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DETERMINATION OF THE W/C RATIO OF HARDENED CEMENT PASTE AND CONCRETE SAMPLES ON THIN SECTIONS USING AUTOMATED IMAGE ANALYSIS TECHNIQUES

J. Elsen [a], N. Lens [a], T. Aarre [b], D. Quenard [c], V. Smolej [d]
[a] WTCB, rue de la Violette 21 - 23, B - 1000 BRUSSELS
[b] DBT, Helleruplund Allé 21, DK - 2900 HELLERUP
[c] CSTB, rue Joseph Fourier 24, F - 38400 SAINT-MARTIN-D'HERES
[d] KONTRON Elektronik GmbH, Oskar-von-Miller Strasse 1, D- 85385 ECHING

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ABSTRACT

This article presents results of the w/c ratio determination of hardened cement paste and concrete samples on thin sections using automated image analysis techniques.
The determination of the w/c ratio of hardened concrete is of prime importance for deterioration diagnosis as well as for concrete Quality Assurance in general. An optical method to determine the w/c ratio of hardened concrete using thin sections has been developed in Denmark and is now in general use in Denmark and the other Scandinavian countries. Two series of samples prepared in laboratory conditions have been used as 'reference samples' to develop and to evaluate procedures for measuring the w/c ratio by image analysis techniques : a series of pure cement paste samples and one of concrete samples. Three different image analysis methods have been employed : an interactive, an automatic method with thresholding of grey values by the operator, and a fully automatic method without thresholding. The second method was retained for further measurements. The measurements on the pure cement paste samples confirm the direct relationship between the w/c ratio of the samples and the mean grey value measured on thin sections.
The results of the measurements of the thin sections prepared out of the reference concrete series also indicate a clear relationship between the w/c ratio and the mean grey value but these measurements are less reproducible. The main cause of this variability with the concrete samples is the difficulty in selecting threshold level values.

Introduction

The w/c ratio is one of the most important parameters in concrete technology not only because of its direct relation with concrete strength but also because of its importance with regard to the durability of the concrete. This is due to the direct relationship between the w/c ratio and the capillary porosity of the cement paste. A higher capillary porosity results in a higher permeability towards aggressive agents such as noxious liquids and gases. Therefore, the determination of the w/c ratio of hardened concrete is of prime importance for deterioration diagnosis as well as for concrete Quality Assurance in general.

An optical method to determine the w/c ratio of hardened concrete using thin sections has been developed in Denmark (1) and is used nowadays generally in Denmark and the Scandinavian countries.

This method is based on the principle that the intensity of the fluorescence of the epoxy in the cement paste is proportional to the capillary porosity. The higher the fluorescence intensity is, the higher the capillary porosity and the w/c ratio are. The fluorescence intensity of thin sections prepared from concrete samples with an unknown w/c ratio are visually compared with a reference series of thin sections of a concrete with a known w/c ratio, a well-defined cement, and a curing under standard conditions for 28 days.

The first results of quantitative measurements of w/c ratio were published by Mayfield (2) on polished specimen using a photo-diode. Quantitative results using image analysis techniques were published by Wirgot and Van Cauwelaert (3). They concluded that the fluorescence of impregnated cement paste can be measured with image analysis techniques coupled with a statistical analysis of the data. The precision was estimated as being insufficient.

The objective of this research is the development of an automated image analysis procedure to determine the w/c ratio optically from fluorescent dye impregnated thin sections of concrete.

Sample preparation

- Description of samples

Two series of samples prepared in laboratory conditions have been used as 'reference samples' to develop and to evaluate the procedures to measure the w/c ratio by image analysis techniques.

Series 1: Pure cement paste samples (CP) with OPC-cement (Belgian - P40).

