State of the Art of Research/Diagnostics of Historical Building Materials in Belgium

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Characterisation of historical building materials and diagnostics of their state of damage are not treated in one specialised institute in Belgium. Belgium has a limited amount of research institutes and universities that are generally occupied on research and diagnostics of (historical) building materials. Most of these institutes treat generally all (historical) building materials, though for historical paints for example, one institute is more specialised (KIK/IRPA in Brussels), and for historical mortars, the university of Leuven is more specialised. Several people from most of those institutes produced recently a document on "Procedures for the restoration of outer walls"[1] (soon to be published, only in Dutch and French). In this document, several procedures on how to analyse a historical structure, on how to characterise different historical building materials, on how to clean a historical structure and on how to protect such a building are described. This document was one of the reference documents for this state-of-the-art report on historic materials and their diagnostics. Further documents are specialised studies (for example a PhD on characterisation of historical mortars from the university of Leuven) or general documents on building materials (for example a technical note from the BBRI on natural stone and test methods used to characterise natural stones,…). At the universities, students make often a "master"thesis on historical building materials in different faculties (science, engineering, and archaeology…), but a full database of all those theses's is not available at this moment.

Historical building materials in Belgium treated in institutes are: natural stones, mortars, plasters, renders, bricks, paints… For most of the materials, the characterisation of the material and diagnostics of damage are similar. In this paper, the first part will handle the characterisation of historical building materials (microscopical-mineralogical, chemical and physical-mechanical tests). The different types of analyses will be discussed generally and where possible, specialised analyses for specifical materials will be highlighted. After the discussion of the different characterisation methods, the identification of different types of damage will be discussed with some examples.

1. PART A: CHARACTERISATION OF HISTORICAL BUILDING MATERIALS

1.1 Visual analysis and sampling

For an identification of a historical material, a **visual analysis** of the whole historical structure (building) and a **macroscopical analysis** of the historical materials in-situ are the first thing to do. Colours of the material, texture, type of materials are among the first things to note. In case of mortars and plasters: type of aggregates, colour of binder and aggregates, grain sizes of aggregate… can be determined by just looking at the sample. A good macroscopical analysis is also necessary for a good (representative) sampling for the other necessary analyses. A minimum amount of sample should be taken to avoid too much damage to the historical structure. Once a

[1] **1**[11] soon to be published BBRI publication "Procedures voor de restauratie van buitenmuren"

profound macroscopical analysis (visual analysis) is executed, one knows the diversity of the materials present at the structure, the degree of damage, and based on the diversity of the material concerned, one can limit the sampling to representative samples.

Sampling of the material is also an important issue. For historical mortars, Callebaut (2000)[1] developed a scheme to take samples for different types of analyses. The same scheme can be used for other historical materials. Before sampling, one needs to define what types of analyses need to be done on the sample, to avoid too much or too less samples taken at the historical building. One has to keep in mind that sampling is often limited in time since scaffolding is not always present. Hughes and Callebaut (1999)[2] specified the minimum amount of sample and the type of sample needed (powder of larger lumps) for different types of analyses on historical mortars, but again, this can be used for other materials also. Sampling can be done by drilling or by hammer and chisel. Depending on the type of material, this needs to be specified before sampling. Sampling to analyse salts present in historical materials can not be done with water, so drilling with water is excluded. Also for historical gypsum and lime mortars/renders/plasters, sampling with water is preferably excluded, so not to dissolve the binder material.

After sampling, analysis of the historical building materials can be subdivided into **microscopical-mineralogical**, **chemical** and **physical-mechanical** analyses.

1.2 Microscopical-mineralogical analyses

For a *petrographical (microscopical) analysis*, a small amount of the historical building material is impregnated under vacuum with a very low viscosity resin, since samples are often weak, porous and friable or exhibit low bond strengths between the particles. Depending on the institute, a fluorescent resin is used or not. The advantage of using a fluorescent resin with fluorescent light microscopy is the easy observation of the micro-cracks and micro-pores. After hardening of the resin, the samples are first cut to a thickness of about 1 cm, glued on a glass plate, and finally further thinned to a thickness of 20-30 µm (depending on the materials analysed). For historical lime and gypsum mortars/renders/plasters, it is advised to avoid the use of water during preparation of thin sections. Since this type of material is not very hard, it can be cut dry, and for thinning the thin section to its final thickness, an oil-based mixture is preferred. This to avoid the dissolution of the binder in the mortars/plasters/renders (Callebaut, 2000)[2].

