Historic mortars from the Coudenberg archaeological site: characterization and source of raw materials

L. Fontaine, R. Hayen and H. De Clercq

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

Abstract

The Coudenberg archaeological site consists of the remains of the former Palace of Brussels, made up of successive building phases ranging from the 12^{th} to the 18^{th} century. After the fire of 1731, the ruins of the Palace were demolished for the purposes of a new urban project: the Royal Square. Although the Palace disappeared from the urban landscape, remains of the basement level are still present beneath the square, and have been made accessible following excavation campaigns carried out over the past twenty-five years. In the case of this study, 17 mortars belonging to the basement of various well-dated building phases were characterized by means of a combination of techniques (optical and scanning electron microscopy, simultaneous thermal analysis as well as an acidic treatment) aiming to identify the mortar composition and to find out the geological source of the raw materials. The binder of the mortars from the 12^{th} to the 17^{th} century is made up of slightly hydraulic lime. The systematic presence of quartz-bearing lime lumps, burnt glauconite grains and ghosts of microfossils/bioclasts allows identifying the raw material used for lime production: the microsparitic sandy limestone rubbles in Brusselian sands of Eocene age (Brusselian stone). The use of Brusselian lime is confirmed by archival documents contemporaneous to the Palace's construction. The granulometry of the sand used as aggregate is comparable to that of Brusselian sands. The binder/sand ratios determined are close to 1:1 in volume units but don't represent exactly the mixing ratio used by the masons in the past since a naturally sandy lime was used. No significant compositional changes were noticed related to the building phases or the mortar function. Finally, both the binder and the binder/sand mixing ratio of a more recent mortar (presumably late 18^{th} century) are distinctly different from the earlier mortars.

Keywords: mortar characterization, raw material, Brusselian lime, Brusselian stone, Brusselian sands

1. Introduction

This paper presents the results of a study of historic mortars from the Coudenberg archaeological site, which consists of the remains of the former Palace of Brussels. This palace was one of the largest and most beautiful of Europe's princely residences up to the beginning of the 18^{th} century. Successive sovereigns had transformed a small 12^{th} century castle into a sumptuous royal residence, but in 1731 a major fire destroyed the whole complex. Buildings that escaped the blaze were partially demolished for the purpose of a new urban planning project in the district at the end of the 18^{th} century — the *Place Royale* (Royal Square); and so the lower parts of certain buildings were preserved, while others were buried. Although the Palace disappeared from the urban landscape, traces of the past are still present beneath the square, and have been made accessible to the public following various excavation campaigns carried out over the past twenty-five years.

Consisting of an archaeological site and a museum of history and archaeology, the Coudenberg constitutes one of Brussels' major archaeological locations (Figure 1). The archaeological site consists of the cellars of the main building, the lower levels of the palatine chapel and the Aula Magna (the great banqueting hall) plus a section of the old *Rue Isabelle* (Isabella Street). The museum itself is housed within the remains of the former *Hôtel d'Hoogstraeten* (Hoogstraeten House), an aristocratic town house adjoining the former palace (Cnockaert et al. submitted).

The characterization of mortars belonging to various well-dated building phases will allow to identify possible changes in composition through time, according to the mortar function and the provenance of raw materials.

Figure 1: Plan overlaying the Coudenberg archaeological site and museum on the current *Place Royale* (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 marked on the plan. The samples M12 to M15 were lifted on the outer corner of the Aula Magna, almost at the level of the Royal Square (a level several meters above the underground archaeological site), where some remains of ancient dwellings pre-dating the construction of the Aula Magna have been discovered.

2. Materials

Seventeen mortar samples were lifted from four building phases of the archaeological site (Figure 1), more precisely: (a) the cellars of the former living quarters of the palace's main building, that ~~are~~ most likely belong to the oldest part of the site dating from the 12^{th} to the 15^{th} century, (b) the rooms underlying the Aula Magna, the main banqueting hall built between 1452 and 1460, (c) the warehouse space under the chapel, dating mainly from the 16^{th} century and subjected to various transitions in the 18^{th} century, and (d) the remains of ancient dwellings pre-dating the construction of the Aula Magna, dated back to the 14^{th} century or perhaps earlier. An overview of all analysed mortars is given in Table 1.

