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# Calcination of Roman cement: A pilot study using cement-stones from Whitby

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#### Abstract

Roman cement was patented in 1796 and established as a major material in the development of European architecture and engineering in the 19th century. However, the market is poorly served for those conservators wishing to use authentic materials in their work and there is a need to reintroduce Roman cement for such purposes.

Cement-stones from Whitby were calcined at various temperatures and residence times in a laboratory kiln. The resulting cements were assessed for setting time, strength development and mineralogical characteristics.

The properties are sensitive to calcination conditions with optimum strengths being achieved at temperatures of approximately 900 °C accompanied by rapid setting of <1 min. A three stage strength development profile was identified characterised by a high initial strength, a dormant period which could last for many weeks and a final increase in strength to an age of 1 year.

Considerable variation in the composition was noted and related to the calcination conditions. Of particular interest is the formation of both  $\alpha'$ -belite and  $\beta$ -belite under differing calcination conditions. Clinker particles were also compared using the SEM in both secondary electron and back-scattered electron imaging modes and the development of morphology observed. © 2007 Elsevier Ltd. All rights reserved.

Keywords: Roman cement; Calcination conditions; Setting; Strength development; Mineralogy

#### 1. Introduction

Although Roman cement was patented in 1796 there is circumstantial evidence that the actual discovery by James Parker was several years earlier [1]. Its name is a classic case of the inventor wishing an association to be drawn between their new material and those of proven quality; Portland cement is another case in point. Roman cement flourished in the UK during the first half of the 19th century until Portland cement became established and the importance of high kiln temperatures for its production recognised. Francis [1] provides a very readable account of the comparative fortunes of these two cements in the 19th century UK market. The continental European market

for Roman cement was more prolonged and it supported major architectural developments such as occurred during the period of European Historicism. The facades of many late 19th century buildings bear witness to its use for render and cast decorative ornament (see Fig. 1 for an example from Krakow).

The original Roman cement was produced using septaria found in the London Clay outcrops on the Isle of Sheppey in the Thames estuary. These septaria typically contained approximately 30% clay, some 65% calcite, and quartz; the septarian cracks were filled with calcite. It should be noted that early chemical analyses did not identify separate sources of silica, i.e. quartz or the clay minerals themselves. The only processing of the raw material prior to calcination was to reduce it to small fragments the size of apples, a task often reserved for child labour [2–4]. Mitchell [4] recognised the need to produce fragments

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Fig. 1. The Trade Academy, Krakow.

of a uniform size to ensure uniform calcination. The calcined stone was ground and packed into barrels.

Other sources of suitable stone were located at sites such as Harwich, where it often occurred as a continuous layer rather than the septaria found in many other locations, and along the Yorkshire coast where the stone was often a by-product of the local alum industry. Indeed, frequent coastal locations discriminates the UK from other major European and American production. That is not to say that all UK sites were located on the coast and The Builder of September 23, 1848 carries an advert for Board's cement, produced at Dunball near Bridgewater, which proudly boasts the merits of a non-coastal source of stone. Other inland sources were identified in clays used for brick making and within cuttings excavated during the expansion of the railway system.

It is apparent that the geology of each location is different and hence the name "Roman cement" is only applicable as a generic description of such natural cements. Additionally, the mineralogy of samples from a single source is variable such that regular quality checks were needed. Many cements were named after the location of the source stone, the owner of the estate, the owner's agent or the region of production. Indeed, the names Mulgrave cement, Atkinson's cement, Yorkshire cement and Whitby cement all refer to cements produced from nodules found in the shales of the Lower Jurassic of the Yorkshire coast. The manufacturer, Francis and Sons produced a cement from a blend of Sheppey and Harwich stones, sometimes referred to as English Cement, a practice which Smeaton [5] suggests was a common practice.

Despite the variability, Roman cements were characterised by a quick set of typically 15 min and a brown colour. The Dictionary of Architecture [6] states that the best cements had an even faster setting time of 6 min. Cements of the most rapid set were generally confined to use in hydraulic engineering works. The typical colour was a light brown although those from Harwich in the UK and Rosendale (Rosendale member) in the USA were much darker. The colour of Harwich cement could be moderated by co-burning with stones from Whitstable and Swalecliffe



Fig. 2. The kiln at Sandsend, Whitby. The Cooper's shed may be seen in the background.

which yielded a much lighter and slower setting cement [2]. However, its strength was not as great as that produced from Sheppey stone alone [3].

Recent interest in this range of cements originates from conservators seeking to implement the ideals of the Charter of Venice of 1964, which states that restoration "is based upon respect for the original material and authentic documents". Pasley [7] was of the opinion that natural cement was more capable of producing an excellent stucco than even the strongest hydraulic limes of the day. The market is poorly supplied with natural cements with only Vicat's Prompt and Edison Coating's Rosendale cement being available in Europe and the USA, respectively. The reintroduction of the latter product in 2004 indicates the growing interest in this group of materials.

The work reported here is of a pilot programme conducted in preparation for a submission for the award of a European Framework 5 contract. Whitby was chosen as the site for study given its proximity to the University and the historic production on the Mulgrave Estate (Fig. 2).

#### 2. Experimental

#### 2.1. Source material

The geological formations of the Lower Jurassic were laid down in marine conditions. In the Whitby area the junction between the Middle and Lower Jurassic is marked by the Dogger of the Lower Bajocian (sideritic sandstone), immediately below which is the Upper Lias of some 205–212 feet (62–64 m) thickness [8]. The uppermost series of the Upper Lias is that of the Alum Shales (zone – Ammonite Hildoceros bifrons) which was the basis of the former extensive alum industry. Concretions frequently occur in beds throughout the sequence of the Yorkshire Lias and often reflect the composition of the surrounding shale yielding essentially calcitic or sideritic nodules [9]; some contain significant amounts of pyrites and these were rejected for

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