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147 Prosjektrapport 1994

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ISBN 82–536–0444–0
ISSN 0801–6461
100 eks. trykt av
S. E. Thoresen as
Cyclus resirkulert papir,
omslag 200 g, innmat 100 g

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Content

Summary

A round robin test has been done to evaluate the applicability and fasibility of two different thin section techniques when applied to high strength concrete. Five Nordic laboratories participated in the test. The tests were performed on laboratory produced concrete with 10 % micro silica fume and water cement ratios 0.20, 0.25, 0.30 and 0.40. Each of the laboratories made thin sections that were circulated between the laboratories for evaluation of water cement ratio.

Traditional thin section preparation is based on. impregnation with a fluorescent epoxy. The fluorescent epoxy makes it possible to determine the water cement ratio of the concrete using microscope and ultra violet light. The epoxies normally used show very poor penetration capability in concrete. This is due to high viscosity of the epoxy. The two thin section techniques used in this work are based on a low viscous epoxy and a recently developped technique called FLR.

The impregnation capability of the low viscous epoxy turned out to be less than satisfactory. Heating of the epoxy to decrease viscosity had no significant effect. FLR technique showed impregnation depths of several millimetres in some specimens. The evaluation showed satisfactory correspondence between real and observed water cement ratio. Comparison of thin sections from different laboratories showed good reproducibility as regards water cement ratio.

Determination of water cement ratio in high strength concrete by thin section technique

1. *Introduction*

Thin section technique has been used commercially for structural analysis since the early seventies. The technique is based on microscopical examination of a very thin segment (approx. 25 μm) of concrete mounted on a thin (thickness approx. 1 mm) plate of glass. The size of the segment is usually about 30 mm x 50 mm.The thin section technique is described in detail, see reference [1], the reader is therefore referred to these publications for basic information on the subject.

One of the important parameters found utilizing the technique is the ratio between water and cement in the concrete mix. Determination of the water cement ratio is done by vacuum impregnation of the concrete with a fluorescent epoxy. The epoxy enters the capillary pore structure of the cement paste and may be observed as a greenish glow through application of ultra violet light and proper filtres. Measurement of the w/c-ratio is possible as a consequence of the proportionality between capillary porosity and the w/c-ratio. Porous cement pastes of high w/c-ratios are recognized by an intense glow whereas denser pastes of lower w/c-ratio turn out as darker. In addition to w/c-ratio the presence of fluorescense also adds information about air content,cracking and paste homogeneity.

With the advent and increased application in the later years of high strength concretes in various fields, the need for a modification of the technique to cover lower water cement ratios has become more and more prominent. Reliable w/c-ratio determination is usually limited downwards to a w/c-ratio of 0.35. With addition of micro silica fume to the concrete even a w/c of 0.40 – 0.45 may cause problems.This limitation is caused by the macromolecular character and consequently high viscosity of the resin and in some cases also the hardener.

To circumvent this problem and increase penetration capability, experiments have been done with epoxy systems of considerably lower viscosities [2]. These experiments have only been partly successful, however, mainly due to health hazards caused by the comparatively high volatility of these epoxy systems.

However, a technique based on liquid replacement of capillary porewater with a solution of ethanole and a fluorescent solute has been more successful. The technique called FLR (Fluorescent Liquid Replacement) [3] has been developed at the Norwegian Building Research Institute as a result of continuous work in this field for several years. This technique has increased

the range of w/c ratio determination down to 0.20 – 0.25, comprising concrete containing micro silica fume.
The last two techniques, and then mainly the latter, form the technical background for this work.
The main purpose of the work has been to evaluate the applicability and feasibility of these techniques in different laboratories. In turn, possibly ending up with a proposal of one of the techniques as a Nordtest method.
To accomplish this, four different Nordic laboratories in addition to NBI were invited to participate in a round robin test with NBI being responsible for project management including the final report.The four participating laboratories were the Danish Technological Institute (TI), Denmark, Technical Research Center of Finland (VTT), Finland, Islandic Building Research Institute (Rb), Island, Swedish National Testing Institute (SP), Sweden.

2. *The samples*

The high strength concrete used in this project was cast in the laboratories of NBI. The production covered mixes with four different w/c ratios, 0.20, 0.25, 0.30 and 0.40, all containing 10 % of micro silica fume by weight of cement. Six 15 cm cubes and three 10 cm cubes were cast from each mix. This giving a total of twentyfour 15 cm cubes to be used for thin section preparation and twelve 10 cm cubes earmarked for compressive strength testing.
Norwegian Ordinary Portland Cement with the designation P 30 was used.
The aggregate was supplied by the Norwegian company Aker singel og grus – Norsk sand. It is produced according to requirements set in Norway.
Maximum diameter d_{max}, of the aggregate was 16 mm.
The samples were stored in water from the second day of curing after demoulding. The compressive strength was tested after 28 days of curing.
Measured compressive strengths are given in table 1.

