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Dealing with uncertainty in material characterization of concrete by education

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Abstract

In this article a trip is taken to characterize concrete through its hardened cement paste based on its microstructure. Optical microscopy is coupled to advanced image analysis, as well as statistical data analysis to characterize heterogeneous concrete material. The article takes a closer look at the determination of the w/c ratio through the analysis of capillary porosity by using image analysis. It is shown that accurate analysis does not shown identical results in the w/c ratio even for standard reference samples with known w/c ratio. In the article it is shown that this is not because the techniques we use to determine the w/c ratios are not accurate enough, but much more because of the local variation in the microstructure. The attention shifts with this article from the analysis technique to what we actually observe in the microstructure. Hopefully this will change our thinking about the distribution in local microstructure variation and will help to set off more research towards the capturing and modelling of these local microstructure variation into consequences at a more structural level.

Keywords: water-cement ratio, microscopy, thin section, image analysis, microstructure distribution

I. Introduction

Some things never seem to change and yet they do. It seems like we are making concrete already more than 100 years in much the same way. We require a certain amount of rocks (both fine and coarse), cementitious material and water to make our trusted, well known concrete material. To be sure we can rely on the quality of our concrete materials the standards regularly prescribe e.g. minimum cement content, maximum water-cement ratios, and more if deemed necessary, see (European Standardization Committee, 2009).

However, times are changing. Slowly but surely our way of making concrete changes from a prescriptive way of defining and regulating our concrete to a more performance based approach (European Economic Community, 2011). Driven by the increased attention for sustainability, allowing a wider range of material components to be part of our concrete, thus fulfilling the wish to reuse and recycle, making concrete by prescribing its components and the recipe is not enough anymore. A need is emerging to determine and satisfy performances of the concrete material which goes beyond the 28 day strength alone.

In this article a trip is taken to characterize material based on its microstructure. Optical microscopy is coupled to advanced image analysis, as well as statistical data analysis to characterize heterogeneous concrete material. It is thought that this can be a way forward in determining material performance in an ever growing world of different ways and components to make concrete.

Before the material characterization is described, a start is made at the structural level; what does the structural engineer need and how does he deal with uncertainties in available data? With this illustration in mind a change in scale and representative volume is made to go down to the microstructure level and its heterogeneity. How accurate can we determine this through the proposed methods, and how can we deal with the uncertainties found in this process?

II. The structural design approach

Just to serve as an example reference is made to the North Boulevard Bridge project in the city of Baton Rouge, Louisiana (McLellan, 2009). Here Louisiana's first high performance concrete bridge was built, open to traffic in 2006. The bridge aesthetics were provided by the smooth surfaces of the graceful and slender precast, prestressed concrete U-beams, and the uniquely sculptured concrete arch-shaped piers, see Figure 1.

To design the box girder, the structural analysis and calculation required a minimum strength of 69 MPa (10,000 psi) at 56 days as one of the performance requirements for the concrete. The term minimum already indicates there could be a varia-

Figure 1: The North Boulevard Bridge in Louisiana made from high strength concrete

tion in the strength of the produced beams. In other words, standing below the bridge, looking up at the box girders, there is certainly one that is the weakest, but which one would this be? If a coring sample would be necessary from the box girders to check the strength, which beam should be taken and how many samples would be needed to indicate if the performance has been met? Keep this example in mind when we come back to a similar choice in sampling for microstructure characterization.

With the performance requirement set, the next step is to design a concrete mix that can fulfil the requirement. In concrete design it was the research work of Abrams (Abrams, 1918) that showed a simple relation between the concrete strength and its water-cement ratio. Abrams' law as it became known, effectively states that for workable concrete, the lower the water-cement ratio, the higher the strength of the concrete. Abrams reported his results through Figure 2, using a water-cement ratio by volume. As nowadays the water-cement ratio is defined by mass, the graph has been replotted in Figure 3, considering that the cement density used by Abrams was 94 lb. per cubic ft. (Abrams 1918).