Code	w/c ratio
CP1	0.25
CP2	0.33
CP3	0.50
CP4	0.60

Series 2: Concrete samples (C) with the following specifications.
(see table 1 for detailed mix design)

Type of cement :	OPC-cement (Denmark - Low-alkali cement)
Type of sand :	Quartz Sand
Type of coarse aggregates :	Rønne granite.
Admixtures :	Air entrainer (Sika-air)
	Superplasticizer (Melment L10)

Code	w/c ratio
C1	0.35
C2	0.40
C3	0.45
C4	0.50
C5	0.55
C6	0.60
C7	0.70

- Thin section preparation

The careful preparation of fluorescent dye impregnated thin sections is of utmost importance to assure a

Table 1. Mix design

Concrete sample	Mass (kg/m³) saturated and surface-dry conditions									
	Cement	Sand 0/0.3	Sand 0.3/0.6	Sand 0.6/2	Coarse agg. 2/8	Coarse agg. 8/16	Air (Sika Air)	SPT (Melment L 10)	Water	Total
C1	400	157	157	313	400	800	0.20	4	137.6	2369
C2	360	157	157	313	400	800	0.18	2.5	142.35	2332
C3	325	169	169	337	400	800	0.17	1.5	145.35	2347
C4	298	169	169	337	400	800	0.25	-	149	2322
C5	276	174	174	346	400	800	0.13	-	151.8	2322
C6	258	174	174	346	400	800	0.13	-	154.8	2307
C7	230	180	180	361	400	800	0.10	-	161	2312

reliable and reproducible w/c ratio determination. The w/c ratio is determined traditionally by comparison with standard reference thin sections of concrete samples. This implies that the thin sections being examined always have to be prepared following the same preparation procedure as applied for the reference thin sections (4).

Image Analysis Procedure

- Introduction

Three different image analysis methods have been evaluated : an interactive method, an automatic method with thresholding of grey values by the operator, and a fully automatic method without thresholding.

Interactive method

On the digitized image, the areas in the cement paste to be evaluated are interactively defined by the operator. The distribution of grey values of each selected area is measured and the mean grey value of each area is considered as a measure for the fluorescence intensity of that area. The measured w/c ratio is calculated as the mean of the weighted mean grey values of the selected areas. The main disadvantages of this method are that the relevant areas have to be defined interactively by the operator for each image making this procedure time consuming in regard to man-hours. A second disadvantage is that this method is not highly reproducible. The areas examined are interactively defined and thus influenced by the subjectivity of the operator. If for example, very homogeneous areas are always chosen by the operator, then the results will not be truly representative for the sample.

However, this method is very useful for measuring the w/c ratio of a concrete for which it is very difficult to identify the cement paste automatically. This is the case for example for a concrete with an extremely high air content, or with a very high content of porous aggregates.

Fully automatic method

In the fully automatic method, a thin section is scanned and the complete grey level histogram of each image is accumulated and stored (see figure 1).

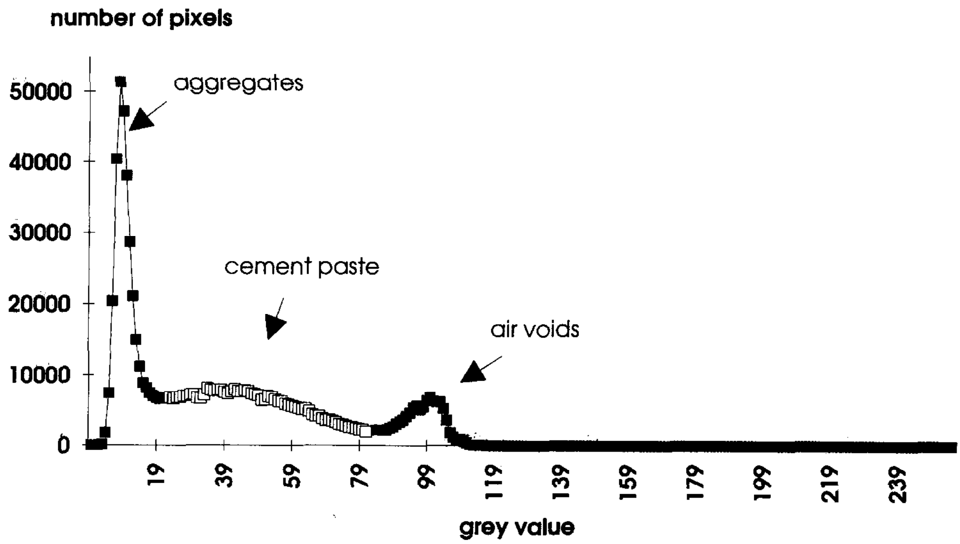

Figure 1. Complete grey level distribution of a thin section prepared from a concrete reference sample

The complete grey level distribution exhibits the following features:

- a considerable portion of the concrete samples consists of opaque material which results in a strong peak at low grey-values.
- at the high grey levels there is a peak due to porosity.
- a broad distribution of grey levels in between is due to the cement paste fluorescence.

