Pergamon

Cement and Concrete Research, Vol. 25, No. 5, pp. 1063-1074, 1995
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0008-8846/95 $9.50+.00

0008-8846(95)00101-8

FLUORESCENT LIQUID REPLACEMENT TECHNIQUE. A MEANS OF CRACK DETECTION AND WATER:BINDER RATIO DETERMINATION IN HIGH STRENGTH CONCRETES

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(Refereed)
(Received September 20; in final form December 2, 1994)

ABSTRACT

This paper presents an impregnation technique based on fluorescent ethanol that makes it possible to do plane section and thin section analysis on concretes of very low w/b-ratios. The technique is called Fluorescent Liquid Replacement technique (FLR) and is based on a principle where the pore water is slowly replaced by ethanol. Tests have been done on virgin concrete samples and in combination with elements of traditional preparation procedure, drying and evacuation. A comparison to epoxy impregnation is done. Plane sections and thin sections are made from five different concretes with w/b-ratios 0.20, 0.25, 0.30 and 0.40 containing 10 % silica fume and w/b-ratio 0.35 without silica fume.

Results show that the FLR technique gives impregnation depths that are several orders of magnitude larger than what is obtained with traditional procedures. Measured fluorescence intensities show that w/b-ratios may be determined in the range from 0.20 to at least 0.40. An extensive crack pattern was seen at lower w/b-ratios using the traditional epoxy impregnation technique and FLR in combination with drying and evacuation which is in agreement with earlier experiences. FLR technique used on wet virgin samples did not produce cracks. The observed cracks seem to be an effect of preparation.

Impregation measurements are supported by exchange studies done by gravimetry and Nuclear Magnetic Resonance technique on samples of hydrated cement pastes

Introduction

This paper emerged as a result of work done to adapt thin section and plane section analysis for use on high strength concretes. An important part of the preparation of thin sections and plane sections from concrete is impregnation of the material with an epoxy containing a fluorescent additive. The fluorescent additive makes internal pore structures like airvoids, cracks and capillary pores visible in ultraviolet light. By capillary pores in this connection we refer to paste pores that fill with impregnation fluid. It is not implied that this (unknown) pore volume corresponds to capillary porosity as defined by Powers [1]. Due to the relationshipship between water:binder ratio and the capillary porosity, a measurement of the water:binder ratio in the original mix may be obtained by comparing fluorescence intensities [2,4]. A problem frequently encountered in thin section and plane section analysis is the normally poor penetration capability of the fluorescent epoxy system [3]. A successful water:binder ratio determination in general is dependent on a sufficient impregnation depth, both to avoid the impregnated layer's is accidental removal by inaccurate grinding, and to obtain reproducible measurements. For thin sections, a total penetration through the whole depth, i.e. at least about 25 μm. is required. This is normally only achieved through application of a vacuum to assist impregnation. Even after a successful impregnation, the penetration depth in concrete is typically only some 50 μm, which gives little room for inaccuracies in final grinding and polishing.

The problem increases in high strength concretes. At water:binder ratios below 0.35, the penetration depth of traditional epoxy systems rapidly decreases to zero. In concretes where silica fume is added, even a water:binder ratio of 0.40 may cause impregnation difficulties.

Furthermore, drying and vacuum impregnation as used in the traditional preparation procedure must be regarded as a rather harsh treatment, and could be the cause of an extensive pattern of internal micro cracks frequently observed in thin sections and plane sections made from concretes of higher compressive strengths [3, 7].

In this work the problems with insufficient impregnation and possible introduction of micro cracks during preparation are solved by immersing concrete specimens in a solution of ethanol and a fluorescent dye instead of vacuum impregnation with a fluorescent epoxy. The process is based on the slow liquid replacement of pore water by ethanol, and is in this work called Fluorescent Liquid Replacement (FLR) technique. In the following, the applicability of the technique is tested on concretes of five different water:binder ratios. Of these, four concretes were made with the addition of silica fume and one without.