The mathematical expression Abrams came up with to aid the material design was a marvelous simplification of all the various components and influences on the measured compressive strength. However, often it is forgotten that it was just an aid in the design process. Taking a closer look at Figure 3, it shows two things. First of all, the w/c ratio should be at the very low end of the scale, probably towards 0.20. Second, even the data of Abrams did not provide a clear strength value for a specific w/c ratio. It is more of a range.

In concrete technology, the possible variation in compressive strength is so well known, that it is sometimes forgotten or overlooked when considering materials properties of concrete not related to strength. Hence, just as a reminder a quick recapitulation. Take e.g. a ready-mix truck of concrete. It is one batch, all mixed in the same procedure. In practice you do not get much more homogeneous concrete than this. Now use this batch of concrete completely to produce standard cubes or cylinders, which are stored under standard conditions and all tested after 28 days for compressive strength.

The results from a testing point of view could

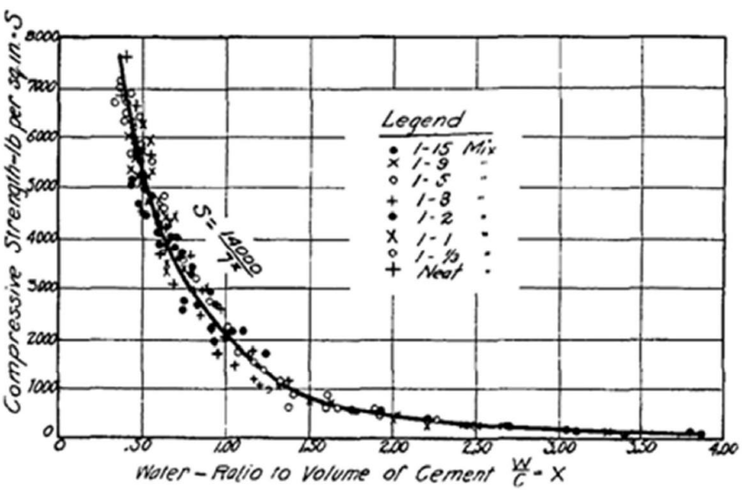

Figure 2: Original plot from Abrams (Abrams, 1918), using water-cement ratio by volume

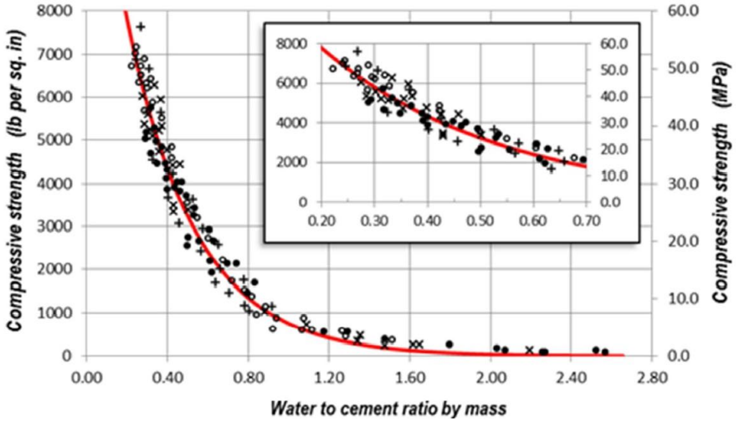

Figure 3: Replotted Abrams graph, using water-cement ratio by mass

be considered strange, because the compressive strength values are not all the same (see Figure 4) even though all the samples are produced from the same batch and hence are from a composition (recipe) point of view all identical. Nevertheless we have come to accept this variation as part of the inherent heterogeneity of concrete. In fact, being able to capture the distribution of the results in a Gauss curve, we have learnt to deal with the material variation at a structural design level. It is exactly the reason why the needed structural strength as a performance requirement is specified as a minimum strength or sometimes characteristic strength.

