Paper 5: Historic Mortars from the Coudenberg Archaeological Site: Characterization and Source of Raw Materials

LAURENT FONTAINE, ROALD HAYEN, SEBASTIAAN GODTS, and HILDE DE CLERCQ

Royal Institute for Cultural Heritage (KIK-IRPA), Belgium

The characterization of historic mortars by means of a concise methodology of material analyses can provide crucial information with regard to the origin of the raw materials and the ancient mortar technology. For the analysis of the historic mortars of the Coudenberg archaeological site, the methodology consists of optical and scanning electron microscopy, simultaneous thermal analysis, and an acidic treatment. These analyses were carried out on a total of seventeen samples lifted from various areas in the site, in function of the building chronology resulting from a historic built investigation. The type of binder and aggregate as well as their volumetric ratio were determined.

Both binder and aggregate present distinctive features, which made it possible to identify the probable geological provenance of the raw materials. The observations are verified with available historical records regarding the construction of the main banqueting hall — the Aula Magna — and the use of raw materials such as limestone and sand for the production of lime and construction purposes.

KEYWORDS mortar characterization, raw material, limestone, lime lump, microfossil, Brusselian stone, Brusselian sands

Introduction

This paper presents the results of a study of historic mortars from the Coudenberg archaeological site, which consists essentially of the remains of the former palace of Brussels (Belgium). More information about the historical background of the site can be found in the introductory article by Cnockaert, et al. (see Paper 1, this issue).

Materials

A total of seventeen mortar samples was lifted from four building phases of the archaeological site (Figure 1), more precisely: (1) the cellars of the former living quarters of the palace's main building, that are most likely the oldest part of the site dating from the twelfth to the fifteenth century, (2) the rooms underlying the Aula Magna, the main banqueting hall built between 1452 and 1460, (3) the warehouse space under the chapel, dating mainly from the sixteenth century and subjected to various transitions in the eighteenth century, and (4) the remains belonging to some ancient dwellings pre-dating the construction of the Aula Magna, dated back to the fourteenth century, or perhaps earlier. An overview of all analysed mortars is given in Table 1, while Figure 2 shows a detailed view of a debris pile found in the Aula Magna, left over from the fire of 1731.



FIGURE 1 Plan overlaying the Coudenberg archaeological site and museum on the Royal Square (1. Main building; 2. Rue Isabelle (Isabella Street); 3. Aula Magna; 4. Chapel; and 5. Hôtel d'Hoogstraeten (Hoogstraeten House)). The location of the 17 mortar samples is identified on the plan. The samples M12 to M15 were sampled on the outer corner of the Aula Magna, almost at the level of the Royal Square (a level several metres above the underground archaeological site), where some remains of ancient dwellings pre-dating the construction of the Aula Magna have been identified.

TABLE 1

Location (Figure 1)	Description Date		Code
Living quarters (cellars)	Bedding mortar, vault	15th century (transformation)	M1
	Bedding mortar, exterior wall	12th century (original construction)	M2
	Bedding mortar, exterior wall and vault	14th century (transformation)	M3
Aula Magna (cellars)	Bedding mortar, interior wall	1452-60	M4
	Bedding mortar, collapsed vault	1452-60	M5
	Bedding mortar, interior wall	1452-60	M6
	Plaster, interior wall	Before 1731	M7
	Infill mortar, exterior wall	1452-60	M16
	Infill mortar, exterior wall	1452-60	M17
Chapel (cellars)	Infill mortar, column	16th century	M8
	Bedding mortar, interior wall	16th century	M9
	Plaster, 16th century interior wall	Unknown	M10
	Bedding mortar, interior wall	18th century (transformation)	M11
Ancient dwellings	Bedding mortar, exterior wall	Before 14th century	M12
	Bedding mortar, cellar interior wall	14th century	M13
	Bedding mortar, cellar vault	14th century	M14
	Render, exterior façade	14th century	M15

LOCATION, DESCRIPTION, DATE, AND CODE OF THE 17 MORTAR SAMPLES. THE CONSTRUCTION DATES WERE OBTAINED FROM A HISTORIC BUILT INVESTIGATION LED BY ARCHAEOLOGISTS OF THE ROYAL ARCHAEOLOGICAL SOCIETY OF BRUSSELS

Methodology

Thin sections for optical microscopy (Zeiss, Axioplan equipped with a Leica DC 300 digital camera) as well as polished sections for scanning electron microscopy were prepared from each mortar sample. The visual analysis of a thin section allows the determination of the mineralogical composition of the mortar while scanning electron microscopy coupled with an energy dispersive X-ray spectrometer (SEM-EDX, Jeol JSM-6300) reveals the elemental composition of the constituents.

