Analysing modern concretes

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Concrete is not what it used to be. The days of just turning to British Standards when analysing a concrete sample are long over. Why? In construction, ready-mixed concrete dominates and advances in concrete technology, with modern cements, additions and admixtures, now make the standard procedures that are available almost impossible to apply without modification or extension.

The slipforming of the core of building DS1, Canary Wharf – reported in *CONCRETE*, October 2000 – usefully demonstrated how mix design can be vital for such operations. In this case, requirements relative to both location and time of day resulted in ten mix designs with varying proportions of retarder, superplasticiser and pfa. But how can such concrete be checked for compliance, particularly if there is a subsequent dispute or if something went wrong? Later in the life of the building, a condition assessment might be requested but this may be difficult to undertake correctly without knowing or being able to determine all the facts.

With the right approach and careful selection of techniques and an experienced interpretation of the findings the situation can be redeemed. The approach starts with distinguishing the possible extra ingredients into organic or inorganic, as these require quite different analysis techniques. Organic components are principally used to plasticise, retard and air-entrain concrete but may also be added at different stages of placing to help bonding or surface applications for curing and waterproofing. Inorganic components are generally additions such as pfa and ggbs, and now microsilica and metakaolin. Many proprietary mixes further complicate the situation, not relying on just OPC or SRPC, but commonly including lime, HAC, white cements or sometimes calcium sulfoaluminate (CSA). Also, modern 'eurocements' include factory-blended materials and a variety of fillers.

The current approach

Concrete investigation mostly involves tests and procedures covered by BS 1881: Part 124 *Testing concrete: Methods for analysis of hardened concrete*, which includes chemical methods for determining the silica and calcium contents so that the approximate cement content and apparent cement type can be estimated. The standard is fraught with problems even for relatively 'standard' concrete; calculations are based

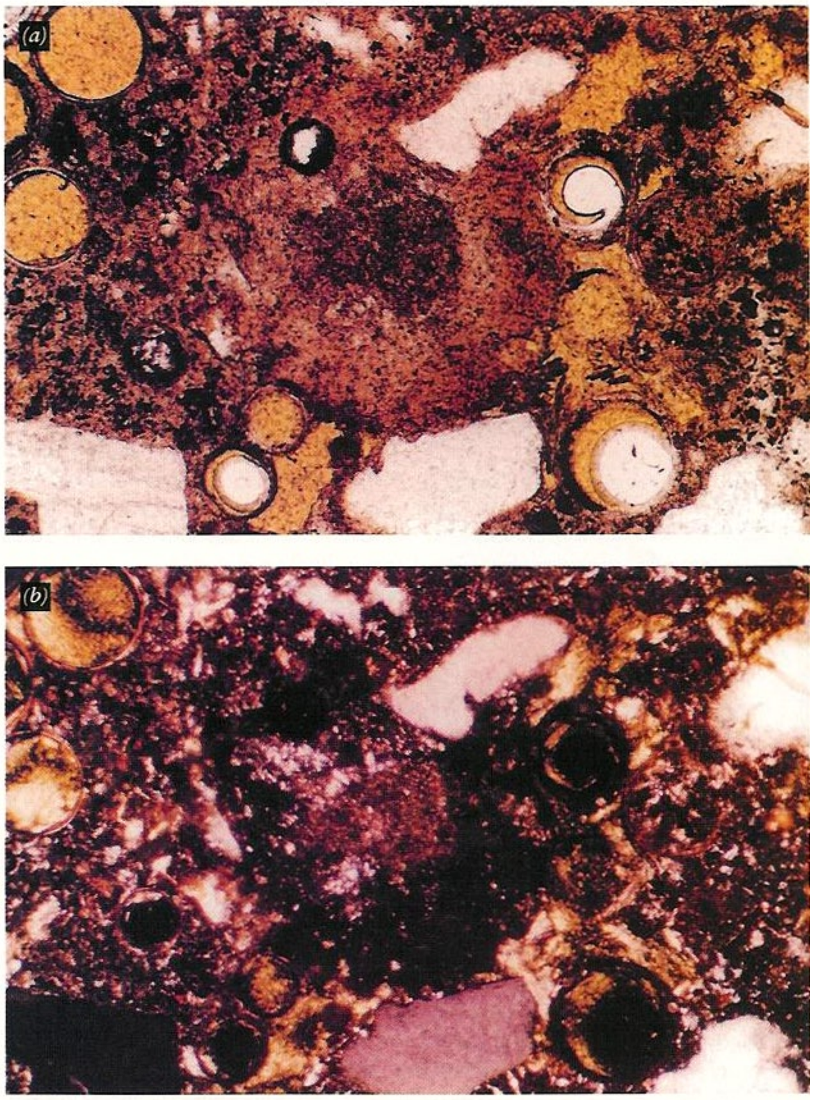

Figure 1: Photomicrograph showing calcium sulfoaluminate addition in a concrete matrix. (a) Plane polarised light (b) Cross-polarised light. (Width of each image 0.5mm.)

upon assumptions, the aggregate may exert a variety of influences upon the findings or the exposure conditions may have led to changes in the concrete bulk chemistry. Sampling procedures can also affect the results.