For the North Boulevard Bridge project the high performance mix proportions used came to be with a w/c ratio of 0.25. A little over 10 liter of high-range water reducer was necessary per m^3 to maintain a workability of the concrete within the specified range. The compressive strength from the concrete produced from this high performance mix was followed and measured based on test cylinders for 23 castings. After 1 day the compressive strength was on average already 47 MPa with a range of 28 - 60 MPa. After 28 days the numbers had increased to an average value of 94 MPa with a range of 68 - 109 MPa. Hence, the minimum strength requirement of 69 MPa after 56 days was not a problem.

It is interesting to note that the variation in the results reduces in the relative sense when going from 1 day test results (spread around 30%) to 28 day test results (spread around 20%). Nevertheless, going back to the original design where the structural engineer had designed the entire structure with a compressive strength of 69 MPa, it is fair to state that the vast majority of the bridge material has a strength well above the design strength.

III. Connection to the material microstructure

From the previous section it is clear that the concrete used is not a homogeneous material, but has a distribution, at least for the property of strength.

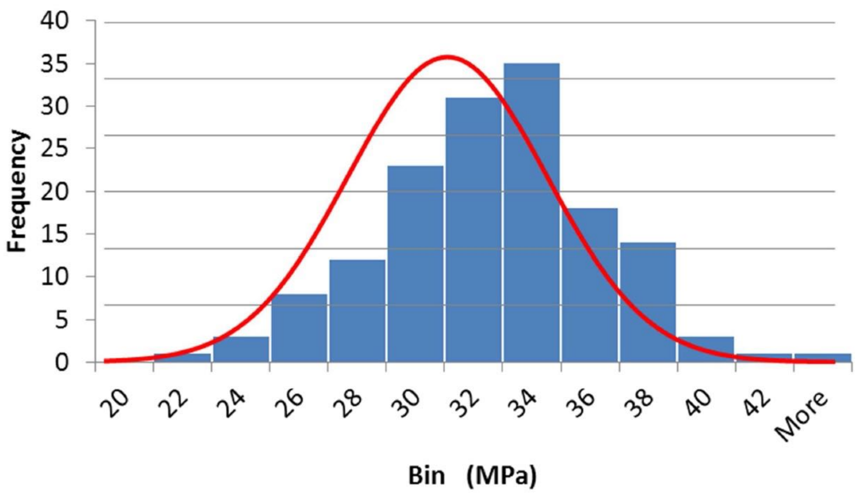

Figure 4: Example of strength distribution of one batch of concrete (histogram)

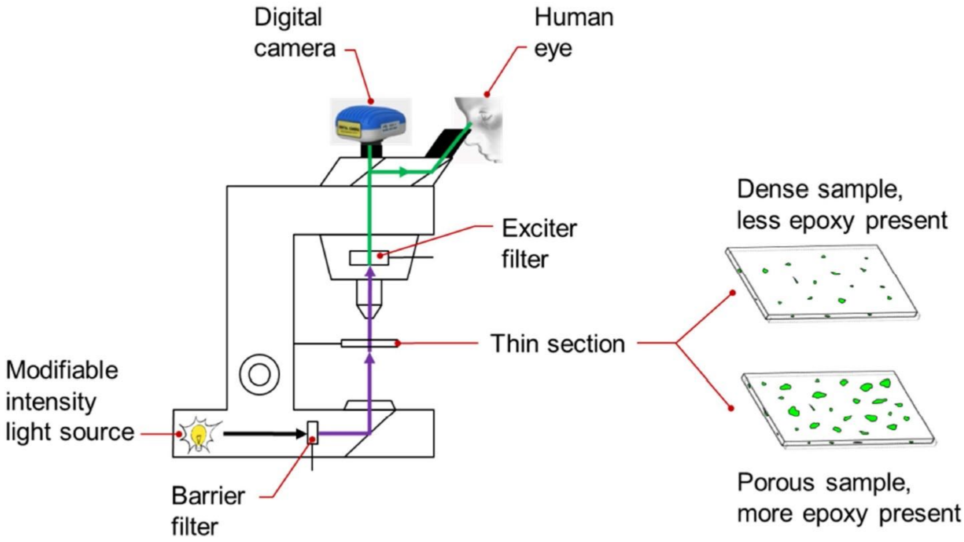

Figure 5: Schematic of optical microscope in transmitted light mode to examine a thin section specimen. A specimen with more porous microstructure shows brighter than a denser microstructure due to the difference in the amount of epoxy present in the microstructure.