Since the silicon content can be regarded as a proxy for the hydraulicity of the binder, a minimum of ten EDX analyses was performed, preferentially in lime lumps to avoid any contamination from the sand constituents, to determine the average atomic ratio of silicon to calcium. According to the standard EN 459-1:2010, lime might contain up to 10 wt% of elements other than calcium.

Further, simultaneous thermal analyses (STA) consisting of thermogravimetric analyses (TGA) coupled with differential scanning calorimetry (DSC) were carried out (Netzsch STA 449 F3 Jupiter®). For that, approximately 30 to 45 mg of a manually crushed mortar sample was heated to 1200° C at a rate of 20° C/min. The weight loss between 200 and 600° C can be attributed to the loss of water chemically bound to hydraulic compounds and is therefore indicative for the hydraulicity of the binder fraction (Bakolas, et al., 1998: 151–60). At higher temperatures, in general up to



FIGURE 2 Detail of a pile of debris, found in the Aula Magna, left over from the fire of 1731. Consecutive layers of debris can be recognized. The black laver is rich in charcoal fragments from the burnt wood coming from the main banqueting hall at the ground level. Right underneath. the structure of the collapsed vaults from the rooms underlying the main banqueting hall can still be recognized. The bedding mortar of the vaults was sampled (mortar M₅).

 800° C, carbon dioxide is released from calcium carbonate resulting in a significant weight loss. In the absence of limestone fragments, the weight loss due to decarbonation of calcium carbonate can be attributed to the air-hardening part of the binder. However, the actual onset temperature (T_{onset}) of decarbonation was in this investigation generally between 635 and 675° C, well above the threshold value of 600° C defined by Bakolas (ibid.). Therefore, the actual onset temperature related to the release of carbon dioxide is considered as indicative for the fraction of hydraulic compounds relative to the air-hardening part of the binder. A hydraulic index (HI_{STA}) is calculated as the percentage of weight loss between 200 and T_{onset} to the total weight loss in the temperature range of 200–800 °C.

The analytical part is completed by a chemical treatment of the mortar samples. For that, 2 to 2.5 g of ground mortar sample is added to 60 ml of a 4 M solution of hydrochloric acid (HCl) to decompose the lime binder, followed by filtration. The filtrate is then heated to 1200° C in a crucible. The retained solid is attributed to quartz sand. The initial binder/aggregate volume ratio is estimated assuming a density of 1.80 kg/dm³ and of 1.10 kg/dm³ for respectively quartz sand and lime binder.

Finally, the size of the aggregate grains or granulometry of the aggregate is approached by setting up a curve consisting of the diameter of about 300 sand grains measured in thin sections.

Results and discussion

First observations

Based on the investigation of the thin sections, the composition of mortar sample M11, lifted from the eighteenth-century integration of the warehouse spaces of the chapel into the cellars of the neoclassical surrounding buildings, clearly deviates from that of the others which are very similar, although they were used during a large period, from the twelfth up to the seventeenth century.

Table 2 summarizes the results of the analytical procedure carried out on mortar samples.

Identification of the binders

Mortars from the twelfth to the seventeenth century

A distinctive feature noticed in all mortars dating from the twelfth to the seventeenth century is the presence of lime lumps, which are characterized by the inclusion of quartz grains as well as varying amounts of black coloured ones (Figure 3a). These grains have a size inferior to 100 μ m, hence significantly finer than the ones used as aggregate. Nevertheless, lime lumps typical for lime that was not completely burnt were noticed containing quartz grains of a comparable granulometry as the aggregate as well as reddish grains (Figure 3b). Both reddish and black coloured grains are attributed to a specific mineral: glauconite. Glauconite is an iron-rich phyllosilicate that in unweathered state exhibits a green tint. During the firing process, an oxidation takes place by which the grains turn red to black with increasing heating temperature. Altered glauconite grains, having a black tone as well, are also dispersed in the binder, rendering their distinction with coal fragments used during the firing process of the limestone difficult.