In table 1, a summary of what can be analysed with the different types of analyses for the different types of historical building materials is given. In the text, the different analyses and their results will be explained generally. With a petrographical analysis under an ordinary polarising microscope, the different mineralogical phases present in the historical building materials can be identified. It also allows the identification of possible damage features, such as secondary salts, cracks, … The depth of damage can also be estimated if a thin section is made from the surface to the inner part of the material. With petrography, the porosity and the texture of the materials can also be determined. The examination of thin sections is one of the most effective methods of studying historical building materials, although it should never be the sole analysis. Other analysis, like XRD or chemical analyses, are “bulk”-analyses. It is not possible to visualise what is being analysed. In a historical lime mortar for example, XRD-analysis can identify calcite and chemical analyses a large amount of CaO and CO_2, but this calcite can originate from the lime binder or from the aggregates. With petrography, one can easily determine if the aggregate in this case in carboniferous or not, and if all the calcite or the CaO and the CO_2 originates only from the binder or not. When examining thin sections, it should be kept in mind that only a small part of often very

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[1] K. Callebaut (2000), Characterisation of historical lime mortars in Belgium: implications for restoration mortars. Unpublished PhD thesis, KULeuven

[2] J. Hughes and K. Callebaut (1999), Practical sampling of historic mortars. In P.J.M. Bartos, C.J.W. Groot and J.J. Hughes (eds) Proceedings of the RILEM International Workshop “Historic mortars: characteristics and tests”, Paisley, 17-26

heterogeneous materials is studied. The analysis of several thin sections of the same material can aid in this problem, but the loss of edges, the possible loss of aggregate grains in mortars/renders/plasters for example, and the formation of pores during preparation of the thin section should be taken into account.

A more detailed microscopical analysis on a smaller scale can be executed with a *Scanning Electron Microscope (SEM)*, equipped with an Energy Dispersive X-ray spectrometer (EDX). This analysis can be used if some phases present in the materials are too small to identify with an ordinary microscope. With an EDX (or WDX)-equipment, a chemical spot analysis can be done on the phases, to have also an idea about their chemical constituents. Depending on the type of SEM, samples of several cm's can be analysed. Samples need to be coated with a gold or carbon layer before analysis.

An *image-analysis system* can be coupled with both types of microscopical analyses. With this type of equipment, a (semi)-quantitative analysis of phases or porosity can be done, based on colour of the phases.

For a mineralogical analysis of historical building materials in Belgium, a (powder) X-Ray Diffraction (XRD) analysis is often executed. This analysis allows the identification of crystalline, mineralogical phases present in the historical building materials, and therefore also the presence of possible damaging secondary mineralogical phases, such as salts. This analysis can be executed on powdered or on pristine samples, as long as the surface of the sample is flat. For powdered samples, a large amount of sample can be powdered and by quartering, a representative sample can be analysed. But for an XRD-analysis generally, only a small amount of sample is needed, even some grains of an efflorescence for example are sufficient. The advantage of this analysis if the limited amount of sample needed and the speed of analysis. Generally, however, this analysis is limited to crystalline phases that are present in quantities more than ~3 %. Another disadvantage is that the evaluation of the analysis is not very evident and is preferably done by people with some experience in XRD-analysis.

From work from Gödicke-Dettmering and Strübel (1996)[1] and Callebaut e.a. (2001)[2], it was concluded that XRD-analysis is an essential tool to identify hydraulic lime mortars and to distinguish hydraulic lime mortars from cement mortars. Gehlenite (C_2AS), a non-hydrated hydraulic phase, is typical for lower burning temperatures, and is not present in cement mortars. With an XRD-analysis, gehlenite can be identified (more difficult with other analysis-methods).