Table 1: Location, description, date and code of the 17 mortar samples. The dates were obtained from a historic built investigation led by archaeologists of the Royal Archaeological Society of Brussels.

Location	Description	Date	Code
(a) Living quarters (cellars)	Bedding mortar, vault	15^{th} century (transformation)	M1
	Bedding mortar, exterior wall	12^{th} century (original construction)	M2
	Bedding mortar, exterior wall and vault	14^{th} century (transformation)	M3
(b)	Bedding mortar, interior wall	1452-1460	M4
	Bedding mortar, collapsed vault	1452-1460	M5

Aula Magna (cellars)	Bedding mortar, interior wall	1452-1460	M6
	Plaster, interior wall	Before 1731	M7
	Infill mortar, exterior wall	1452-1460	M16
	Infill mortar, exterior wall	1452-1460	M17
(c)	Infill mortar, column	16th century	M8
	Bedding mortar, interior wall	16th century	M9
Chapel (cellars)	Plaster, 16th century interior wall	Unknown	M10
	Bedding mortar, interior wall	18th century (transformation)	M11
(d)	Bedding mortar, exterior wall	Before 14th century	M12
	Bedding mortar, cellar interior wall	14th century	M13
Ancient dwellings	Bedding mortar, cellar vault	14th century	M14
	Render, exterior façade	14th century	M15

3. Methodology

Thin sections for petrographic investigation by means of optical microscopy (Zeiss, Axioplan equipped with a DeltaPix Invenio 5DII high resolution digital camera) as well as polished sections for scanning electron microscopy (SEM-EDX, Jeol JSM-6300) were prepared from each mortar sample. Since the silicon content can be considered as a measure for the hydraulicity of the binder, minimum ten EDX analyses were performed, to determine an average SiO_2 weight percentage in relation to the total amount of SiO_2 and CaO. The binder in lime lumps and the intergranular binder look similar in texture and colour under the optical microscope. Hence the SiO_2 content of the binder was preferentially measured in lumps to avoid any contamination from the sand constituents.

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). At higher temperatures, in general up to 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 hydraulicity index (HI_{STA}) is calculated as the percentage of weight loss between 200 °C and T_{onset} of the total weight loss in the temperature range of 200-800 °C.

The analytical part is completed by an acidic 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/dm^3 and of 1.10 kg/dm^3 for respectively quartz sand and lime binder.

Finally, the granulometry of the aggregate is approached by setting up a curve consisting of the mean diameter of 300 sand grains measured in thin sections. The aggregates are classified according to the definitions given by the Belgian standard NBN B11-011:1981 '*Building sands: definition and identification of the granulometry*'.

4. Results and discussion

4.1First observations

The petrographic investigation of the thin sections revealed that all mortar samples look very similar, despite their large period of application ranging from the 12th up to the 17th century. Only the composition of mortar sample M11 clearly deviates. This mortar belongs to a presumably 18th century transformation of the warehouse spaces of the chapel as they were integrated in the cellars of the neoclassical surrounding buildings. Table 2 summarizes the results of the analytical procedure carried out on the mortar samples.

Table 2: Binder type, aggregate size classification and binder/aggregate volume ratio of the mortar samples. The binder type includes the weight percentage of silica (SiO_2) in relation to the total amount of silica and lime (CaO). The hydraulicity index (HI_{STA}) is calculated from simultaneous thermal analyses. Wt.% SiO_2 higher than 12 and HI_{STA} higher than 30% are supposed to reflect a dissolution of the $CaCO_3$ of the binder (asterisks indicate samples where the binder appears partially dissolved in thin section).