To support the work, the FLR process was also studied gravimetrically and by Nuclear Magnetic Resonance (NMR) technique using hydrated cement pastes (HCP).

Experimental

For convenience, this part only covers the FLR preparation technique and a description of the concrete specimens that were made to test the use of the technique. A description of the further treatment of the samples and measuring techniques is given under measurements and results.

Description of preparation procedure using FLR technique

Except for the impregnation, the thin section and plane section preparation using FLR technique closely resemble the traditional epoxy preparation procedure. The traditional procedure is fully described elsewhere [4]. Only a brief summary is therefore given here to point out the differences that result from changing to FLR technique.

The preparation of thin sections and plane sections starts by cutting a specimen to a convenient shape and size. After cutting, the specimen is ground and polished on one side. Contrary to the normal procedure where the specimen is dried at 30 °C – 50 °C before impregnation, the specimen is immersed in a solution of ethanol and fluorescent dye for four days. The concentration of fluorescent dye in the solution should be 1.0 % by weight of solvent. During the four days of immersion, the fluorescent solution replaces free water in the concrete specimen. After the specimen is removed from the solution, excess fluorescent dye is removed by slight repolishing. The ethanol is allowed to evaporate until the surface appears dry. The surface is then covered with a thin coat of fluorescent epoxy. Excess epoxy is squeezed away by pressing the coated surface of the specimen against a plane surface. A 20 mm thick steel plate covered with a sheet of 0.1 mm plastic is used to avoid sticking. In case the specimen is to be used as a plane section, the preparation is now terminated by a slight grinding to remove excess epoxy.

For thin section preparation, the traditional procedure is followed by gluing the sample to a glass plate, cutting it with a saw and finally grinding it to specified thickness.

Final "fixing" of the surface with epoxy was done to eliminate the possibility that the surface should change character over time and to fill larger air pores.

The preparation was done on a Struers Diskoplan TS lapping machine and a Struers DAP V polishing machine. The fluorescent dye was of type Hudson Yellow which absorbs ultraviolet light at a frequency of 515 nm and emits at 535 nm.

Specimens

Investigations were carried through on a specimen set of five different concretes numbered 1 to 5. All concretes were cast as 150 mm cubes. Concretes 1 to 4 were high strength concretes containing 10 % by weight of silica fume and with compressive strengths measured after 28 days above 100 MPa. Concrete 5 is a well hardened concrete without silica fume and with a compressive strength of about 40 MPa. All concretes were demoulded 24 hours after casting and stored in water for one year before they were used in this work. Characteristics of the different concretes are given in table 1 below.

TABLE 1
The table gives data on the different concretes.

Concrete	water binder-ratio	Cement content (kg/m^3)	Condensed silica fume (%)	Compressive strength (MPa)
1	0.20	585	10	111
2	0.25	540	10	107
3	0.30	450	10	119
4	0.40	360	10	101
5	0.35	450	0	40

The samples, one plane section (70 mm x 70 mm) and two thin sections (30 mm x 45 mm, and a thickness of 25 μm), were prepared from each of the concretes except for concrete number 5 where only thin sections were made. All samples were cut from the center of each cube.

Measurements and results

The plane and thin sections, were studied in a combined incident and transmitting light microscope of type Leitz Laborlux 12 POL using fluorescent light. The observed parameters were impregnation depth, relative intensity of fluorescence and cracking. The hydrated cement pastes were studied gravimetrically and by Nuclear Magnetic Resonance technique (NMR) to determine the amount of exchange between ethanol and pore water.

Impregnation depth

The impregnation depths were measured by cutting a part from each sample vertically from the prepared surface after impregnation. This gave a cross-section of the penetration which could be studied in the microscope. In addition to being influenced by the water:binder ratio, the impregnation depth was also influenced by aggregate and cracks near the surface. The measured impregnation depth for each w/b-ratio is therefore given by the observed maximum and minimum values. The results from FLR impregnated samples are shown in Table 2. Measurable impregnation using epoxy was only obtained on concrete 5 (0 μm - 50 μm).