Other mineralogical analyses exist (such as differential thermal analysis), but are not frequently used in Belgium for the analysis of historical building materials. They are often even harder to evaluate than XRD-analysis, and rather complicated to execute.

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[1] T. Gödicke-Dettmering and G. Strübel (1996), Mineralogische une technologische Eigenschaften von hydraulischen Kalken als Bindemittel für Restaurierungsmörtel in de Denkmalpflege. Giessener Geologische Schriften 56, 131-154

[2] K. Callebaut, J. Elsen, K. Van Balen and W. Viaene, 19th century hydraulic restoration mortars in the Saint-Michael's church (Leuven, Belgium): natural hydraulic lime or cement? *Cement and Concrete Research*, Vol. 31/3, 2001, 397-403

Several references to publications can be found in Belgium where microscopical or mineralogical techniques are used to characterise historical building materials or to identify damage in historical building materials[1],[2],[3],[4],[5].

1.3 Chemical analyses

On the historical building materials, bulk chemical analyses or specialised chemical analyses can be done to determine the chemical compounds present in the material. As already mentioned earlier, a chemical analysis is a bulk analysis and it is often impossible to determine from what phase in the historical material the chemical compounds originate. Depending on the type of material, different kinds of acids are used to dissolve the material to be analysed. For mortar/plasters/renders, it is important to analyse separatly the binder and aggregate fractions. Therefore, the fractions need to be separated. Depending on the type of binder, different acids can be used (for an air-hardening lime mortar, a dilluted HCl-solution can be used; for a hydraulic lime mortar, first a HCl-solution and later on a $NaCO_3$-solution can be used; …). For mortars, the chemical compounds in the binder (characterisation of the binder) can be analysed by Atomic Absorption Spectrometry (AAS), Atomic Emission Spectrometry (AES), Inductive Coupled Plasma-Massa Spectrometry (ICP-MS) … Different laboratories use different methods, and for historical mortar analysis, the method is not standardised. In Belgium, mostly AAS and AES are used, after separating the binder and aggregates with a HCl-solution 1:9 (1 part of concentrated HCl and 9 parts of distilled water; Callebaut, 2000[2]). This concentration is based on years of experience at the university of Leuven (Civil Engineering laboratory). The amount of CaO, MgO, Al_2O_3 and Fe_2O_3 are determined with AAS, the amounts of Insoluble residu (IR) and SO_3 are determined by gravimetry. The amount of CO_2 is determined by volumetry and the amount of free $Ca(OH)_2$ by titration. But it needs to be mentioned once more, the chemical analysis of historical (lime) mortars is not yet standardised even on an international scale, and the description above is only what is being done in Belgium at the university of Leuven (more specialised in historical mortar analyses).

For natural stones and bricks as historical building material, chemical analysis is less used. Microscopical and mineralogical analyses are more frequently used to characterise those materials and to determine the damage present.

On historical building materials with efflorescences, chemical analysis could be used to determine the amount and type of salts present in the material. Therefor, the sample is crushed and treated with distilled water and filtered. The soluble salts are determined by analysing the amount of soluble ions in the filtrate. Mg, Ca, Na, K… are analysed with AAS for example, while Cl^-, SO_4^{2-}… are measured with spectrophotometry. This analysis is especially important for the determination of the presence of soluble salts in bricks and mortars/renders/plasters. With XRD-analysis (mineralogical analysis, see above), the mineralogical type of efflorescence can also be

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[1] K. Callebaut, W. Viaene, K. Van Balen and R. Ottenburgs, Petrographical, mineralogical and chemical investigation of 17th and 19th century lime mortars in the St.-Michael's church (Leuven, Belgium). *International Journal for Restoration of Buildings and Monuments* 5 (4), 1999, 523-542

[2] K. Callebaut and F. de Barquin, Microscopy and image-analysis as complementary tools in the study of staining of natural stones: preliminary results. *Annales géologiques des pays Helléniques*, édition spéciale, volume XXXIX, 2001, 541-548

[3] J. Hughes, A. Leslie and K. Callebaut, The petrography of lime inclusions in historic lime based mortars. *Annales géologiques des pays Helléniques*, édition spéciale, volume XXXIX, 2001, 359-364

[4] J. Elsen, P. Degryse and M. Waelkens, Mineralogical and petrographical study of ancient mortars from Sagalassos in view of their conservation. *Annales géologiques des pays Helléniques*, édition spéciale, volume XXXIX, 2001, 331-346

[5] J. Venstermans en J. Elsen (1994), Optische mikroskopie van bouwmaterialen met behulp van slijpplaatjes. WTCB-tijdschrift 1994/2, 3-9

analysed, but with a chemical analysis, the amount of salts present can be more precisely determined.