One small part of the standard includes a more definitive determination of the cement type by employing reflected light microscopical examination of highly polished specimens that have been etched to highlight the different cement phases. Strictly, the BS 1881 method only distinguishes between OPC and SRPC type cements, although experienced application of the reflected light techniques may yield a greater range of information. Modern cements are often very finely ground and finding representative residual cement particles can be difficult.

Other parts of BS 1881 include a wide range of tests to determine physical characteristics that help to give clues to the original make-up of concrete. Although tests for strength may be applicable whatever the constituents, tests such as those to assess the original water/cement ratio are pointless if the composition includes anything other than natural aggregate and Portland cement.

What else can be done?

To look forward, it is often worthwhile to first look back. Historic materials rarely fall into the neat and tidy assumptions behind the British Standard methods, and the best approach in their investigation has been a combination of petrography* with chemical methods. Petrography is the best starting point, as it can usually identify the constituents and alert the investigator to a variety of potential or existing problems (Figure 1). Most types or blends of cement can be resolved along with pfa or ggbs (Figure 2).

Ultrafine additions, such as microsilica or metakaolin, are more difficult to detect by optical microscopy alone. They tend to densify the cement matrix and deplete or consume the mineral portlandite. The presence of microsilica is sometimes betrayed by coarse agglomerations. Petrography will not resolve admixtures but only their results such as entrained air or plastic settlement cracks. The slides for petrography may also be used for scanning electron micro-analysis to obtain additional constituent data and check for the few components that may not be resolved optically.

Petrography can also be used to determine both the cement content and aggregate grading by undertaking point counts on polished slices, thin-sections and highly polished specimens. One big advantage is that the methods may be effective on all concretes, irrespective of aggregate composition, and those containing additions – an impossibility with BS 1881 chemical procedures.

If organic matter is suspected, a few qualitative tests may be run to provide initial confirmation before undertaking more sophisticated analyses. The presence of a waterproofing agent may be ascertained by the simple bead test†, whereas the presence of a possible plasticiser might be suggested by assessing the loss on ignition.

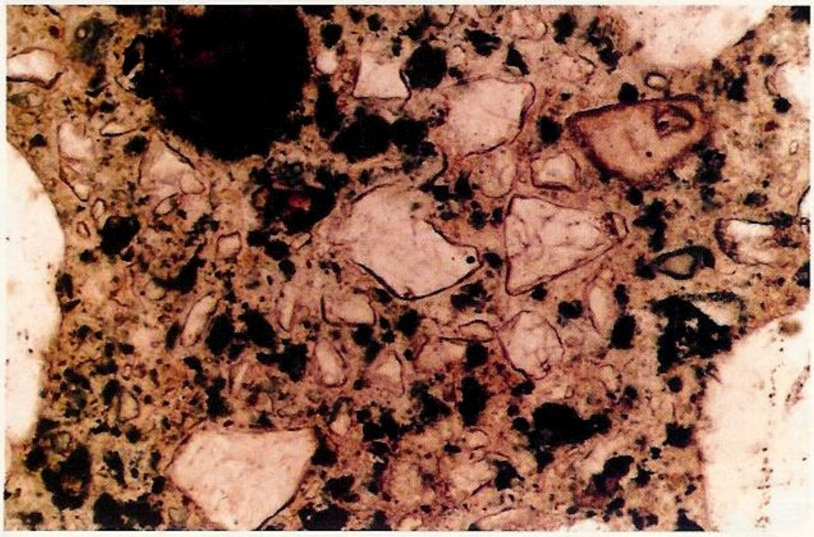

Figure 2: Photomicrograph of cement matrix showing how obvious angular granules of ggbs may be to a petrographer. (Width of image 0.5mm.)

A good summary of the current approach to the investigation of concrete is given in Concrete Society Technical Report 32 *Analysis of hardened concrete*.

Narrowing the field

Organic admixtures are extremely complex. Identifying and quantifying them is made very difficult by their low dosage levels, their use in combination, and the chemical reactions that take place between the admixture and the cementitious matrix. Detailed knowledge of the chemical composition and experience of a wide range of analytical techniques enable the chemist to select the most appropriate method of analysis. Inadequate or a total lack of background information on the admixture under investigation leads to tedious and time-consuming analysis. Inexperience of these materials could lead to misinterpretation of the test data and might result in serious consequences for the client.

Infrared spectrophotometry is the most appropriate method of analysis for identifying an organic component (Figure 3 and 4). Provided the correct solvent is used to extract the desired organic species from the sample, this technique can rapidly determine the generic admixture and provide clues to the next stage of analysis. Pyrolysis-gas chromatography, gas-liquid chromatography, ultraviolet spectrophotometry and traditional wet chemical methods could all be used as complementary or standalone analytical techniques in isolating a specific admixture. Again, petrographic examination may help, as certain features such as plastic settlement may imply either incorrect mixing or an excess of admixture.