Besides quartz grains and altered glauconites, some remains of small foraminiferal tests (microfossils) are noticed in lumps typical for an incompletely burnt lime (Figure 3b), as well as patches of microcrystalline calcite. The last are probable remains of the microsparitic cement of the original sandy limestone. Finally, completely burnt microfossils or bioclasts, especially small foraminiferal tests and urchin spines, are identified in the binder (Figures 3c and 3d), as well as fragments of microsparitic sandy limestone containing green glauconite grains and ostracod shells (Figure 3e).

From these microscopic findings, it seems that a microsparitic sandy limestone containing glauconite grains and microfossils/bioclasts, like small foraminiferal tests, urchin spines and ostracod shells, was used for the production of the lime binder, more specific a sandy limestone from Brussels or its immediate surroundings.

In the region of Brussels, two geological layers, mainly made up of sand, contain sandy limestone: the Brussels Formation and the Lede Formation, both of Eocene age (Camerman, 1955: 5–26, 51–64). These formations are in a stratigraphic continuity, with the Lede Formation on top of the Brussels Formation, and were consequently

TABLE 2

Place	Code	Binder (atomic% Si, HISTA (%))	Classification aggregate (maximum grain size, mm)	Binder/aggregate (volume ratio)
Living quarters	M1	slightly hydraulic lime (20.3, 33.4)	fine sand (0.35)	2:3
	M2	slightly hydraulic lime (9.1, 24.0)	fine sand (0.36)	4:3
	M3	slightly hydraulic lime (8.7, 28.7)	middle coarse sand (0.54)	1:1
Aula Magna	M4	slightly hydraulic lime (8.7, 14.3)	fine sand (0.33)	2:3
	M5	slightly hydraulic lime (10.8, 35.8)	fine sand (0.34)	1:1
	M6	slightly hydraulic lime (9.4, 19.7)	fine sand (0.34)	3:4
	M7	slightly hydraulic lime (11.1, 26.4)	fine sand (0.36)	1:1
	M16	slightly hydraulic lime (33.5, 57.5)	fine sand (0.36)	2:3
	M17	slightly hydraulic lime (32.9, 68.8)	fine sand (0.38)	1:2
Chapel	M8	slightly hydraulic lime (8.0, 24.0)	fine sand (0.47)	5:4
	M9	slightly hydraulic lime (7.2, 22.1)	fine sand (0.41)	4:3
	M10	slightly hydraulic lime (8.2, 22.7)	fine sand (0.33)	1:1
	M11	air lime (2.5, 14.6)	fine to middle coarse sand (0.38)	3:1
Ancient dwellings	M12	slightly hydraulic lime (8.0, 29.3)	fine sand (0.34)	1:1
	M13	slightly hydraulic lime (4.9, 15.8)	fine sand (0.31)	1:1
	M14	slightly hydraulic lime (11.6, 25.4)	fine sand (0.27)	1:2
	M15	slightly hydraulic lime (9.8, 33.5)	fine sand (0.26)	4:5

BINDER TYPE, AGGREGATE SIZE CLASSIFICATION, AND BINDER/AGGREGATE VOLUME RATIO OF THE MORTAR SAMPLES. THE BINDER TYPE INCLUDES THE ATOMIC PERCENTAGE OF SILICON IN RELATION TO THE TOTAL AMOUNT OF SILICON AND CALCIUM, WHEREAS THE HYDRAULIC INDEX (HISTA) IS CALCULATED FROM SIMULTANEOUS THERMAL ANALYSES

frequently extracted together (Dusar, et al., 2009). The sandy limestone of the Brussels Formation (or Brusselian stone) consists of rubbles arranged in fairly regular rows and contains mainly calcium carbonate (CaCO₃). Its content ranges from 60 to 80%, while that of the sandy limestone of the Lede Formation (or Lede stone) 50 to 70% (Camerman, 1955: 5–26, 51–64, and Groessens, 2011: 165–75). Due to its higher CaCO₃ content, the Brusselian stone was preferred above the Lede stone for the lime production, the latter being mainly used as a building stone.



