HISTORIC LIME-BINDERS: AN EXAMPLE OF $19^{\rm TH}$ CENTURY DUTCH MILITARY PLAIN CONCRETE

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ABSTRACT

Before the general acceptance of Portland cement as the main binder for concrete in the late 19th century, other, locally available binders were occasionally used. In the case of the Netherlands, which did not produce Portland cement, traditional lime-based binders were not uncommon. With a strong tradition of trass production, trass-lime binders were used for hydraulic concrete structures as well. For Dutch military works such binders were commonly used for plain concrete structures in the middle and late 19th century, often in combination with crushed bricks as coarse aggregate. In this paper, the possibilities of the application of optical microscopy to characterise such concretes is discussed.

1 INTRODUCTION

The binders used in historic concrete deviate from modern binders and their correct identification is relevant for conservation and construction history. First, it gives information on the historical development of binders used for concrete and can identify rarities, novelties in application, special design intentions or that the binder was typical for its construction period. Such information can be used to gain insight into modern construction history and when evaluating heritage values related to the historic concrete. Secondly, such information is relevant for the technical aspects of the conservation process. Sound understanding of the composition of the concrete (e.g. porosity, quality of compaction, water content) and identify the properties of the concrete (e.g. porosity, quality of compaction, water content) and identify the constituents (binder, aggregate, admixtures). The identification of the used binder and the composition of the concrete is necessary in order to choose a compatible repair mortar.

Research has been previously carried out on the use of binders in the 19th century, yet mainly focusing on their application for brick work, the differences between natural cement and Portland cement, and in case of the use for concrete mainly in combination with reinforced concrete [1-3]. In this paper a first study is presented on the possibilities and limits to identify 19th century binders used in brikkenbeton, a concrete made with broken bricks as coarse aggregates and commonly used for Dutch 19th century military structures. A better

understanding of the properties of such binders is relevant as historic concrete structures are currently increasingly reviewed as part of the cultural heritage, not only in the Netherlands. In practice, lack of knowledge on the application, identification and properties of less known binders such as lime and trass applied for concrete can be noticed. Detailed material characterization is necessary for correct damage diagnosis and the development of compatible repair mortars and relevant for historic investigations.

2 HISTORIC CONTEXT

Historic concretes dating from the 19th century deviate from modern concrete in many ways, such as used binders, aggregates, and mix design. For the correct identification, an understanding of their history and use is beneficial. As many detailed documents of the 19th century concrete still exist, hypotheses can often be made on the used binder based on the period, location and building typology.

2.1 Development of binders in the 19th century

In the 19th century, building construction underwent several changes, one of them being the increase use and improvement of concrete and the increasing scientific insight into binders. In this period the term cement could refer to any type of hydraulic binder, such as hydraulic limes, pozzolana, ground brick, or natural or Portland cement [4-6]. The following properties, besides being hydraulic, were seen as characteristics of cements: the main constituent of cements was silicon dioxide (SiO₂) and the raw materials were exposed to high temperatures [7, 8].

Before the introduction of artificial hydraulic binders during the late 18th and 19th centry, lime and trass were the main binders used in the Netherlands. Trass is made by grounding tuff stone, which was imported from the German Eifel region and is a natural pozzolana with latent hydraulic properties. Only when used together with an activator such as Portland cement or lime, a hydraulic reaction will take place [9, 10]. The benefits of using trass for hydraulic structures, previously already used by the Romans, was only rediscovered by the Dutch in the 17th century , who became known for its production and international distribution [7, 11, 12].

In the 18th century a general increase of research on binders amongst engineers can be observed. The properties of hydraulic binders were investigated to overcome the dependency on Dutch tarras or Italian pozzolana. The usability of Vitruvius works, previously the main reference work for hydraulic binders, for other regions than mentioned in the works was questioned, leading to research such as by the French L. J. Vicat on the manufacture of hydraulic lime using local raw materials [4, 13]. Other milestones often mentioned in context of the history of modern binders are the works of the British J. Smeaton, who studied the hydraulic properties of different binders such as lime and trass, J. Parker's patent for Roman cement, and J. Aspdin's patent for Portland cement [see for example 4, 14, 15-18].