TABLE 2
Maximum and minimum impregnation depths measured for different w/b-atios using FLR technique.

Concrete	w/b-ratio	Impregnation depth (mm)
1	0.20	0 - 0.1
2	0.25	0.5 - 1.5
3	0.30	0.5 - 2.5
4	0.40	1.5 - 2.5
5	0.35	> 10

Fluorescence intensity

The measurements of fluorescence intensity were done by using the photoautomat exposuremeter already connected to the microscope. The intensity was measured both on plane sections and thin sections.

The surfaces of the plane sections were divided into 9 imaginary areas and the measurement was done on the hydrated cement paste in the middle or as close to the middle of each area as possible. Where aggregate covered the major part of the section, the section was rejected.
The surfaces of the thin sections were similarly divided into six areas where the intensity was measured as described for the plane sections. Due to the smaller size, only four measurements were done on each of the thin sections from concrete 5. The measured mean intensities and standard deviations are shown in Table 3.

TABLE 3
Water-cement ratios and corresponding mean fluorescence intensities and standard deviations calculated for thin sections and plane sections.

Concrete	w/b	Thin section		Plane section	
		Mean intensity	σ_x	Mean intensity	σ_x
1	0.20	0.0318	0.0059	0.333	0.111
2	0.25	0.0554	0.0205	0.570	0.143
3	0.30	0.0867	0.0206	1.14	0.176
4	0.40	0.184	0.0384	2.51	0.186
5	0.35	-	-	3.10	0.419

Cracking

Cracks of widths in the range of about 1.0 μm and upwards are easily detected in the microscope using ultraviolet light. Both FLR-impregnated and epoxy-impregnated samples were examined for cracking. In order to see the effect of drying and evacuation as used in the traditional procedure, FLR technique was applied to virgin samples as well as to samples dried at 50 °C or 105 °C for 6 hours and samples exposed to evacuation at 10 millibar for 15 minutes. Drying at 105 °C is not used in traditional preparation procedure, but is included for comparison because of its frequent use where complete removal of free water is required.

Epoxy-impregnated samples and samples produced with FLR technique in combination with drying or evacuation did all show varying degrees of cracking at w/b-ratios below 0.40. Cracks were also observed at 0.40, but then as longer irregular cracks and not a continuous finer pattern. The extent of the cracking increased with reduction in w/b-ratio and drying temperature. Evacuation produced a crack pattern similar to or a little worse than drying at 105 °C. Virgin samples prepared with FLR technique alone did not show any sign of cracking. See also photos, Fig. 1 and qualitative survey, Table 4.

TABLE 4

Qualitative schematic outline indicating the extent of microcracking in concretes impregnated with FLR technique and epoxy. The table indicates the extent of cracking where FLR technique is used on virgin samples and in combination with drying or evacuation.

Concrete	w/b-ratio	FLR				Epoxy
		Virgin	50 °C	105 °C	Vacuum	
1	0.20	0	2	2	2	2
2	0.25	0	2	2	2	2
3	0.30	0	1	2	2	2
4	0.40	0	1	1	1	1
5	0.35	0	1	1	1	1