Place	Code	Binder (wt.% SiO_2, HI_{STA}(%))	Aggregate classification (max. grain size in mm)	Binder/aggregate ratio (volume ratio)
(a) Living quarters (cellars)	M1	slightly hydraulic lime* (20.3, 33.4)	fine sand (0.35)	ca. 2:3
	M2	slightly hydraulic lime (9.1, 24.0)	fine sand (0.36)	ca. 4:3
	M3	slightly hydraulic lime (8.7, 28.7)	middle coarse sand (0.54)	ca. 1:1
(b) Aula Magna (cellars)	M4	slightly hydraulic lime (8.7, 14.3)	fine sand (0.33)	ca. 2:3
	M5	slightly hydraulic lime (10.8, 35.8)	fine sand (0.34)	ca. 1:1
	M6	slightly hydraulic lime (9.4, 19.7)	fine sand (0.34)	ca. 3:4
	M7	slightly hydraulic lime (11.1, 26.4)	fine sand (0.36)	ca. 1:1
	M16	slightly hydraulic lime* (33.5, 57.5)	fine sand (0.36)	ca. 2:3
	M17	slightly hydraulic lime* (32.9, 68.8)	fine sand (0.38)	ca. 1:2
(c) Chapel (cellars)	M8	slightly hydraulic lime (8.0, 24.0)	fine sand (0.47)	ca. 5:4
	M9	slightly hydraulic lime (7.2, 22.1)	fine sand (0.41)	ca. 4:3
	M10	slightly hydraulic lime (8.2, 22.7)	fine sand (0.33)	ca. 1:1
	M11	air lime (2.5, 14.6)	fine to middle coarse sand (0.38)	ca. 3:1
(d) Ancient dwellings	M12	slightly hydraulic lime (8.0, 29.3)	fine sand (0.34)	ca. 1:1
	M13	slightly hydraulic lime (4.9, 15.8)	fine sand (0.31)	ca. 1:1
	M14	slightly hydraulic lime (11.6, 25.4)	fine sand (0.27)	ca. 1:2
	M15	slightly hydraulic lime (9.8, 33.5)	fine sand (0.26)	ca. 4:5

4.2Identification of the binders

a. Mortars from the 12th to the 17th century

A distinctive feature noticed in all mortars dating from the 12^{th} to the 17^{th} century is the presence of lime lumps, which are characterized by the inclusion of quartz grains as well as varying amounts of black coloured grains (Figure 2a). The grains have a size inferior to 100 µm, hence significantly finer than the ones used as aggregate. Nevertheless, lime lumps typical for of a limestone that was not completely burnt were noticed containing quartz grains of a comparable granulometry as the aggregate as well as amber-coloured grains (Figure 2b). Both amber and black coloured grains are attributed to a specific mineral:

glauconite. Glauconite is an iron-rich phyllosilicate exhibiting in a non-weathered state a green tint. During the firing process, oxidative reactions take place by which the grains turn reddish to black coloured with increasing heating temperature. Black-coloured glauconite grains are also found dispersed in the binder, rendering their distinction with coal fragments used during the firing process of the limestone difficult.

Besides quartz grains and underburned glauconites, some remains of small foraminiferal tests (microfossils) are noticed in lumps that are typical for an incompletely burnt limestone (Figure 2b), 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 2c and 2d).