In the Netherlands the general use of artificial cements such as Roman cement or Amsterdamsch cement (Dutch cement made from local silt) for governmental buildings was forbidden due to bad experience, probably caused by poor production methods, and instead the use of trass advised [7], often in combination with lime. As lime mainly lime from Tournai (Doornik, Belgium), from the Dutch region of Limburg, or shell lime was used [7, 8]. Dutch examples of 19th century plain concrete structures using other limes are known, such as the sluices of the Zuidwillemsvaart canal (1824-1825), using a mix of 20 parts Belgian lime from Visé and 15 parts of trass from Cologne; the foundations of the St.-Andries sluice and the steam pumping station Bommerlerwaard (1854-1855) using a mix of 6 parts of Belgian hydraulic lime from Chaudfontaine and 7 parts trass [7, 8].

2.2 Early plain concrete military structures

Parallel to the development of binders, the possibilities of concrete was explored. Plain concrete had been used commonly before the development of reinforced concrete in the middle and late 19th century. These concrete structures do not only differ in the structural design from reinforced concrete but often in the used constituents. An often neglected aspect is hereby the use of hydraulic lime and trass, which had a long tradition of use for mortars in the Netherlands. For plain concrete the binder was often a mix of trass and lime, and resulting concrete named in Dutch *trasbeton* (literally trass-concrete) [19]. Other obsolete terms for lime based concrete are *traskalkbeton* (trass-lime concrete) and *kalkbeton* (lime concrete), latter made either only with lime or a lime-trass mix. These concretes were used for foundations, hydraulic, underwater and military structures, as they were durable in marine environments and had good resistance towards shocks and settlement [10, 20, 21]. The latter property made it suitable for military structures, as traditional brick structures could not withstand the impact of new explosive shells.

Plain concrete made with lime and trass was used until the early 20th century for military building, often parallel to Portland cement. In the Dutch military guidelines from 1906, the use of a trass-lime cement was still advised for plain concrete structures [22]. In other countries, such as Germany, its use had decreased by then [23, 24]. It was not only the decreasing price of Portland cement, but also the assumed poorer properties of lime-based concrete. Compared to concrete made with Portland cement, it had a lower strength and did not harden quickly enough in mass concrete as air could not access the concrete [7, 23].

2.3 Aggregates

Early plain concrete structures vary from modern concrete in the type, shape and grading of the coarse aggregates. As the Netherlands did not have many natural resources for natural stone, but a strong tradition in brick production, early plain concrete structures were often made with crushed bricks. In Dutch such concrete was known as *brikkenbeton* and was strongly advised for large plain concrete structures in the late 19th century [5, 7, 8]. Several Dutch military defence lines are known for their use of brikkenbeton such as the Nieuwe Hollandse Waterlinie

(from 1816 onwards), the Stelling van Amsterdam (1880-1920, now a UNESCO World Heritage Site) with the forts near Abcoude (1887), Pampus (1895) and near IJmuiden (1888) [25], the sea defence Hellevoetssluis (17th century, with major extension 19th century) or the fortressed garrison town Naarden (e.g. elements of bastion Katten, 1878, 1895). For military structures the use of debris from torn downed walls or streets was allowed, but not roof tiles, lime rubble, or garbage [22]. The use for civil structures is known as well, such as quay walls in the harbour of Rotterdam in 1874 [26].

The grading of aggregates had not been addressed in the middle of the 19th century, and general tendencies to use relatively large coarse aggregates and no middle fraction can be observed. This reflects the perception of concrete as a mix of a mortar with coarse aggregates, and not as a combination of binder, fine and coarse aggregates, and water as it is done nowadays. The advised size for coarse aggregates was between the size of 'pigeon egg' (ca. 3.5 cm) and a 'chicken egg' (5-6 cm) [5, 7, 8, 27]. Concerning the geometry, angular aggregates such as crushed natural stone or crushed bricks were preferred, the use of gravel possible [7, 8, 17].

3 MATERIAL CHARACTERIZATION

As little is known about the composition of early plain concrete, besides the previously mentioned written historic documents, investigations were carried out on a sample of broken brick concrete dating from the middle of the 19th century from the Defence Line Nieuwe Hollands Waterline (Figure 3.1). The underlying research question was if the investigated concrete reflects the state of the art of the construction time, and to initiate reference data for further investigation of such concretes, as hardly present day reference material for comparison is available. From the historic context the use of hydraulic lime would be most likely, optional with the addition of trass, yet the use of other binders such as natural cements not excluded.