Table 1
Compressive strength measured after curing in water for 28 days.

w/c-ratio	Compressive strength (MPa)
0.20	100.6
0.25	119.5
0.30	107.4
0.40	110.7

The upper and lower part (approx. 20 mm) of the 15 mm cubes were cut away. The remaining concrete was packed in a layer of wet paper and thick plastic foil and shipped to each of the participating laboratories in wooden boxes.

An illustration of how the cubes were cut and where the thin section were taken is shown below in fig. 1.

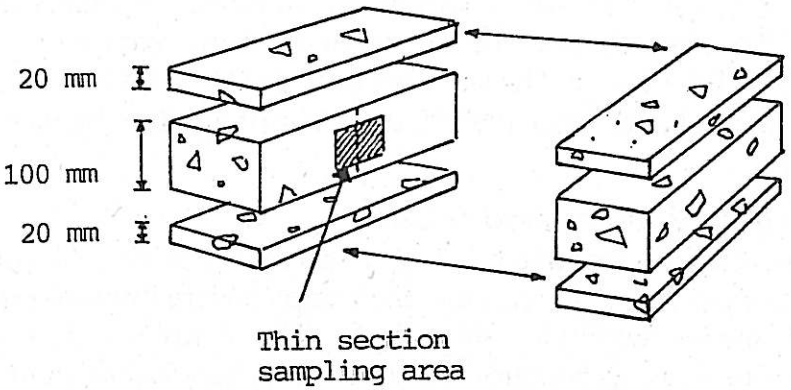

Fig. 1
Exploded view showing sampling area of thin sections from 15 cm concrete cubes.

3. *Experiments*

Preparation of thin sections followed traditional procedure, differing only in the impregnation techniques. These were, as mentioned above, impregnation with low-viscosity fluorescent epoxy and fluorescent ethanol solution. Each laboratory were to produce one thin section from each w/c-ratio using the two impregnation techniques, giving a total of 8 sections produced in each laboratory. As various problems raised partly due to inexperience with the new techniques, each of the laboratories ended up with at least doubling the number before acceptable preparation quality was achieved.
Equipment and facilities for production of thin sections varied between the laboratories. These differences which were purely of technical nature (different types of evacuation and grinding equipment and microscopes). The differences did not influence on the final quality of the thin sections.
A fluorescent dye of type Hudson Yellow, Struers Epo Dye or similar was used.Each laboratory was allowed to use the dye they normally use. The dye absorbs ultraviolet light at a frequency at 515 nm and emits at 535 nm.

After preparation all thin sections were gathered together and circulated between the laboratories for water cement ratio evaluation.
Common to both impregnation techniques is that no reference material exists. Measurement of w/c-ratio is therefore limited to a relative ordering of the thin sections according to brightness and the four known water cement ratios. Each laboratory was instructed only to label the thin sections with codes. The

corresponding water cement ratio was not revealed until the round robin test was terminated.

Low viscosity epoxy

An epoxy system from Nils Malmgrän AB with designations Epoxi INP 003 (resin) and NM 24S (hardener) was selected for this part of the work. It has the quality of a fairly low viscosity in addition to show an acceptably low volatility to avoid health hazards. Concentration of fluorescent dye was 1.0 percent by weight of the epoxy. The samples were polished on the side that were to be investigated and dried at 35 °C before impregnation by vacuum.

Fluorescent Liquid Replacement (FLR)

A 1.0 % solution of fluorescent dye in ethanol was mixed prior to starting the experiments. After polishing on one side, the samples were immersed directly into the ethanol solution without prvious drying. The samples were kept in the solution for four days. Experience shows that this time is sufficient for adequate replacement of the porewater by the fluorescent ethanol solution. After drying in air, the fluorescent dye was fixed by application of a thin layer of normal Ciba Geigy fluorescent epoxy (resin: BY 258 or similar; hardener: MY 2996). Excess epoxy on the surface was removed by very light grinding before the sample was glued to a glass.
The preparation from then on follows the traditional preparation procedure.

4. *Results*

Presentation of the results is devided into two sections, one briefly summarize the tests on low viscosity epoxy and the other reporting the results from Fluorescent Liquid Replacement.

Low viscosity epoxy

The impregnation capability of the low viscous epoxy system turned out to be less than satisfactory. The impregnation depth was checked by sawing off the impregnated specimens and inspecting the sawn surfaces in microcope using ultraviolet light. No significant penetration of the fluorescent epoxy could be observed. Heating the epoxy to 50 °C before impregnation gave no improvement. Further work on low viscous epoxy was therefore cancelled.

Fluorescent Liquid Replacement

Similar inspection of specimens impregnated with ethanol solution revealed penetration depths from a few microns in the low w/c-ratio specimens to several millimetres observed in those with the highest w/c-ratio. A survey where the participating laboratories have ranged the thin sections according to fluorescent brightness in the microscope is presented in tables 2 to 6.. Each table shows the observations made in each of the participating laboratories. The presentation is arranged to show the real w/c-ratios in the left column.