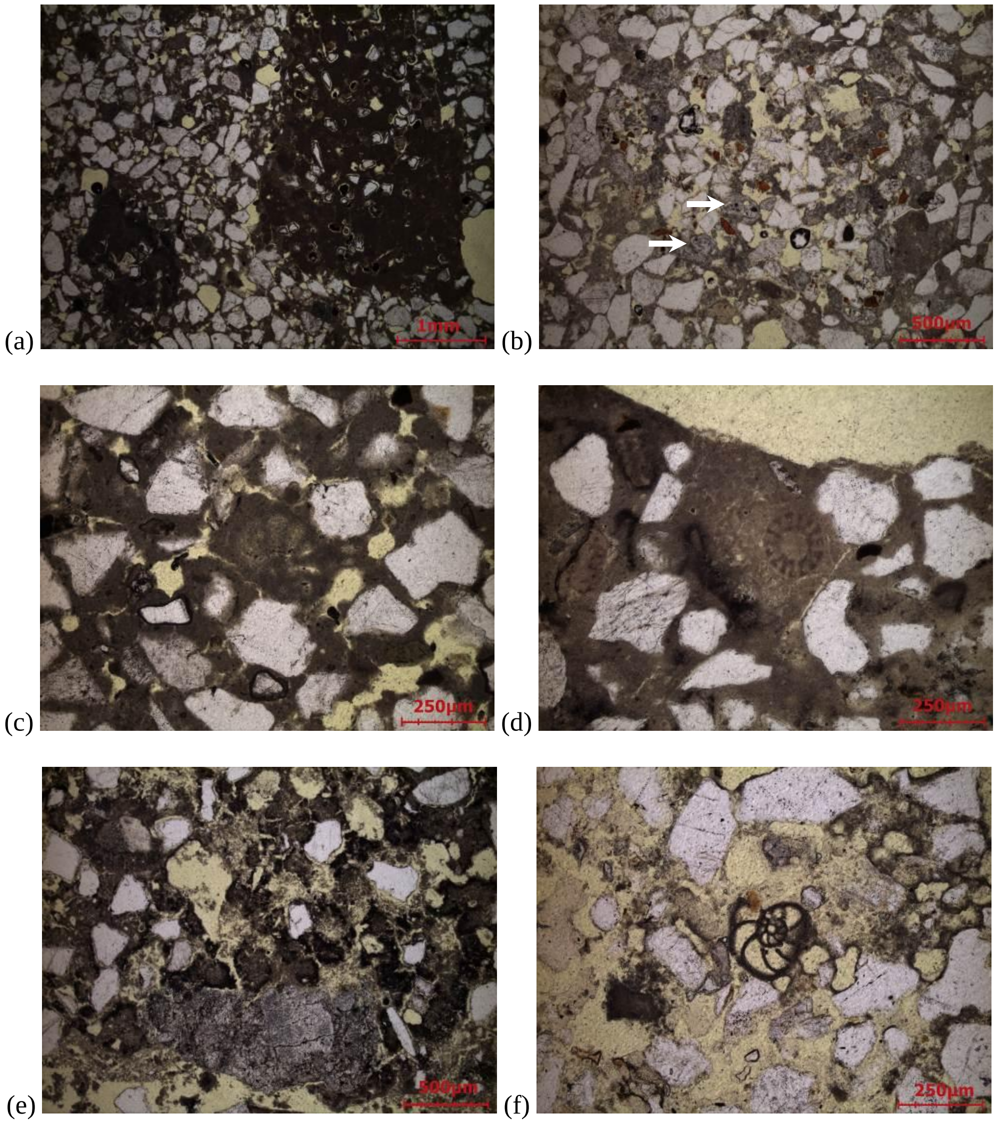

Figure 2: Thin section photomicrographs from a selection of mortar samples. (a) Lime lumps containing quartz grains and burnt black-coloured glauconite grains (sample M6), (b) an incompletely burnt limestone fragment containing quartz grains, amber-coloured glauconite grains, small foraminiferal tests (arrows) and patches of microcrystalline calcite (sample M15), (c) and (d) ghosts of microfossil/bioclast preserved in the binder matrix, respectively a small foraminiferal test (sample M4) and an urchin spine (sample M9), respectively, (e) an almost pure limestone fragment (sample M11) and (f) a small foraminiferal test deriving from the sand (sample M13).

From these microscopic findings, it is confirmed that a microsparitic sandy limestone containing glauconite grains and microfossils/bioclasts, like small foraminiferal tests and urchin spines 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). These formations are in a stratigraphic continuity, with the Lede Formation on top of the Brussels Formation, and were consequently 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 strata containing mainly calcium carbonate ($CaCO_3$). Its $CaCO_3$ content ranges from 60 to 80 per cent, while that of the sandy limestone of the Lede Formation (or Lede stone) varies between 50 and 70 per cent (Camerman 1955, Groessens 2011). Due to its higher $CaCO_3$ content, the Brusselian stone was preferred above the Lede stone for the lime production, the latter being mainly used as a building stone.

According to an ancient register of the city of Brussels, the *Perquement boeck mette taitsen* (old Dutch), containing notes related to 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), with the Diegem stone being considered of a superior quality. Diegem and Evere refer to extraction sites of the Brusselian stone situated at about 10 km to the north of the former Palace of Brussels. Both quarries are located in the vicinity of the Senne River. The lime was produced close to the extraction sites and partly near the ancient quays of the Senne at the north side of the city of Brussels (Camerman 1955). The last lime kilns were located at a 2 km distance from the former Palace of Brussels. Today, the current name Lime Quay (*Kalkkaai* in Dutch or *Quai à la Chaux* in French) still witnesses these historic lime production activities.