3.1. Materials and Methods

A single core was extracted from the historic structure. For microscopic evaluation, thin sections and polished sections were prepared. During all cutting, sectioning and polishing processes tap water was used as the coolant liquid. Before proceeding with thin and polished section production, small cuts of concrete specimens were impregnated with low-viscosity epoxy mixed with 1 wt% hudson-yellow pigment. Standard petrographic thin sections were produced by PELCON automatic thin sectioning machine. For the transmitted light microscopy a Leica DMEP petrographic microscope equipped with polarization accessories and semi-apochromat (fluorite) objectives were used. Optical photomicrographs of the thin sections were acquired with a Leica DFC310FX digital camera at 1392×1040 uninterpolated resolution and exported as compressed JPG format files.



Figure 3.1 Photographic overview of sample (core size 145mm diameter), showing differently coloured broken bricks as coarse aggregate and, compared to modern Portland cement, whiter matrix. Along the aggregates air pockets are visible

Polished-thin sections were prepared to be used both under optical and electron microscopes in order to increase accuracy of the characterization. Surface polishing protocol was grinding with #320 and #1200 SiC grinding papers and final polishing stage was accomplished by 6µm, 3µm, 1µm and ½µm diamond paste. Finally the specimen surfaces were cleaned by 10s ultrasonic bath in ethanol.

For microanalysis and electron imaging a Philips XL30 environmental electron microscope equipped with EDAX energy dispersive spectrometer (EDS) was used under hi-vac chamber condition. EDS detector was a SUTW (sapphire) type with a calibrated resolution of 133eV at 15kV accelerating voltage. The take-off angle was 35.3°. The polished specimens to be analyzed were carbon coated in a Leica EM CED030 carbon evaporator at a thickness of 10nm, identical to the coating thickness on microanalysis mineral standards used (from ASTIMEX Scientific ltd.). During the quantitative EDS microanalysis an accelerating voltage of 15kV was adopted. A stable beam current of 1 nA was regularly measured by means of a picoammeter and a Faraday cup. Before and after each spot analysis, average beam current was measured and included into the ZAF quantification. The detection limit was assumed to be in the order of 0.2 wt.% or less for individual elements.

3.2. Macroscopic examination

Macroscopic examination of the concrete was carried out on a cross-section with a diameter of approximately 145mm. From the visual inspection, the use of broken bricks as coarse aggregate was easily identified (Figure 3.1). The distribution of brick coarse aggregate was quite uniform. Point counting analysis resulted a volumetric coarse aggregate portion of about 61%. Maximum aggregate size was measured at about 60mm. Coarse aggregate particles exhibited low circularity being generally sub-rounded/sub-angular. Occasional entrapped air (or air pockets) around the coarse aggregates was observed which was possibly due to manual compaction. An overview of the volumetric proportions of the concrete components are given in Table 1, based on macroscopic and microscopic point counting analyses.

Component	Vol. Percentage	Notes		
Paste	21%	<i>Lime-based. Sporadic unhydrated clinker components (i.e. Belite). see features for more.</i>		
Fine Aggregate	15%	Mainly quartz/quartzite sand. Plagioclase, K- feldspar, chert, volcanic and metamorphic sand and altered rock fragments form the rest of the fine aggregate.		
Coarse Aggregate	61%	D_{max} =60mm. Entirely crushed brickstone. Low circularity. Sub-rounded/sub-angular. Evenly distributed.		
Air	3%	<i>Entrapped air, occasionally due to poor compaction. Air content excludes widespread cracks.</i>		

Table1:	Volumetric	proportions of	concrete making	materials and	air

3.3 Microscopic examination

Observation of ghost textures and lime lumps suggest that the binder was a trass-lime, probably with a relatively low trass content. Almost all trass was found to be reacted. Its (former) presence in the mix, however, was evidenced by ghost textures (Figure 3.2) of almost completely consumed volcanic glass and pumice (Figure 3.3), relatively high amounts of volcanic rocks and minerals in the fine aggregate fraction compared to common Dutch (river) sand, and more dense binder matrix surrounding partly consumed trass particles, indicating pozzolanic reaction. Another evidence that the binder is a hydraulic lime was sporadic occurrence of unhydrated belite in the matrix.