For historical paints, infra-red analysis (IR) is often used, since most analyses mentioned above are rather difficult to execute on paints (the layers are mostly too thin for a good petrographical analysis, and a lot of phases present in paints are not crystalline, so cannot be determined with XRD-analysis). Microscopical analysis with SEM is also often used, and more specialised chemical analysis with gas-chromatography is also used to determine constituents in the paint.

1.4 Physical-mechanical analyses

Depending on the amount of material present for analysis, and the questions asked at the beginning of the investigation, physical-mechanical analyses can be done to determine parameters such as strength, porosity… of the historical materials. The disadvantage of these analyses is that mostly a rather large amount of sample is needed, and that is not always possible with historical materials.

1.4.1 Porosity

A popular method for studying porosity of (historical) building materials is by mercury porosimetry. Mercury is pressed into the pores of the sample by high pressures and the volume of mercury used is measured. From these data, the pore-radius distribution and the porosity of the material can be determined. The lowest limit of pore size with this measurement is 10nm. Smaller pores are measured by adsorption analyses with nitrogen or water. The advantage of this method is that a pore-size distribution is measured, and that only small samples are needed. The disadvantage of mercury porosimetry is that since only a small sample is tested, several samples should be measured to have a representative idea about the pore-size distribution and the porosity. This method also only allows the measurement of open cilindrical pores. Due to the high pressures used in this test, the method is also not very well suited for weaker materials. The danger with those materials is that the weak pore walls break under the high pressure and that false data are obtained. This was made evident for historical lime mortars by Callebaut (2000)[2]. An alternative of porosity measurement (and pore-size distribution) is by image-analysis, as mentioned above. However, image-analysis is limited to larger pores, not on a nm-scale. At the BBRI, image-analysis and mercury porosimetry are compared for several building materials as reference tests for measuring porosity. This research is still ongoing and not yet published.

Another alternative to measure the total and open porosity, is by impregnating samples with water under vacuum and measuring the weight before and after impregnation and under water. This method is especially used for natural stone and bricks, and is specified in European standards f.e. for natural stones (NBN EN 1936 “Natural stone test method – Determination of real density and apparent density, and of total and open porosity”) and bricks used in “modern” buildings. Since these standards are made especially for “new” building materials, the amount of samples specified in the standard is rather high (6 cubic samples of 7x7x7 cm). For historical building materials, it is often impossible to obtain so much samples, but the test can be done on smaller samples, or even on cores drilled from the historical building. Due to the use of water, this method is maybe less suited for lime or gypsum mortars/plasters/renders (solubility of the binder).

Other methods exist for measuring porosity of building materials, but those mentioned here are the ones most frequently used in Belgium.

1.4.2 Compressive strength

The strength of historical building materials is sometime asked. For the compressive strength test, mostly cubic samples are needed. Those samples are tested in a press that increases gradually the force untill the material breaks. Again, mostly, a larger number of samples is asked, but for historical building materials, a smaller amount of samples can be used. This test can be used to analyse the compressive strength of natural stones (an European standard exist for

"modern" natural stones, NBN EN 1926 "Natural stone test method – Determination of compressive strength) or bricks. For historical mortars/plasters/renders, the thickness of the sample is mostly very limited, therefore, this method is mostly not used in Belgium for such types of material with limited thickness. For those types of materials, the dynamic E-modulus can give an idea of the strength of the material. For this method, prisms with a more limited thickness can also be used.

1.4.3 Water absorption

When a water column is applied on a porous material, the water penetrates the material. The water volume absorbed after a definite time is a characteristic of the material.