(e)

(f)

FIGURE 3 Thin section photomicrographs from a selection of mortar samples: (a) lime lumps containing several quartz grains as well as altered black-coloured glauconite grains (sample M6, bedding mortar from the interior walls of the Aula Magna), (b) incompletely burnt limestone fragment containing quartz grains, altered reddish glauconite grains, microfossils (small foraminiferal tests), and patches of microcrystalline calcite (sample M15, façade rendering on a presumably fourteenth-century dwelling), (c) completely burnt microfossil (small foraminiferal test) found in the binder matrix (samples M4, bedding mortar from the interior walls of the Aula Magna), (d) completely burnt bioclast (urchin spine) found in the binder matrix (M9, bedding mortar from the interior walls of the chapel), (e) microsparitic sandy limestone fragment, containing an unweathered glauconite grain and an ostracod shell fragment (sample M14, bedding mortar from the cellar vault of a presumably fourteenth-century dwelling), and (f) almost pure limestone fragment (sample M11, probable bedding mortar from the late eighteenth-century transition of the chapel).

According to a large register of the city of Brussels, written in old Dutch and called the *Percquement boeck mette taitsen*, containing notes concerning the construction of the Aula Magna built between 1452 and 1460, the lime was produced by firing roughly broken fragments of Diegem stone or, possibly, Evere stone (Dickstein-Bernard, 2007: 37–64). The Diegem stone was considered as the best quality, while the Evere stone of an inferior quality. Diegem and Evere refer to extraction sites of the Brusselian stone and are situated north of the Coudenberg palace, at a distance less than 10 km. Both quarries are in the vicinity of the Senne river. The lime was produced partly close to the extraction sites and partly at the north side of the city of Brussels near the ancient quays of the Senne (Camerman, 1955: 5–26, 51–64). These lime kilns of Brussels were located about 2 km away from the Coudenberg palace. Today, a part of the current quays bears the name Lime Quay (*Kalkkaai* in Dutch or *Quai à la Chaux* in French) as witness of these lime-producing activities.

EDX analyses of the binder, containing quartz-bearing lime lumps, result in an average silicon content ranging from 4.9 to 11.6 atomic%, most of the values being around 9 atomic% (Table 2). It has to be mentioned that mortar samples M1, M16, and M17 are characterized by a significantly higher silicon content (up to 33.5 atomic%). Atomic% Si values higher than 20 were not taken into account because it is assumed that the binder of these mortars is partially dissolved because of the long term underground conditioning. This hypothesis is supported by microscopic observations of thin sections. The binder can hence be classified as a slightly hydraulic lime (Van Balen, et al., 2003). These results confirm that the lime derived from the fired Brusselian stone was naturally slightly hydraulic due to the presence of small amounts of clay minerals (Camerman, 1955: 5–26, 51–64). A typical result of a simultaneous thermal analysis of a mortar sample consisting of Brusselian lime is illustrated in Figure 4a.

Mortar from the eighteenth-century transition

EDX-analyses of the binder of the mortar lifted from the late eighteenth-century transition of the chapel resulted in an average silicon content of 2.5 atomic%. Such a low value indicates the use of air lime as a binder. Additionally, the results obtained from the simultaneous thermal analysis confirm the use of air lime and reveal the addition of some gypsum (Figure 4b). A limestone fragment, almost entirely consisting of CaCO₃, was observed in the thin section (Figure 3f). The firing of a limestone similar to the fragment detected in the thin section might form air lime. It is supposed that this detected fragment originates from the stone used for the production of the lime binder. However, on the basis of this sole unburned limestone fragment, it is very difficult to identify with high precision the source of the air lime. Nevertheless, the high purity of the lime might indicate the use of lime from Wallonia produced by firing rather pure limestone of the Primary geological era (Anonymous, 1930: 893-940). The use of Walloon air lime in this mortar seems difficult to justify because the production activities of lime using the Brusselian stone lasted until the early twentieth century (Camerman, 1955: 5-26, 51-64). Knowing that the chapel was spared during the fire of 1731 and destroyed some forty years later for the construction of the new royal district, while its cellars underneath were preserved and integrated in the neoclassical buildings (Bonenfant & Fourny, 2007: 67-112), this mortar could be much younger than previously thought.