0 = none; 1 = some; 2 = extensive

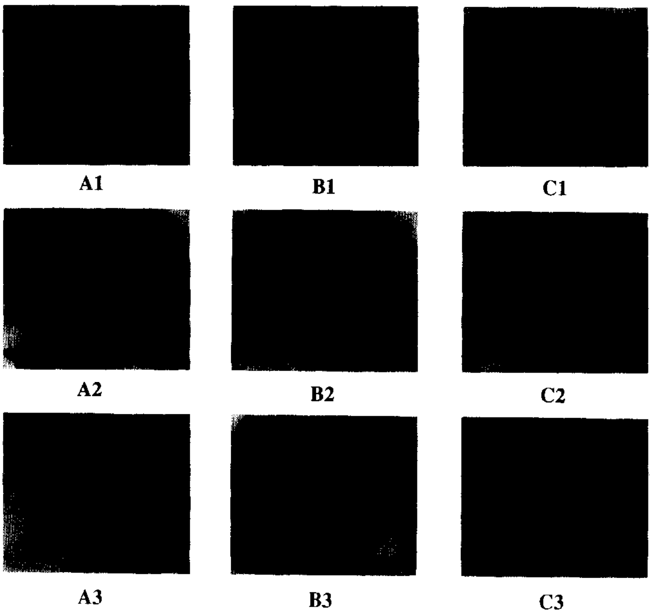

FIG. 1

Selection of photographs showing the extent of microcracking in concretes 1, 2 and 4. Photographs A1, B1 and C1 (concrete 1) show virgin samples treated by FLR directly after storage in water. A2, B2 and C2 (concrete 2) show samples after drying in 50 °C for 6 hours. A3, B3 and C3 (concrete 4) show the effect of drying in 105 °C for 6 hours.

Amount of exchange between ethanol and pore water

A good and reproducible impregnation depends largely upon the extent of exchange between the ethanol and the pore water. The amount of exchange was determined both gravimetrically and with Nuclear Magnetic Resonance spectroscopy, NMR.

The gravimetric determination was done by measuring the weight reduction, Δm, of a sample from saturated surface dry condition after four days of liquid replacement. The weight reduction may be expressed as the difference between the mass of water that has left the pore system and the mass of ethanol that has entered.

$$\Delta m = m_{\text{water, out}} - m_{\text{ethanol, in}}$$

Assuming a volume by volume exchange this gives

$$\Delta m = m_{\text{water, out}} - m_{\text{water, out}} \cdot \rho_{\text{ethanol}} / \rho_{\text{water}}$$

where ρ_{ethanol} is the density of ethanol ($\rho_{\text{ethanol}} = 0.7893$ g/ml) and

$$m_{\text{water, out}} = \frac{\Delta m}{0.2107}$$

Using this expression, a set of three samples of hydrated cement paste with w/b-ratio of 0.4 and a content of 10 % silica fume was tested for amount of exchange after four days immersion in pure ethanol. The samples, each weighing between 2.5 g to 3.5 g, and with a thickness of 3 mm to 5 mm, gave a mean value of $m_{\text{water, out}}$ compared to the amount of evaporable water (105°C) of 74.8 %, leaving the same space for ethanol.

Measurements by Nuclear Magnetic Resonance

The NMR measurements were done on a 200 MHz Bruker CXP spectrometer at the University of Oslo. The spectrometer was set to observe the protons (hydrogen atoms) in water and ethanol.

The experiments were done by placing a water-saturated sample of white HCP in an NMR-tube surrounded by a known mass of ethanol. Sample diameter was approximately 3.5 mm and length about 10 mm. Tube diameter was 5 mm. The sample which was made with w/b-ratio of 0.40 had been cured in water for more than ten years. The w/b-ratio was chosen as well-cured samples of white cement with lower w/b-ratios were not available. White cement was used as a safeguard as the higher iron content of normal cements may interfere with the NMR parameters. The actual masses were m_{HCP} : 0.3441 g and m_{ethanol}: 0.3048 g. The HCP-sample was situated outside the coil supported by a thin glass rod so that only the ethanol could be observed. See Fig. 2.

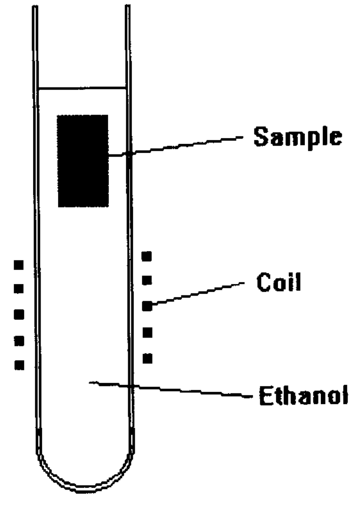

FIG. 2
The illustration shows how the NMR tube and the HCP-sample is placed relative to the RF-coil.