This is not a problem for structural engineers because they define their performance requirements in terms of a minimum or characteristic strength.

One could ask if this heterogeneity is also present for other material properties of concrete. If so, it could perhaps help us to understand why certain parts of the structure degrade faster than other parts. It could help us in the understanding why the ingress of substances like e.g. chloride does not have a uniform frontline like in our models, but has instead a much more variable front. In order to see if it is possible to detect such microstructural difference a closer look is taken at the concrete microstructure.

Staying with the parameter strength, it was the research work of Abrams that linked the compressive strength of workable concrete to the water-cement ratio. Even though for normal concrete mix design, the concrete with w/c = 0.25 is outside the workable concrete range specified by Abrams, addition of a high-range water-reducing admixture created the right direction for designing a high performance concrete. Hence, even in this particular situation, the w/c ratio introduced by Abrams was not that far off. It helped in the design direction.

Following this importance of the w/c ratio, would it then be possible to also use the w/c ratio to characterize the material microstructure of concrete? In earlier times this first connection was made by Idorn (Idorn, 1967) and later by Thaulow and co-workers (Thaulow et al., 1982). The reasoning behind this was relatively simple. If a material has a certain property like strength, the value of that property should come from the material itself; in other words, its microstructure. A material with a weak microstructure would result in a material with a low compressive strength. Also, if the material microstructure would show strong characteristics, then the material as a whole would probably show strong.

The next step in the reasoning may have caused some misconception in the past. Historically, the reasoning is as follows. If the microstructure is responsible for the overall performance of the material, and if the microstructure is determined by its components then knowing the amount of components, like knowing the amount of water and the amount of cement (the water-cement ratio) creates a direct relation to properties like strength. Hence, reasoning shows that it is perfectly logical that Abrams found that water-cement ratio plays such an important role in the determination of the compressive strength of concrete.

Two mistakes were made in this reasoning. First the microstructure of a material is not only determined by its components (its mix design), but also by the process in which it is made (e.g. temperature, mixing, placing, curing, etc.). Second, the water-cement ratio had obtained too strong a position in the determination of the compressive strength. It had gotten to the point that the water-cement ratio determined the strength. However, already in the work of Abrams it is shown that one unique water-cement ratio does not result in one unique value of the compressive strength.

Nevertheless, led by the Nordic countries in Europe a method was developed and turned into a NORDTEST standard (NT Build 361 (Nordtest 1991-02), 1991), based on the analysis of microstructure using thin sections and optical microscopy. The method has been used extensively over the last 20 years to determine w/c ratio of hardened concrete. According to Round Robin tests performed in Denmark, the expected accuracy of the method is ± 0.02 when the analysis is performed by an experienced microscopist (Jakobsen et al., 1995).

A drawback of the method is that the analysis include the personal judgment of the microscopist, who estimates as best he can, in comparison with reference samples, the w/c ratio of the microstructure under investigation. This aspect together with the observation that the determined water-cement ratio came often back as a range rather than a fixed number may well have resulted in the very limited use of the technique for w/c ratio determination in other countries around the world.

It is here that the story also may have reached its own tail. As the w/c ratio has obtained a high common practice for the compressive strength, determining the w/c ratio back through the microstructure of a concrete expects again a high precision in the determination of the w/c ratio, as they are considered to be linked directly together. As the determination of the w/c ratio does not provide the accurate number that is expected, there must be something wrong with the method, forgetting in the process that one w/c ratio does not provide one strength, but a range of strengths, which is reflected in itself by a range of microstructures. Hopefully this article will bring back some nuance and understanding in this relationship between w/c ratio and strength.