EDX analyses of the binder result in an average SiO_2 content ranging from 4.9 to 11.6 wt.%, most of the values being around 9 wt.% (Table 2). The SiO_2 content of lime lumps is generally some percent lower than that of the intergranular binder, suggesting a contamination from the sand constituents. It has to be mentioned that mortar samples M1, M16 and M17 are characterized by a significantly higher SiO_2 content (up to 33.5 wt.%). Wt.% SiO_2 values higher than 20-12 were not considered any further as it is assumed that the $CaCO_3$ of the binder is partially dissolved as a result of the long term underground conditioning. This hypothesis is supported by microscopic observations of the 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 some clay minerals (Camerman 1955).

b. Mortar from the 18th century transition

EDX analyses of the binder of the mortar lifted from the late 18th century transition of the chapel resulted in an average SiO_2 content of 2.5 wt.%. 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 presence of some gypsum. A limestone fragment, almost entirely consisting of $CaCO_3$, was observed in the thin section (Figure 2e). The firing of such a limestone might form air lime. It is supposed that the 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 almost impossible to identify with high precision the source of the air lime. Nevertheless, the high $CaCO_3$ content of the lime might indicate the use of lime from Wallonia produced by firing rather pure limestone of the Secondary Primary geological era (Anonymous 1930). However, the use of Walloon air lime in this mortar seems difficult to justify because the lime production activities of lime using the Brusselian stone lasted until the early 20th century (Camerman 1955). 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 and Fourny 2007), this mortar could be much younger than previously thought.

4.3Identification of the aggregate

All mortar samples contain an aggregate of a similar mineralogical composition: quartz sand with minor amounts of potassic feldspar, glauconite, flint and microfossils/bioclasts. Figure 2f illustrates a small foraminiferal test belonging to the aggregate.

Rather fine sand was used as aggregate for all mortars (Figure 3), except for samples M3 and M11. The sand of the mortar lifted from the cellars of the chapel dating from late 18th century (sample M11), is only a little coarser while the one of sample M3 (living quarters, 14th century) corresponds to a middle coarse sand.

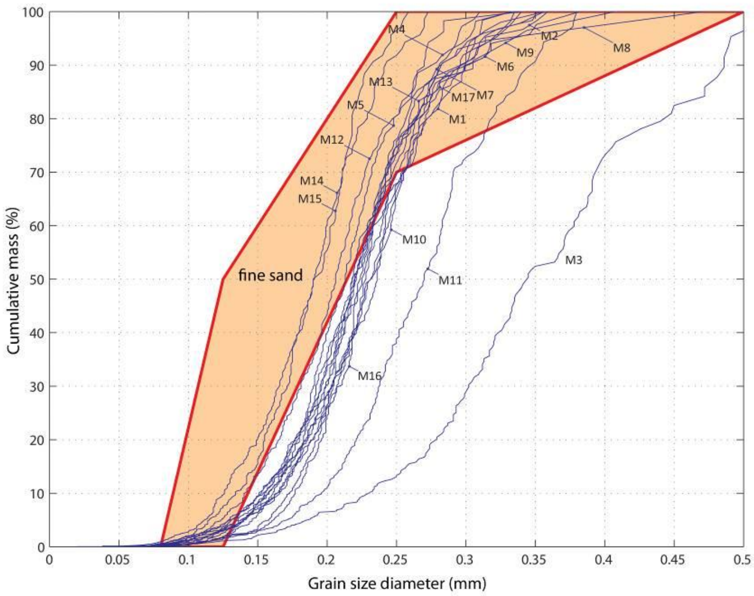

Figure 3: Cumulative grain size distribution of the sands used in the different mortars.

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 (Broothaers 2000). Within the Brusselian sands, three types are classically recognized: 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 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 has a size 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 wasn't a core activity but more a side activity of stone extraction (Camerman 1955). Since lime and building stones from the Diegem site were used for the construction of the Aula Magna (Dickstein-Bernard, 2007), it is likely that the sand used as aggregate originates from the same extraction place. For the other mortar samples, it is difficult to allocate a precise source of the sand, since one extraction site can provide different sand types according to the exploited stratigraphic level. It has to be mentioned that Brusselian sands were formerly also exploited within inside of the city of Brussels (Camerman 1955, Groessens 2011).

4.4The binder/aggregate ratio

With the exception of M11, M14 and M17, all mortar samples show a similar binder/sand volume ratio close to 1:1, regardless their age (12th to the 17th century) and function (bedding mortar, plastering, rendering or infill). A significant lower ratio of 1:2 is observed for samples M14 and M17, which could be explained by the an imperfect mixing of the lime with the sand or by the partial dissolution of the binder, as discussed in point 4.2. The most recent bedding mortar M11 (cellars of the chapel, 18th century) is, in contrast to all other mortars, characterized by a very high an extreme binder/aggregate ratio of 3:1.