The ***pipe-method*** (RILEM test N° II.4 of RILEM commission 25-PEM[1]) is used to measure the quantity of water absorbed under low pressure by a definite surface of a porous material and after a definite time. This method is possible in-situ, as well as in the laboratory, to characterise for example the effect of natural weathering, of an impregnation or waterproofing treatment, … The apparatus is different if the object of measurement is vertical or horizontal. The pipe is applied on the material by interposing a tape of putty. One fills the pipe with water through the uppe opening up to the graduation 0. One can read directly on the graduated tube the quantity of water absorbed by the material in function of time (generally 5, 10, 15 minutes).

The ***water absorption coefficient (capillarity)*** test (RILEM test N° II.6. of RILEM commission 25-PEM[12]) is used to measure the amount of water absorbed in a certain time by one surface of a rectangular sample of an initially dry material that is in contact with a free watersurface. The samples are dried to constant mass. Then four of the six surfaces are made watertight. Two surfaces opposite each other are not treated. Then the samples are introduced into a tank in such a way that the rising of water occurs in the direction perpendicular to the stratification planes (for natural stones). Deionised water is added so that the samples are immersed to a height of 2 mm. The water level is kept constant throughout the test by adding new water when necessary. The tank is covered to avoid evaporation from the wet samples. At time intervals, initially very short then longer, the samples are lightly wiped with a dampened shammy cloth and quickly weighed, then put back into the tank. The time that has elapsed from the beginning of the test is noted just like the mass. The choice of the time depends on the type of material. The mass of water absorbed per unit area is plotted as a function of the square root of time. The slope of the first straight line through the origin corresponds to the water absorption coefficient A.

1.4.4 Determination of the sieving curve of aggregates (for mortars/plasters/renders)

Analysis of the sieve curve (grain size distribution of the aggregate in mortars, plasters or renders) can be done by dissolving the binder (different acids can be used based on the type of binder, see chemical analysis) and sieving the washed aggregate through gradual finer sieves. For this analysis, however, a rather large amount of sample is needed, depending on the size of the largest aggregates. Since sample size is mostly restricted by conservation philosophies and by the size of the mortar joint, this analysis is often impossible. But that doesn't exclude that if the amount of sample is available, this analysis can provide valuable information on the aggregate types and their distribution. This information is often needed for the restoration of historical mortars with a compatible restoration mortar.

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[1] Commission 25-PEM Protection et érosion des Monuments, Recommended tests te measure the deterioration of stone and to asses the effectiveness of treatment methods. Materials and Structures, vol. 13 n° 75, 175-253 (1980)

2. PART B: DAMAGE TYPES AND HOW TO IDENTIFY THEM

In this part, several types of damage often occuring on Belgian historical buildings and their materials, will be discussed (recognition of the damage…). Furthermore, some methods mentioned above that are best suited to identify the damage will be highlighted. Some examples with pictures will illustrate this part of the paper. The order in which the types of damage are discussed has nothing to do with the importance of the damage, but is random.

2.1 Frost damage on historical building materials

This type of damage is observed on all types of historical building materials (natural stones, bricks, mortars…) and is evidenced by flaking off of parts of the materials, parallel to the surface. When a section through the material is investigated, cracks parallel to the surface can be observed. This damage is especially observed in countries where winters with a lot of frost-thaw cycles are common. There exists a lot of literature on frost damage and frost mechanism in national and international literature, but that is beyond the scope of this paper.

The best method to identify frost damage in historical building materials is by petrography. A thin section is made perpendicular to the surface of the material, so that the surface and a large part of the inner material is present on the thin section. With ordinary light or with fluorescent light, cracks parallel to the surface are easily observed if frost damage is present in the material (see fig. 1). With petrography, the depth of the damage can also be estimated.