IV. Representative volume

When a microscopy technique is involved, at some point the discussion need to touch upon the subject of representative volume. Through the very basic of the procedure of using a microscope one zooms in on the details. But at what point has the magnification become too large and are the details that are being looked at not representative any more for the whole structure? This dilemma is addressed through the topic of representative volume.

A material microstructure can always be considered as a repeating system. As long as the smaller volume through repetition can recreate the original larger microstructure, the representative volume is still there and can be used to say something about the complete microstructure.

For concrete under a microscope this system of repeated representative volumes could break down rather quickly when concrete is made with larger aggregates in the order of 32 mm or above; having a thin section with an area of 50 x 30 mm is probably not representing the entire microstructure anymore. When as a rule of thumb at least three times the largest aggregate is being used to obtain a representative volume, the thin section sample size should be in the order of 100 x 100 mm. Even though at TNO it is possible to produce such large thin sections, it is expensive and not very practical.

Ordinarily the microstructure of importance is the hardened cement paste, as well as the distribution of the smaller filler materials in interaction with the hardened cement paste. As this article focuses on the water-cement ratio, the representative volume of the level of the largest aggregate is not needed. However, at the level of the hardened cement paste the microstructure within a thin section of 50 x 30 mm is generally well within the necessary representative volume.

Next step is to determine what at that microscopic level is the required representative volume. This turns out to be a bit of a chicken and the egg story. When the microstructure is very homogeneous, larger magnifications are possible before the image in the microscope is not representative for the generalized microstructure anymore. At that time two choices can be made.

First and most easy choice is to zoom out; go to lower magnifications until the representative volume is restored. However, this approach loses details observed at higher magnifications. Therefore, the second approach is also used a lot. This consists of collecting more image information through multiple images. Ordinarily this was done by moving the sample around and try to mentally summarize the different field of views. Nowadays, the more chosen approach especially when quantifying parameters is to take multiple images and average through image analysis software and procedures. In this article the road of taking multiple images has been followed.

V. w/c ratio determination

At TNO w/c ratio determination is performed routinely by our microscopists in the way described by NT Build 361 (NT Build 361 (Nordtest 1991-02), 1991). However, as a side research line over the past years procedures have been developed to determine the w/c ratio of concrete through the use of image analysis techniques.

On this path we have not been the first to try so. For example Jakobsen et al. (Jakobsen et al., 1995) and Elsen et al. (Elsen et al., 1995) have tried and reported on these techniques already in the nineties of last century. A combination of camera resolution, computer possibilities and image analysis software may have resulted in a resting state of the developments possible in this area.

TNO gained renew interest in the topic around 2008. We have reported on our findings regularly in the conference series of EMABM with articles in 2009 (Einarsson et al., 2009) and in 2011 (de Rooij