Fluorescent microscopy showed that the capillary porosity of the cementitious matrix of the historic concrete is high. In Figure 3.5, the capillary porosity of the historic concrete is compared with a modern concrete. Results showed that the historic concrete exhibits a capillary porosity higher than that of a modern concrete with W/C=0.60



Figure 3.2 A photomicrograph showing ghost textures (both 100x, left cross polarised light. Right plane polarised light)



Figure 3.3 A pumice fragment slightly reacted with the matrix. Plane polarized light. Field of view: [\leftrightarrow 1.43mm].



Figure 3.4. White arrow indicates an unmixed lump of lime. Black arrow indicates a nest of unhydrated belite crystals. Plane polarized light. Field of view: [$\leftrightarrow 0.70$ mm].



Figure 3.5. Comparison between the historic concrete matrix (left) and a modern concrete (W/C=0.60, right) under identical UV-light illumination conditions. Field of view: $[\leftrightarrow 1.43mm]$.

Other than the trass-lime hydration products, the binder features sporadic formations of interesting species. Ettringite deposition is widely available in the cavities around the coarse aggregates, inside air pores and cracks (Figure 3.6). Its presence is known in historic mortars and there is not enough evidence to nominate the ettringite formation as the source of widespread cracking in the matrix. Another minor feature of the matrix was an isotropic formation showing a conchoidal fracture pattern typically resembling an reminiscent ASR gel (Figure 3.7). The chemical composition of the isotropic phase is given and compared to a typical ASR gel in Table 2. Though the observations indicate an ASR gel, its presence is not to be expected in a lime based binder as in general its pH value too low, and possible sources for the silica such as reactive aggregates are questionable. Irregularly distributed charcoal fragments were found in the lime-trass mortar matrix (Figure 3.8). Although origin and purpose of its existence is not clear, one possibility is that it could be a remnant of the lime burning.



Figure 3.6 Typical ettringite depositions in an air pore. Crossed polarized light. Field of view: $[\leftrightarrow 0.70 \text{ mm}]$.

Table 2: Chemical composition (wt%) of the isotropic phase and atypical ASR gel based on quantitative standards-based EDS analyses.

	SiO ₂	CaO	K ₂ O	Na ₂ O
Isotropic phase	38.8	19.5	3.0	1.2
Typical ASR gel [28]	46.1	26.5	5.0	1.3



Figure 3.7 Isotropic glassy phase near a brick-matrix interface. Under the isotropic gel-like formation, a partly birefringent crystallized wood-fragment can be seen. Plane (left image) and crossed (right image) polarized light. Field of view: [\leftrightarrow 2.87mm].



Figure 3.8. Charcoal fragment in the historic binder matrix. Plane polarized light. Field of view: [\leftrightarrow 1.43mm].

4 **DISCUSSION**

The use of lime based binders for historic plain concrete structures has been mentioned in several historic documents. However, little has been published about methods to identify these in samples taken from historic concrete buildings. Their correct identification is not only relevant to confirm historic documents but also to as part of the material investigations during conservation.

The investigated sample did indeed conform historic documents describing the use of lime based binders with trass additions for plain concrete made with broken bricks. Hydraulic lime from Tournai (Doornik) was the most commonly used hydraulic lime in the Netherlands in this period, and is the most likely candidate. For the identification of trass an understanding of the historic and geographical context is beneficial, as most evidence is circumstantial. Trass is made from tuff and in the Netherlands no natural sources which could explain the present fragments with a volcanic origin exist, indicting the use of trass. The fragments were still visible, as historically trass was ground less fine than modern trass.

The used aggregate, crushed brick and the maximum aggregate size of 6cm reflect 19th century practice. An interesting point is the crushed brick that comprises the entire coarse aggregate fraction. Such high amounts of masonry granulate would not be allowed by current concrete codes, yet is historically correct for a concrete dating from the middle of the 19th century. The occurrence of voids around the aggregates can be explained by manual compaction. The possible higher water content is however unusual, as in general plain concrete was supposed to be made with a very low water content and the fresh concrete resemble 'humid earth' [5].

5 <u>CONCLUSION</u>

In this paper, a first study to identify a binder in a historic concrete was presented and shown possible by means of optical microscopy. As historic mix designs differ from modern concrete, sufficient background knowledge of the historic context is beneficial, in order to guide the investigation and to evaluate which properties can be explained by previous construction methods, constituents used and mix design. In this case, a concrete which appears unusual from a modern point of view is, placed in its historic context, normal for its time and building typology. Further research is required to broaden our knowledge on 19th century binders and their application in early concrete structures, and thereby create sufficient reference data for future conservation projects.

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