The NMR-technique is quantitative when used in combination with a reference sample. In this case the reference sample was made by adding a known amount of pure ethanol to a predried sample of white hardened cement paste.

Recording of the amount of exchange was done by measuring the amount of water entering the ethanol from the HCP-sample at certain time intervals. The first spectrum was recorded one minute after the HCP-sample was brought in contact with the ethanol and the last after 102 hours. A weight reduction in the HCP-sample of 4.9 % was recorded during the experiment. Two spectra taken after 1 minute and after 62 hours are shown as examples in Fig. 3. The peak to the left in each spectrum represents the sum of OH-protons in the ethanol and the protons in water. An increase in peak area of 56.0 % can be seen. The other two peaks originating from the ($-CH_2-$) and ($-CH_3$) groups in ethanol respectively, show a decrease of 11.9 %. These groups are specific to the ethanol and are thus a direct measure of the fraction of ethanol present during the whole experiment. In pure ethanol, the peak intensities are proportional to the number of protons in each group, giving a ratio between the intensities of the three peaks $CH_3/CH_2/OH$ of 3:2:1. A more detailed description of NMR may be found in various textbooks [5].

In the following a calculation of the amount of water entering the ethanol is done. As the NMR measurements only observe the signal from the protons within the coil and not the whole ethanol solution surrounding the HCP the amount of water entering the ethanol is expressed as the ratio between the number of moles of water and the number of moles of ethanol.

As the total intensity of the OH/H_2O-peak, I_{tot}, at any given time may be expressed as the sum of intensity contributions from the protons in water, I_{H_2O}, and the OH-protons in ethanol, I_{OH}, within the coil, we can write

$$I_{tot} = I_{H_2O} - I_{OH} \qquad (1)$$

The proton signal intensity and the number of moles of protons contributing to the signal may be regarded as proportional. From the CH_3-peak and the CH_2-peak we then have

$$I_{CH_3} = 3\,k\,n_E$$

and

$$I_{CH_2} = 2\,k\,n_E$$

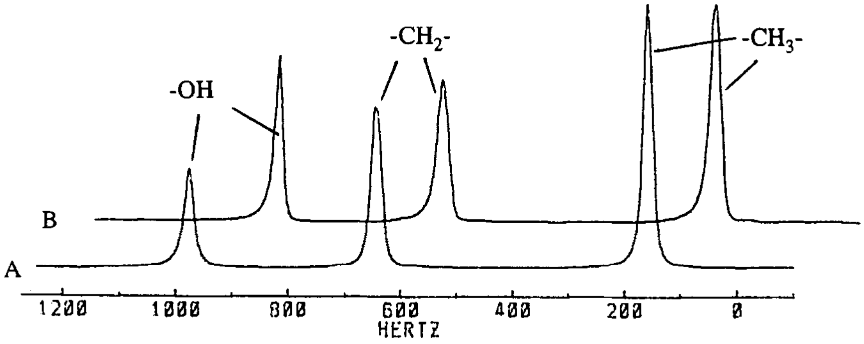

FIG. 3
The illustration shows proton NMR-spectra from the ethanol. Spectrum A is recorded one minute after the HCP-sample was placed in the ethanol. Spectrum B is taken 62 hours later and shows a marked increase in the OH/H_2O-peak to the left caused by water entering the ethanol from the HCP.

(where k is a proportionality constant and n_E is the number of moles of ethanol present within the coil. The right side is multiplied by 3 and/or 2 to correct for the number of protons contributing in the ethanol molecule which contribute to each peak.