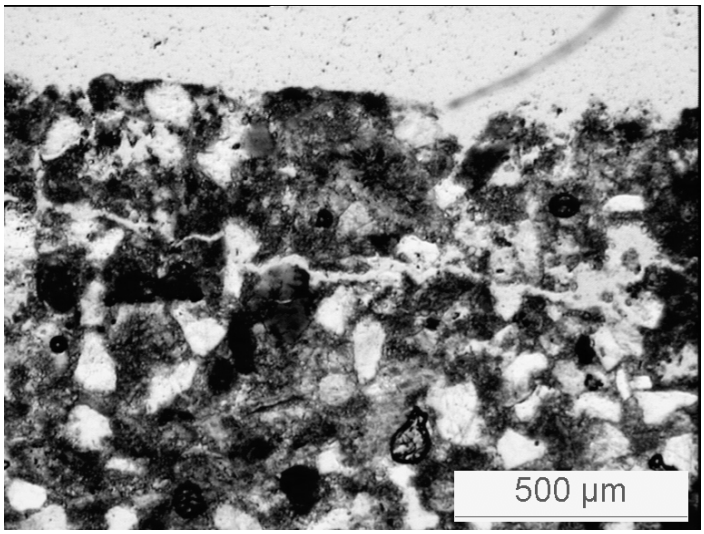

Figure 1: view with petrography of frost damage in a sandy limestone (type Balegem, frequently used in historical buildings in the north-west part of Belgium). Typical damage due to frost is the presence of cracks parallel to the surface of the stone (top of the picture). Since the picture is limited in size, only one crack is visible, but in an overview of the thin section, several parallel cracks can be observed. The depth of frost damage in this case was estimated to 1 cm.

2.2 Efflorescences and salts on/in historical building materials

Efflorescences occur as powdery white layers or bundles of salt crystals on different types of historical building materials. The best known example is on bricks, but on mortars or even natural stone, salts may also be present. If salts are only present at the surface, they have mostly an unesthetical aspect for the building materials. The danger is if salts are also present deeper in the material, and those salts can cause serious damage due to the crystallisation pressure. This crystallisation pressure can cause the material to break (formation of cracks) and eventually to pulverise. Some examples of cases with “salt damage” will be discussed.

On bricks, efflorescences are well known. They occur mostly as a powdery white layer at the surface. The damage is thus very easily recognised macroscopically. But other analyses, like

microscopical analyses, are needed to determine if there are also salts present deeper in the materials, salts that can break up the structure due to crystallisation pressure. With petrography, this can be easily checked. To identify the types of salt present in/on the brick, a mineralogical analysis (XRD of efflorescence) or a chemical analysis is needed. With a mineralogical analysis, the salt minerals present are identified (see fig. 2). With a small amount of efflorescence, an XRD-analysis is possible, and the results are given rather quickly. To have a more precise idea about the amount of salts present, a chemical analysis is necessary. If the mineralogical phases are known with an XRD-analysis, one can calculate on the base of the chemical analysis the amount of the different types of salt crystals present.