However, the strong peak at low grey-values is not caused by the aggregates only. It can be very easily determined by visual observation using the microscope that a portion of the unhydrated cement particles are as dark as the aggregate grains. This implies that it is not possible to measure a thin section of concrete automatically with the complete grey level distribution and to pick out afterwards the cement paste grey level distribution very accurately.

The main conclusion of the measurements with this method is that it is not possible to select the cement paste area with the fully automatic method without loosing the black unhydrated cement particles.

Automatic method with thresholding

In an automatic measurement with thresholding a series of parameters are defined at the start of the measurement by the operator. The grey level thresholds for the selection of the cement paste are selected by the operator and if necessary, all images are rapidly scanned to skip eventually all images which could give wrong results due to preparation errors, porous aggregates etc. After these initial operations, the measurements are performed automatically.

- **Equipment used and measuring conditions for the automatic method with thresholding**

The following equipment has been used to measure the w/c ratio on thin sections with fluorescence microscopy.

Optical research microscope (ZEISS Axioplan).
Scanning stage (MARZHAUSER).
Microscope control processor (MCP-KONTRON Elektronik).
Vidicon type tube camera with a resolution of 1600 TV lines (MTI precision 81).
Image analysis system (IBAS system; KONTRON Elektronik).

Measurement conditions :

Epi-fluorescence with EPI-plan objective :	10X
Optovar :	1.25
Filter set :	BP 450-490 / LP515
Camera gain :	Stored Manual Mode
Scale factor :	1 pixel = 1.20 x 1.24 micron
Area of one image :	614.4 x 634.9 micron
Number of Images measured :	15 x 12 = 180

A grid of 1080 images is defined over the thin section, 45 in the X-direction and 24 in the Y-direction. Every third image in the X-direction and every second image in the Y-direction is considered in a systematic sampling plan, i.e. a total of 180 images are considered.
The total area considered is thus 614.4 micron x 634.9 micron x 15 x 12 or 9.22 x 7.62 mm^2.

The following initializations are performed whereby a number of parameters are selected by the operator before the actual measurement loop :

- The intensity of the illumination of the microscope is calibrated on a reference thin section prepared from a block of pure fluorescent epoxy. The illumination of the microscope lamp is always kept at its maximum intensity and the adjustment to a fixed mean grey value of the reference thin section is performed using an optical wedge-shaped neutral density filter.
- The threshold grey levels for the selection of the cement paste area are selected and checked on several images.
- All images can rapidly be scanned and the operator can skip those images which would give obviously wrong results. Wrong results can be obtained due to porous aggregates and to preparation defects.
- A shading correction can be applied. The scrap parameter, the area considered and the area measured can interactively be chosen by the operator.

Each image is transformed during the automatic measurement in the following steps :

1) The incoming video signal is integrated in four steps (sequence of TV cycles).
   This is used to improve the signal/noise ratio of an image.
2) Very small objects are eliminated on the basis of their area, in pixel units by a scrap command.
   A second scrap command is used to include the unhydrated cement particles in the phase which will be measured. These particles cannot be selected by their grey values.
3) All isolated particles (holes) inside the aggregate grains are filled.
4) Three erosions are being applied to the image with an octagon structuring element. This is used to

correct for the halo around the air voids, and to correct the rim around aggregates more precisely in thin sections with a low w/c ratio.

Results

Cement paste samples

The measurement on cement paste samples is much more straightforward because of the absence of any aggregates. Only an upper-limit for the grey values has to be selected by the operator, in order to avoid air voids. The lower limit for the grey values has thus always been set to zero for the measurements shown in table 2 and figure 2.