Using both the CH_3-peak and the CH_2-peak we can write

$$I_{CH_3} - I_{CH_3} = 5\,k\,n_E \qquad (2)$$

In the same way the intensity contribution to the OH/H_2O-peak from water may be written as

$$I_{H_2O} = 2\,k\,n_{H_2O} \qquad (3)$$

Combining equations (2) and (3) gives the ratio between the number of moles of water and ethanol within the coil at any time during the experiment

$$\frac{n_{H_2O}}{n_E} = \frac{5}{2}\left(\frac{I_{H_2O}}{I_{CH_3} + I_{CH_2}}\right)$$

Using equation (1) we get

$$\frac{n_{H_2O}}{n_E} = \frac{5}{2}\left(\frac{I_{tot} - I_{OH}}{I_{CH_3} + I_{CH_2}}\right)$$

As $5\,I_{OH} = I_{CH_3} + I_{CH_2}$ we then have

$$\frac{n_{H_2O}}{n_E} = \frac{5}{2}\left(\frac{I_{tot}}{I_{CH_3} + I_{CH_2}} - \frac{1}{5}\right)$$

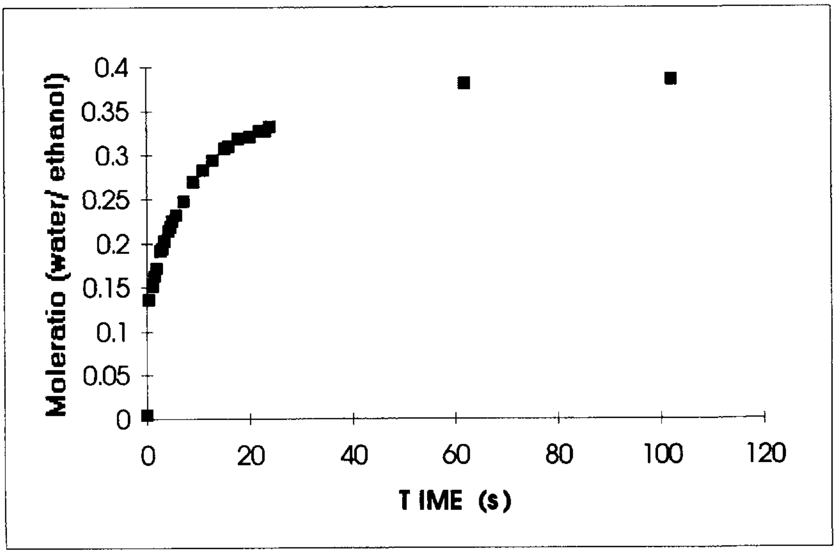

FIG. 4
The plot shows the amount of water entering the ethanol vs. time. The amount of water is expressed by the ratio n_{H_2O}/n_E.

A plot showing the increase of water in the ethanol expressed by the ratio n_{H_2O}/n_E versus time is shown in Fig. 4. It shows that a considerable amount of water is extracted after 24 hours and that an equilibrium value is reached after less than three days (62 hours). The ratio n_{H_2O}/n_E observed after 102 hours is 0.38.

Whether the water in the pore system is replaced by ethanol may be seen by comparing the total volume of liquid in the NMR-tube at the start of the experiment and after 102 hours. The HCP-samples used in this work showed an evaporable water content of 17.6 % w/w determined by drying to constant weight at 105°C. The total volume of water in the saturated HCP poresystem and ethanol may be calculated to 0.4469 ml at the beginning of the experiment. Assuming homogeneous mix between water and ethanol in the pore volume and outside the HCP after 102 hours and correcting for the increased density of the mix [5] a reduction of the total volume of about 2.1 % should be observed. Assuming, on the contrary, that no ethanol replaces the pore water, using the n_{H_2O}/n_E-ratio of 0.38, an increase in total volume of 7.8 % would result. No increase in total volume of liquid could be seen, however, indicating that roughly 60 % to 70 % of the pore volume is filled by ethanol.