The advantage of these methods is that they can detect low levels of organic admixtures and, providing reference materials are available, can help quantify some of the more common admixtures used in the industry. Another reason for using

* Petrography: the systematic description of textures and mineralogy using both macroscopic and microscopic procedures. The term petrography strictly applies to rocks but the procedures are relevant for any mineral-based man-made material: hence 'concrete petrography'.

† Bead test: a drop of water is placed on the surface of the concrete and the meniscus angle and the time taken for the water to be absorbed are noted.

reference materials is to reproduce the sample matrix and reduce any analytical uncertainties.

Where petrography is unable to help answer all the questions about the mineral components, x-ray diffraction is an effective method for identifying mineral species and is most useful for positively identifying reaction products and unusual additions. Thaumasite and other sulfate mineral species – phases that would analyse as indistinguishable sulfates by normal chemical analysis – are readily identifiable by x-ray diffraction.

Another major problem with chemical analysis is that it relies upon crushing large volumes of sample, often quite indiscriminately, and may not identify important concentrations of minerals such as reaction products. A 5% bulk sulfate content by mass of cement may appear normal but could be very significant if concentrated in the outer surface zone, for instance. Petrography would identify the concentration, scanning electron microscopy would be used to analyse the local concentration and x-ray diffraction to confirm the mineral species.

The future

It is unlikely that the British Standard analysis methods will ever be able to keep up with developments in concrete technology, especially now that European normalisation has further slowed the development and updating of standards.

The 'art' of investigating both historic and modern concrete involves the selection of the most appropriate combination of complementary techniques given the available information. The right answers will not be achievable without specialist knowledge and techniques.

Sampling is also a progressively difficult procedure. Many investigation techniques look in great detail at small areas and, without care, important general information can easily be missed. Sadly, with the way most sampling is carried out by non-specialists, it would be difficult to consider many concrete samples received for investigation to be either sufficiently representative or uncontaminated.

Many concrete investigations simply require site coring teams to take a number of samples with little consideration for the structural context or the implications of any analyses that may be required. Getting a concrete materials specialist on to site to guide such investigations remains the exception: often the cost is misguidedly seen to be unnecessary. Investigations may fail to maximise their potential and early misdiagnosis can lead to greater future costs.

The challenges set by modern concretes can be met, but doing so requires a change in the way investigations are conducted both on site and in the specialist laboratory, and this may be the hardest challenge of all.

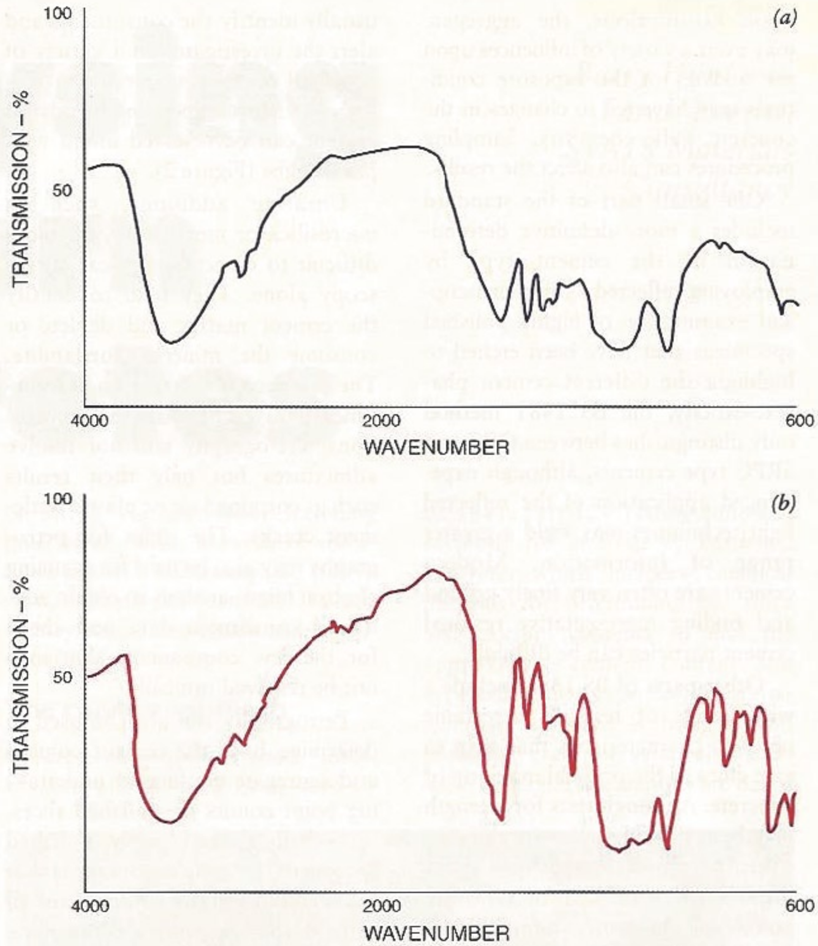

Figure 3: Typical spectra from infrared spectrophotometry analysis of admixtures formerly contained within a concrete sample. (a) Lignosulfonate (used as a plasticiser) (b) naphthalene sulfonate formaldehyde (used as a superplasticiser).

Figure 4: Infrared spectrophotometry in progress.