For all mortar samples from the 12th to the 17th century, the binder/aggregate volume ratio determined by acid attack surely deviates from the exact ratio used applied by the masons because they worked with who used a naturally sandy lime, as discussed in point 4.2. For this reason, they probably have added somewhat less than one volume of sand to one volume of lime.

5. Conclusion

All mortars dating from the 12th to the 17th century (16 of 17 samples) 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 like small foraminiferal tests and urchin spines. This composition corresponds in all aspects with the limestone rubbles of Eocene age of the Brussels Formation (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 (1452-1460). The lime binder derived produced from Brusselian stone can be qualified classified as slightly hydraulic as EDX analyses revealed an average SiO_2 content of about 9 wt.%. The quite fine sand used as aggregate also originates from the Brussels Formation (Brusselian sands). No significant differences in the lime/sand mixture proportion are noticed between bedding, plaster, render or infill mortars. The binder/sand volume ratio determined by means of an acid dissolution reaction does, however, not represent exactly the ratio used deviates from the one applied by the masons at the time of construction because as they worked with used a naturally sandy lime.

The most recent, presumably late 18th century, bedding mortar lifted from the chapel differs from all others. Although the fine to middle coarse sand used as aggregate can could be attributed to a Brusselian sand, this mortar is characterized by a very high binder/sand volume ratio of 3:1 and by the use of an air lime binder containing some gypsum. The high purity of the lime might indicate the use of a lime from Wallonia produced by firing rather pure limestone of the Secondary Primary geological era as it was suggested by from a limestone fragment found detected in the thin section. However, the use of Walloon air lime for the late 18th century transition of the chapel ~~is~~ seems unlikely as the lime production activities using the Brusselian stone lasted till the 20th century. It is therefore supposed that this mortar is linked to interventions post-dating the 18th century integration of the chapel into the surrounding neoclassical buildings.

References

Anonymous (1930), Les ressources du sol belge en matières utiles – Annexe à la légende générale de la carte géologique détaillée de la Belgique, Extrait des Annales des Mines de Belgique, 30 (3), 893-940

Bakolas, A., Biscontin, G., Moropoulou, A. and Zendri, E. (1998), Characterization of structural byzantine mortars by thermogravimetric analysis, Thermochimica Acta, 321,151-160

Bonenfant, P. and Fourny, M. (2007), La dernière fenêtre sculptée de l'ancienne chapelle de Charles Quint à Bruxelles, Annales de la Société Royale d'Archéologie de Bruxelles, 68, 67-112

Broothaers, L. (2000), Zandboek Vlaanderen, Ministerie van de Vlaamse Gemeenschap, Afdeling Natuurlijke Rijkdommen en Energie, 112

Camerman, C. (1955), Le sous-sol de Bruxelles et ses anciennes carrières souterraines, Annales des travaux publics de Belgique, 2-3, 5-26 and 51-64

Cnockaert, L., Demeter, S., Henriques de Granada, A. and Honoré, F. (submitted), The Coudenberg archaeological site in Brussels: the stakeholders involved in the renovation process, Conservation and Management of Archaeological sites

Dickstein-Bernard, C. (2007), La construction de l'Aula Magna au palais du Coudenberg – Histoire du chantier (1452-1461?), Annales de la Société Royale d'Archéologie de Bruxelles, 68, 37-64

Dusar, M., Dreesen, R. and De Naeyer, A. (2009), Renovatie & restauratie – Natuursteen in Vlaanderen, versteend verleden, Kluwer, 562

Groessens, E. (2011), Quelques pierres blanches au pays de la pierre bleue (Belgique), in Carriers et bâtisseurs de la période préindustrielle, Europe et régions limitrophes, Gély and Lorenz (eds.), Editions du Comité des travaux historiques et scientifiques, 165/175

Van Balen, K., Van Bommel, B., van Hees, R., Van Hunen, M., Van Rooden, M., Van Rhijn, J., Callebaut, K., Van der Loos, R. and Van der Klugt, L. (2003), Kalkboek: het gebruik van kalk als bindmiddel voor metsel- en voegmortels in verleden en heden, Rijksdienst voor de Monumentenzorg, 296