Discussion

Impregnation depth

The observed impregnation depths using FLR technique are several orders of magnitude larger than what is obtained with traditional epoxy impregnation techniques on concretes of ordinary strengths [7]. The observed differences in impregnation depth within each of the samples is mainly due to the aggregate, which impedes the impregnation. When a solute is transported by a solvent through a porous medium, a separation or "chromatographic effect" between the solute and the solvent may occur, with poor reproducibility of w/b-ratio as a possible consequence. A separation should leave the fluorescent dye behind in the outer layers of the sample as the ethanol moved inwards. Such a behaviour is not seen, although it should not be ruled out. Differences in intensity between the outer and inner parts of the impregnated area are not observed, however, and as the impregnation depth is good a possible separation seems to be of secondary importance. One problem experienced with the technique, is that larger pores are not filled with fluorescent material during impregnation. These pores will therefore be difficult to detect in the microscope. It is easily remedied by dripping fluorescent epoxy over the surface and removing the excess before final grinding and polishing.

Fluorescence intensity

The different w/b-ratios are easily distinguished by the intensity of the fluorescence both in thin sections and plane sections. The relation between the intensity of fluorescence (I) and the w/b-ratio (N) is found to be logaritmic over the range from w/b = 0.20 to w/b = 0.30. It follows the equation

$$\log I = \alpha N + \beta$$

Linear-logarithmic plots of measured fluorescence intensities in thin sections and plane sections vs. w/b-ratio are shown in Fig. 5 below. It can be seen from the plots that the linearity is lost at w/b-ratios above about 0.4. Water:binder ratio determination using FLR technique is therefore best suited for concretes of w/b-ratios below 0.4, as w/b-ratios above this are difficult to distinguish.

The coefficients α and β are determined to approximately 0.55 and -0.08 for the thin sections and 8.1 and -1.3 for the plane sections. The difference in α reflects the greater span in intensity that is observed for plane sections. A possible effect of binder phase composition (such as cement type, content of silica fume and flyash) on α and β will be investigated in a later work.

The plane sections are brighter than the thin sections. This is a consequence of the larger thickness of the fluorescent layer of material in the former compared to the latter. Lack of silica fume seems to increase the fluorescence intensity corresponding to w/b-ratio between 0.05 and 0.10.

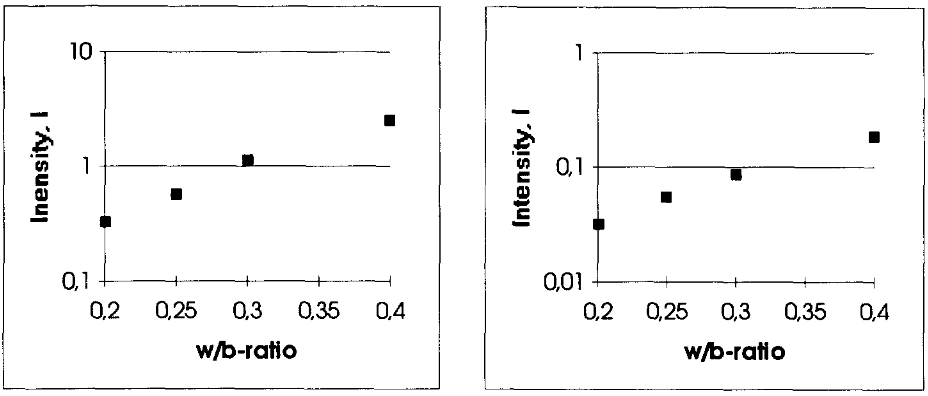

FIG. 5
Fluorescence intensities plotted v.s w/b-ratio measured in plane sections (left) and thin sections (right).