Table 2

sample code	w/c ratio	mean grey level operator 1 - operator 2	sample standard error (operator 1)	number of images measured
CP1	0.25	14.3 - 13.1	0.20	54
CP2	0.33	21.0 - 20.6	0.41	50
CP3	0.50	32.3 - 32.8	0.41	50
CP4	0.60	37.3 - 35.4	0.42	50

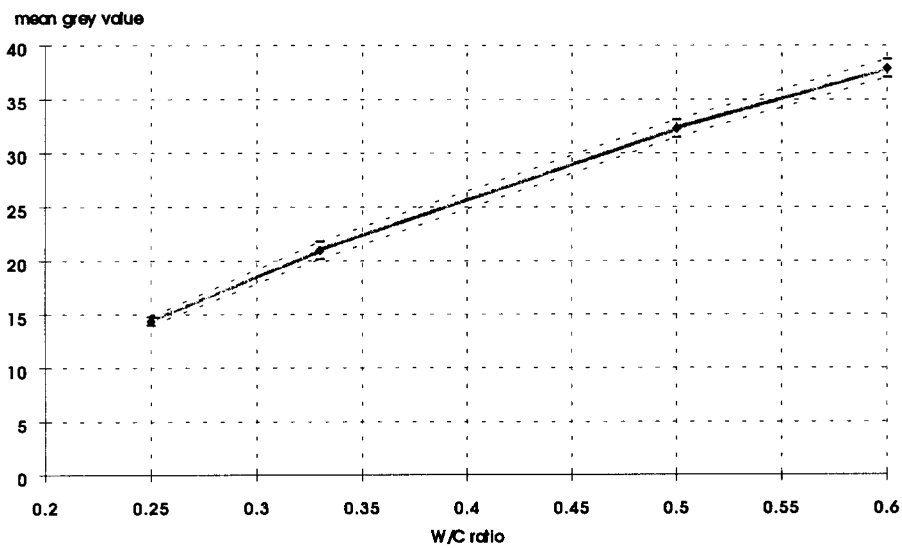

Figure 2. Mean grey value and its 95% confidence interval for the four pure cement paste samples as measured with automated image analysis techniques (operator 1).

- **Concrete Reference Series**

The results of the measurements for the concrete samples are shown in table 3 and figure 3.

Evaluation of the method

The measurements on the pure cement paste samples confirm the direct relationship between the w/c ratio of the samples and the mean grey value measured on thin sections.

Table 3

sample code	w/c ratio	mean grey level	sample standard error	number of images measured
Cb1	0.35	15.6	0.19	133
Cb2	0.40	21.5	0.38	87
Cb3	0.45	29.3	0.52	81
Cb4	0.50	32.9	0.40	96
Cb5	0.55	36.3	0.37	108
Cb6	0.60	41.6	0.55	97
Cb7	0.70	48.7	0.83	85

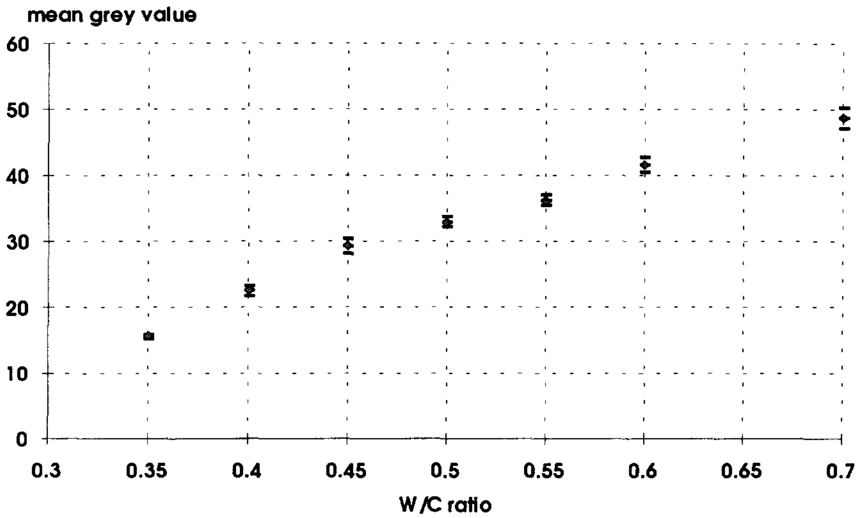

Figure 3. Mean grey value and its 95% confidence interval for the thin sections of the seven concrete reference samples as measured with automated image analysis techniques.

