immersion.*

Authors' Curriculum Vitae

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