Identification of the aggregate

All mortar samples contain an aggregate of a similar mineralogical composition: quartz sand with minor amounts of potassium feldspar, glauconite, flint, and micro-fossils/bioclasts. The K-feldspars and glauconites present in the quartz sand can be recognized in the SEM-EDX mapping presented in Figure 5, while Figure 6 illustrates some microfossils/bioclasts making part of the aggregate (small foraminiferal tests and urchin spines).

Rather fine sand was used as aggregate for all mortars (Figure 7), except M₃ and M₁₁. The sand of the mortar lifted from the cellars of the chapel dating from late eighteenth century (sample M₁₁) is a little coarser, while the one of sample M₃ (living quarters, fourteenth century) corresponds to a middle coarse sand.

Both the mineralogical composition and the granulometric properties are comparable to the sands from the Brussels Formation (Brusselian sands). This formation includes several types of sand, more or less well defined and situated under or next to each other, forming elongated, irregular bodies with possible alternations and gradual transitions (Gullentops & Wouters, 1996, and Broothaers, 2000). Within the Brusselian sands, three types can be identified: Kraaiberg, Neerijse, and Diegem (from bottom to top). The Neerijse and Diegem sand types are characterized by the finest granulometry (80 wt% of the grains are between 0.080 and 0.250 mm) and by the lowest glauconite content (lower than 10 wt%). From the evaluation of the aggregate features of the mortars of the Coudenberg site, it seems that all mortars, except M11 and M3, contain a Brusselian sand of the Neerijse or the Diegem-type. The coarser sand of the mortars M11 and M3 could correspond to the Kraaiberg-type since 80 wt% of the grains is between 0.160 and 0.630 mm. However, the Kraaiberg-type sand is characterized by a glauconite content up to 20 wt%, which is not observed in both mortars.

It is well known that the extraction of Brusselian sands was a by-activity of stone extraction (Camerman, 1955: 5–26, 51–64). Since lime and building stones from the Diegem site were used for the construction of the Aula Magna (Dickstein-Bernard, 2007: 37–64), the sand used as mortar aggregate could originate from the same place. For the other mortar samples, it is difficult to propose a precise source for the sand, since one extraction site can provide different sand types according to the stratigraphic level of the quarry. However, it is more likely that heavy goods like sand come from extraction places situated in the vicinity of the Coudenberg palace, since Brusselian sands were formerly also exploited inside of the city of Brussels (Camerman, 1955: 5–26, 51–64, and Groessens, 2011: 165–75).

The binder/aggregate ratio

With the exception of M17, M14, and M11, all mortar samples show a similar binder/sand volume ratio close to 1:1, regardless of their age (twelfth to the seventeenth century) and function (bedding mortar, plastering, rendering, or infill). A significant lower ratio of 1:2 is observed for samples M17 and M14, which could be explained by the imperfect mixing of the lime with the sand or by the partial dissolution of the binder, as discussed before. The most recent bedding mortar M11 (cellars of the chapel, eighteenth century) is, in contrast to all other mortars, characterized by a very high binder/aggregate ratio of 3:1.



FIGURE 4 Results from the simultaneous thermal analysis of (a) the bedding mortar (sample M6) from the interior walls of the Aula Magna and (b) the bedding mortar (sample M11) from what is considered as the late eighteenth-century transition of the chapel. The weight loss determined by thermogravimetric analysis (TG, wt%, green curve) and the result of the differential scanning calorimetric analysis (DSC, mW/mg, blue curve) are both presented. The pic round 140° C in Figure 4b reveals the presence of gypsum in small amounts.