The results of the measurements on the thin sections prepared out of the reference concrete series also indicate a clear relationship between the w/c ratio and the mean grey value but these measurements are less reproducible. The main reason for this with concrete samples is the difficulty in selecting the threshold level values. The influence of the selection of the threshold levels on the measured grey value has been evaluated for three samples (C2, C4 and C6). The same area was evaluated consecutively with different threshold levels. The results for sample C6 are illustrated on figure 4 for the evaluation of the influence of the lower level on the mean grey value. Only the selection of 11 as the lower level can clearly be evaluated as being wrong.

The differences determined for the mean grey value are rather small for different upper threshold limit values. However, the differences for the mean grey value are quite big for different lower threshold limit values, especially for lower w/c ratios.

Conclusions and further research

The main difficulty of measuring the fluorescence intensity of the cement paste phase in concrete samples automatically, is that we need to include the unhydrated cement particles within this phase.

If we look at the complete grey level distribution of a concrete thin section fluorescence intensity

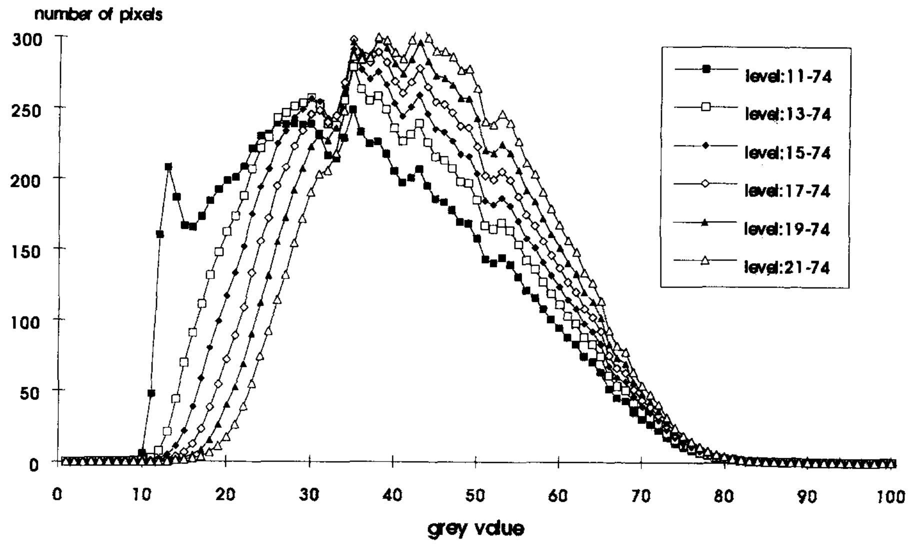

Figure 4. The number of pixels measured (normalised) is illustrated for each grey value and this for different lower level selections for thresholding (sample C6).

measurement we observe three main peaks (see figure 1). These three peaks correspond roughly to the three following phases: aggregates, cement paste and air voids. But not all the grey levels of the unhydrated cement particles which are part of the cement paste are within the peak corresponding to the cement paste. This implies that it is not possible to measure a thin section automatically with the complete grey level distribution and to pick out afterwards the cement phase grey level distribution. One solution is to select the phases interactively by the operator at the start of the measurement. The unhydrated particles are then included in the cement phase for each image separately by image analysis processing techniques. The grey level distribution of the complete cement paste phase is then measured automatically.

All the measurements here have been performed for cement paste and concrete samples which have been cured under laboratory conditions for 28 days. The results cannot of course be transferred in a simple way to field concrete.

More research is needed to assess the reproducibility of this method and the variability between different laboratories.

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