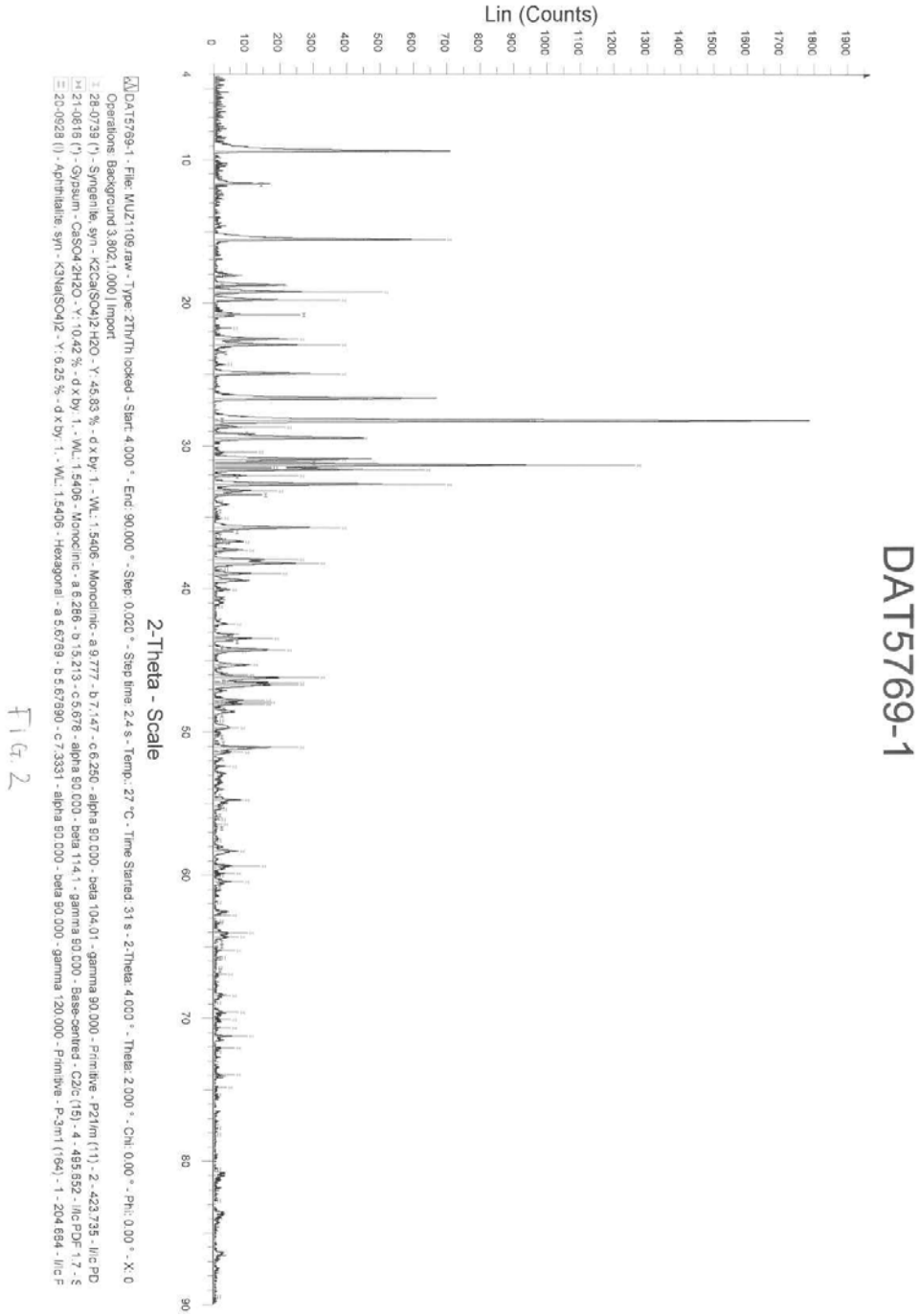

Figure 2: XRD-diagram of efflorescences on a historical brick. Analysis revealed the presence of gypsum ($CaSO_4.2H_2O$), syngenite ($K_2Ca(SO_4)_2.H_2O$) en aphthitalite ($K_3Na(SO_4)_2$).

In natural stones, secondary salts can crystallise and cause damage to the stones. A well known example is the formation of a gypsum crust on carbonate-rich natural stone (this type of damage also occurs on historical lime mortars). Calcite from the natural stone (or the mortar) reacts with SO_2 in the environment to form gypsum. This crust is not dangerous on its own, but a thin layer of the original building material dissapeared and changed into gypsum, and due to rain fall or later cleaning phases, this gypsum crust is easily removed. This way, the original building materials degrades slowly. This gypsum crust is easily recognised macroscopically as a black crust, but with microscopical analyses, the crust can be identified more in detail. If necessary, an XRD-analysis can confirm that the crust consists of gypsum.

Gypsum can also crystallise deeper in the original building materials due to the same mechanism decribed above. This is very common in calcite-rich natural stones in historical structures exposed to a high polution level. The presence of gypsum deeper in the natural stone is easily identified with petrography (or a SEM-analysis). In figure 3, gypsum (needle-like crystals) is present in pores deeper in an Euville natural stone (French bioclastic limestone, often used in Belgium at the end of the 19th and the beginning of the 20th century). If the crystallisation pressure of this gypsum becomes too large for the strength of the natural stone, the stone will crack. In figure 3, this is not yet the case, but the danger is present.