FIGURE 5 Elemental composition of the bedding mortar (sample M5) from the collapsed vault of the Aula Magna (Figure 2), obtained by means of SEM-EDX on a polished section. The main elements silicon, calcium, iron, potassium, magnesium, and aluminium are represented. The quartz grains are recognized in the mapping of silicon, while the lime binder is mainly visible in the mapping of calcium. Some glauconite grains $[K(Fe,AI,Mg)_2(Si,AI)_4O_{10}(OH)_2]$ can mainly be recognized in the mapping of iron, while the K-feldspars $(KAISi_3O_8)$ are visible in the mappings of potassium and aluminium.



FIGURE 6 Thin section photomicrographs from a selection of microfossils derived from the sand used for the preparation of (a) the bedding mortar from the interior walls of the Aula Magna (sample M6, small foraminiferal test) and (b) the bedding mortar from the walls of a presumably fourteenth-century dwelling (sample M13, urchin spine).



FIGURE 7 Cumulative grain size distribution of the sands used in the different mortars.

For all mortar samples from the twelfth to the seventeenth century, the binder/ aggregate volume ratio determined by acid attack does not represent the exact ratio used by the elders because they worked with a naturally sandy lime, as discussed before. For this reason, they probably have added less than one volume of sand to one volume of lime. Historical sources mention a mortar composition of one *muid* (obsolete unit of volume) of lime for three carts of sand for the mortars of the Aula Magna (Dickstein-Bernard, 2007: 37–64). Assuming a binder/sand volume ratio of approximately 1:1, it is possible to estimate the ratio between these old units of volume: one *muid* of lime had to be more or less equivalent to three carts of sand.

Conclusion

Thirteen out of sixteen mortars dating from the twelfth to the seventeenth century are composed of slightly hydraulic lime and fine quartz sand with a binder/aggregate volume ratio close to 1:1. All mortar samples contain quartz-bearing lime lumps. On the basis of the microscopic findings, it is assumed that the stone used for the lime production is a microsparitic sandy limestone containing glauconite grains and microfossils/bioclasts (small foraminiferal tests, urchin spines, ostracod shells). This composition corresponds in all aspects with the limestone rubbles of Eocene age of the Brussels Formation (or Brusselian stone). The use of Brusselian stone from Diegem or Evere for the production of the lime binder is confirmed by historical records contemporaneous to the construction of the Aula Magna (1452–60). The lime binder derived from Brusselian stone can be qualified as slightly hydraulic as EDX analyses revealed an average silicon content of about 9 atomic%. The sand used as aggregate also originates from the Brussels Formation (Brusselian sands). The fine sand has features typical for the Neerijse or Diegem-type sands. The granulometry of the middle coarse sand of the mortar M₃ is closely related to the Kraaiberg-type. It should be noted that the binder/sand volume ratio determined by means of an acid dissolution reaction does not exactly represent the ratio used by the elders because they worked with a naturally sandy lime. They probably added less than one volume of sand to one volume of lime. No significant differences in the lime/sand mixture proportion were obtained between the bedding, plaster, render or infill mortars.

The most recent, presumably late eighteenth-century, bedding mortar lifted from the chapel differs from all others and contains only lime lumps without quartz grain inclusions. EDX analyses of the binder resulted in an average silicon content of 2.5 atomic%, indicative for air lime as is confirmed by the simultaneous thermal analysis which also revealed the presence of gypsum in small amounts. The high purity of the lime might indicate the use of a lime from Wallonia produced by firing rather pure limestone of the Primary geological era. The composition of a limestone fragment found in the mortar justifies this assumption. However, the use of Walloon air lime for the late eighteenth-century transition of the chapel is unlikely as the lime production activities using the Brusselian stone lasted till the twentieth century. It is therefore supposed that this mortar, furthermore characterized by a very high binder/ aggregate volume ratio of 3:1, is linked to interventions post-dating the eighteenthcentury integration of the chapel in neo-classical surrounding buildings. The fine to middle coarse sand used as aggregate resembles Brusselian sands, as it was the case for the mortars from the twelfth to the seventeenth century.

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