Measured values corresponding to the different thin sections along with the laboratory where they were made are shown in the other columns. The different laboratories are marked by the capital letters A to E. Laboratory E prepared two sets of thin sections marked 1 and 2 in the tables.

Table 2
W/c-ratios measured in laboratory A

Real w/c	Preparing labs. (A - E) and measured w/c-ratios					
	A	B	C	D*	E	
					1	2
0.20	0.20	0.20	0.30	-	0.20	0.20
0.25	0.25	0.25	0.25	-	0.25	0.25
0.30	0.30	0.30	0.20	-	0.30	0.30
0.40	0.40	0.40	0.40	-	0.40	0.40

*) Laboratory A was the first to measure w/c-ratios. Problems with the solubility of fluorescent dye that were experienced in lab D were not solved at that time.

Table 3
W/c-ratios measured in laboratory B

Real w/c	Preparing lab. and measured w/c ratios					
	A	B	C	D	E	
					1	2
0.20	0.20	0.20_d	0.20_e	0.20_d	0.20_e	0.20_e
0.25	0.25	0.25_d	0.25_d	0.25_d	0.25_d	0.25_e
0.30	0.30	0.30_e	0.30_d	0.30_e	0.30_d	0.30_e
0.40	0.40	0.40_e	0.40_e	0.40_e	0.40_e	0.40_e

$_d$ difficult to range
$_e$ easy to range

Table 4
W/c-ratios measured in laboratory C

Real w/c	Preparing lab. and measured w/c ratios					
	A	B	C*	D	E	
					1	2
0.20	0.20	0.20		0.20	0.20	0.20
0.25	0.25	0.25		0.25	0.25	0.25
0.30	0.30	0.30		0.30	0.30	0.30
0.40	0.40	0.40		0.40	0.40	0.40

*) not measured

Table 5
W/c-ratios measured in laboratory D

Real w/c	Preparing lab. and measured w/c ratios					
	A	B	C	D	E	
					1	2
0.20	0.20	0.20	0.20	0.20	0.20	0.20
0.25	0.25	0.25	0.25	0.25	0.25	0.25
0.30	0.30	0.30	0.30	0.30	0.30	0.30
0.40	0.40	0.40	0.40	0.40	0.40	0.40

Table 6
W/c-ratios measured in laboratory E

Real w/c	Preparing lab. and measured w/c ratios					
	A	B	C	D	E	
					1	2
0.20	0.20_e	0.20_e	0.20_e	0.25_d	0.20_e	0.20_e
0.25	0.25_d	0.25_e	0.25_d	0.20_d	0.25_d	0.25_e
0.30	0.30_d	0.30_e	0.30_d	0.30_e	0.30_d	0.30_e
0.40	0.40_e	0.40_e	0.40_e	0.40_e	0.40_e	0.40_e

$_d$ difficult to range
$_e$ easy to range

5. *Discussion*

The low viscous epoxy system used in this work did not show sufficient penetration capability for application in thin section production from high strength concrete. The poresystem in high strength concrete is so fine that only very small molecules with small intermolecular forces are allowed to enter. Very few epoxies are based on monomers that fulfil these requirements. The epoxies that do, are as mentioned earlier restricted by high volatility. Heating the epoxy and thus reducing its viscosity mainly through reduction of intermolecular interactions did not increase the impregnation depth. This is probably an indication of too large molecules rather than intermolecular forces.

Impregnation by Fluorescent Liquid Replace technique was for the most part successful. Only two of the twentynine tested w/c-series were determined incorrectly. The results show, however, that. However, as could be expected from a new technique, some of the laboratories ran into a few problems during preparation. Among these were uneven impregnation - which was observed once in laboratory C - and insufficient solubility of the fluorescent dye in ethanol which was experienced by laboratory D. Deviations between the real w/c-ratios and those observed in the round robin test were in fact also confined to thin sections from these laboratories. Uneven impregnation has later been observed when the polished surfaces of two samples come together. Differences in the solubility of dye in ethanol is probably caused by variations in the number of polar groups among the molecules.

Visual comparison of thin sections from different laboratories showed good agreement in brightness at equal w/c-ratios.

Laboratories B and E also reported whether the thin sections were easily separated according to w/c-ratio or not. Difficulties in separating thin sections were observed on samples with the w/c-ratios 0.20, 0.25 and 0.30. The thin sections were, nevertheless, in general separated correctly.

Later introduction of an additional liquid replacement of porewater with pure ethanol and slight drying in air before impregnation has improved this weakness.

The thin sections made using FLR technique showed no signs of micro cracking as opposed to thin sections made by vacuum impregnation where pronounced cracking were observed. This is in agreement with earlier work done during the development of the FLR technique [4].

6. *Concluding remarks*

It seems very difficult to solve the problem of penetration using epoxies for the impregnation of high strength concrete. This is clearly demonstrated by the total lack of penetration exhibited by the epoxy used in this work.

Fluorescent Liquid Replacement technique showed satisfactory results combined with reduced health hazards. The technique may with a few recently added refinements be proposed as a Nordtest method.

7. *References*

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Mollatt 24.2.