The precision in readings varies more over the w/b-range for plane sections than for the thin sections. This is especially seen with the lower w/b-ratios. The reason is most probably that the readings here are more influenced by local differences in impregnation depth than at higher w/b-ratios where the impregnation is deeper. The precision is still within acceptable limits with an uncertainty of at least 5 x 10^{-2}. From this and the greater span in intensity, it seems that plane sections are equally or better suited for w/b-ratio determination than thin sections. Differences in intensity are evident also without using the photoautomat.

A round robin test to check the feasibility and reproducibility of w/b-ratio determination using the FLR technique has been run with good results [8].

Cracking

The cracks seen using traditional preparation technique and FLR in combination with drying or evacuation are not reproduced in FLR-treated virgin concrete samples. The cracking therefore seems to be an effect of preparation only.

The principle of the liquid replacement technique is based on the ability of two miscible liquids to replace each other in a porous structure. When a concrete sample containing water is placed in a vessel containing a large amount of ethanol compared to the amount of water in the pore structure of the sample, a slow dilution process starts. The pore water moves out into the vessel and ethanol into the pore structure. At the end of the process, an equilibrium is reached where, ideally, the concentration of water outside and inside the concrete are equal and most of the pore water has been replaced. The surface tension of the ethanol is smaller than for water. When the ethanol afterwards evaporates from the pores, cracks are thus less likely to occur.

Cracking is mainly observed at lower w/b-ratios no matter which drying procedure is used, except using "pure" FLR technique (Table 4). There seems to be a boundary between 0.3 and 0.4. This is in accordance with earlier work [7]. It seems that high strength concrete is very susceptible to vacuum treatment. Additional tests did reveal cracking after periods of evacuation down to a couple of minutes. A more thorough investigation of cracking as a result of drying and evacuation will be published in the near future.

Amount of exhange between ethanol and water

The amount of water exchange after four days measured by gravimetry, 74.8 %, and by NMR technique, between 60 % and 70 %, are in relatively good agreement and shows that a considerable amount of exchange is achieved. The somewhat lower value measured by NMR may be explained as the samples used for gravimetry were in contact with a very large amount of ethanol compared to the NMR samples where the amount of ethanol was restricted by the relatively small volume of the tube.

The measurements suffer from the weakness that the w/b-ratio is higher than in the the concretes prepared with FLR technique. The samples are on the other hand cured for a much longer period than the concretes. This in combination with the w/b-ratio of 0.40, should still give a relatively dense pore structure with very few capillary pores to assist exchange, at least compared to the concretes in the upper w/b-ratio range.

NMR-spectroscopy is a powerful technique in studies of the pore structure and transport processes in porous media like hydrated cement pastes. Further work done on the paste itself, where studies of transport mechanisms play an important part, are already in progress and will be published shortly.

Concluding remarks

Due to its great impregnation capability, the FLR technique provides a possibility to measure w/b-ratios in high strength concretes that is unmatched by traditional techniques. The technique is best suited for w/b-ratio determination below about 0.4. The FLR technique must therefore be regarded as a supplement to traditional impregnation technique as regards w/b-ratio determination. W/b-ratio may be determined both on plane sections and thin sections. Gravimetric measurements and NMR experiments indicate that a considerable exchange between water and ethanol takes place, approaching equilibrium after four days.

The extensive pattern of microcracks in high strength concrete that has earlier frequently been observed using traditional impregnation techniques seems to be purely an effect of preparation, since it is not observed when the FLR technique is used. The FLR technique therefore seems to be an interesting tool to detect crack development in general due to influences like loading, freeze/thaw, corrosion etc. It has recently, after the development of the FLR technique, been applied to high strength concrete subjected to rapid freeze/thaw cycles (ASTM C666) [9]. It was clearly demonstrated that deterioration of the concrete in terms of reduced dynamic elastic modulus, was accompanied by development of microcracks and increased degree of water saturation.

Acknowledgements

The author wants to thank Dr. Eddy Walther Hansen of SINTEF Oslo for enthusiastic participation in discussing the results of the NMR-part of this work.

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