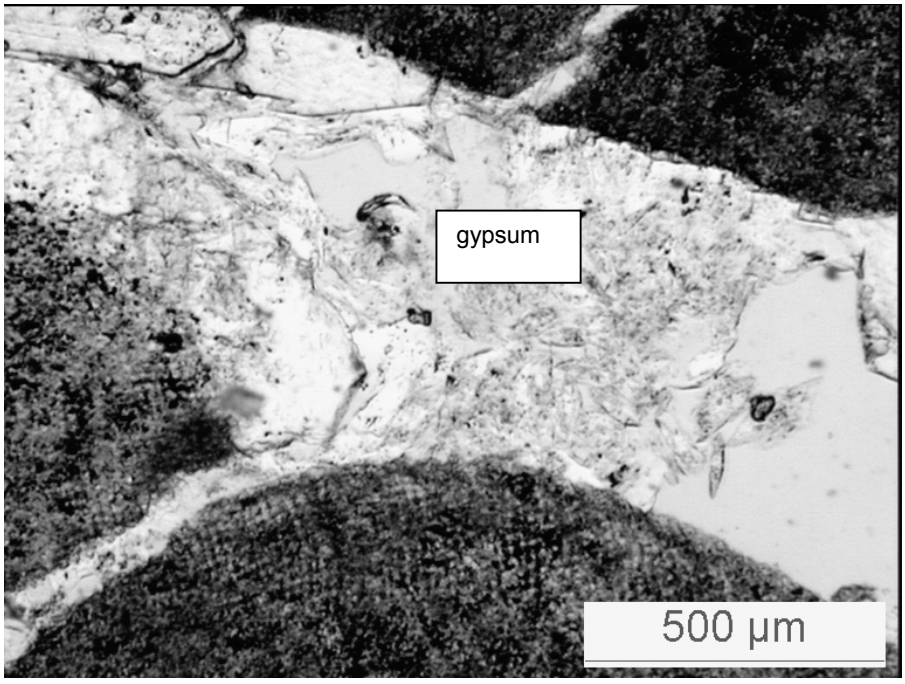

Figure 3: gypsum crystallisation in pores of a porous limestone (French Euville limestone) as observed with petrography. The crystals are present is needle-like partikels filling up the pore.

3. CONCLUSIONS

In Belgium, several institutes and universities involved in research on (historical) building materials, use different types of analyses to characterise the material and/or the damage. A summary of analyses often used in Belgium and most occuring historical building materials is given in Table 1.

Table 1: summary of the different types of analyses and what can be done with the analyses on different types of materials.

analysis	**mortar/render/plaster**	**natural stone**	**brick**	**paint**
petrography	Identification of binder, aggregate, additives; determination of porosity; presence/type of damage	Identification of minerals, fossiels + type of stone; determination of porosity; type and depth of degradation	Identification of aggregates (filler); determination of porosity; presence and type of damage	Number of layers; damage
SEM-analysis	Detailed identification of phases (also chemical with EDX or WDX); determination of porosity	Detailed identification of phases (also chemical with EDX or WDX); determination of porosity	Detailed identification of phases (also chemical with EDX or WDX); determination of porosity	Detailed identification of phases (also chemical with EDX or WDX); determination of porosity
Image-analysis	(semi)-quantification of phases and porosity			
XRD-analysis	Mineralogical identification of binder, aggregate, additives; identification of possible damaging mineralogical phases	Mineralogical identification of stone; identification of possible damaging mineralogical phases	Mineralogical identification of binder, aggregate, additives; identification of possible damaging mineralogical phases	Mineralogical identification of phases
Chemical analysis	Quantification of chemical elements in binder; determination of amount of aggregate; chemical identification of possible damaging phases	(mostly not done) identification of possible damaging phases	(mostly not done) identification of possible damaging phases	Specialised analyses (FT-IR, GC) to identify the chemical compounds in paints
Physical-mechanical analyses	Determination and quantification of porosity; determination of strength; determination of water absorption; determination of sieving curve	Determination and quantification of porosity; determination of strength; determination of water absorption	Determination and quantification of porosity; determination of strength; determination of water absorption	