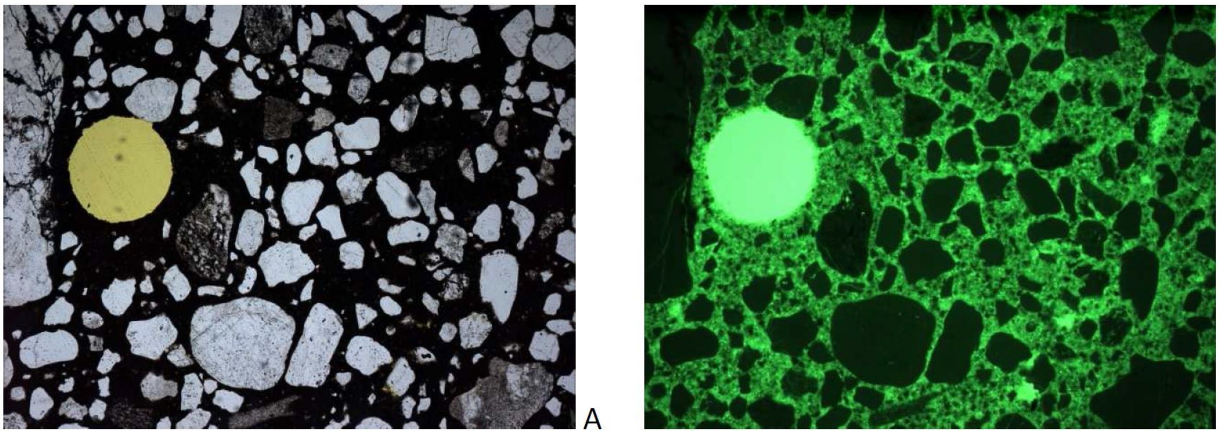

Figure 6: Same location of a concrete thin section observed under two different light modes: (A) normal light mode; (B) ultra violet light mode.

et al., 2011). This article follows up on the line reported in these two articles. For details on the technique please see the mentioned articles. Furthermore, publications to describe the entire procedure are in preparation for journal publication. Here only a short highlight of the used principles are given.

The backbone of the NT Build standard is presented in Figure 5. A concrete sample is prepared into a thin section, which is thin enough to look through. Normally thin sections are 25 - 30 μm thick, sometimes thinner. In the process an epoxy is used to stabilize the porous and brittle microstructure of concrete at these fragile scales. The epoxy enters the sample through a vacuum impregnation process causing it to settle at the dried pores of the capillary pore system. The epoxy contains a yellow dye that reacts in the ultra violet light range by exciting light. With the right filters this transforms an ordinary thin section to change from a normal light image as shown in Figure 6A, to an ultra violet light image as shown in Figure 6B.

An experienced microscopist can compare the general brightness of the UV-image to a set of reference samples with known w/c ratios to determine the w/c ratio of an unknown sample. The technique is based on the relation that a sample with a higher w/c ratio has a higher capillary porosity, which shows up as a brighter image due to the higher amount of epoxy being present.

Over the last years TNO has worked consistently to improve this technique from an experienced microscopist observation to a computer interpreted value with a calculated number rather than the approximate estimation of the experienced microscopist. In other words, we would like to take the honest but subjective interpretation of the individual as much as possible out of the equation. The current article is one step further on this path.

VI. Should two images provide the same answer?

In previous publications the technique how to determine a value for a water-cement ratio using computer analysis has already been described. It is possible. What remained is the question if two images taken from the same thin section should give the same w/c ratio result? That topic is analyzed in more detail here.

To obtain a reasonable answer on this question naturally the microstructure to be analyzed should be relatively homogeneous. Hence the investigation was not performed on damaged samples or on concrete with placement or degradation problems. Instead the analysis were performed on the reference concrete samples TNO has for its Portland cement with a known water-cement ratio. As these thin sections are made from samples with a known water-cement ratio, the first assumption is that the analysis of two spots of the hardened cement paste in the same thin section should provide the same water-cement ratio.

Unfortunately, regardless of how accurate and precise we tried to perform our analysis, almost never the results were exactly the same. Naturally, as the thin section was only containing one microstructure, for long we concluded that something must be wrong with our method, as we did not obtain the answer we were expecting. That is until we reached the point that no other conclusion was possible but the obvious one: it was not the method itself, but the local variation in the microstructure that we picked up.

Similar to the truck mixer of concrete not producing compressive strength results with all the same value, the production of reference samples did not result in an equal microstructure at all the same locations. But if the microstructure is not everywhere the same, how would we know which microstructure would represent the actual true water-cement ratio?

Again we followed the all too familiar path also used in compressive strength measurements: we need more images to be able to average towards the most probable water-cement ratio.

VII. Analysis of multiple images

More images, especially in the digital age is not a problem. However, it does make a difference how to analyze these images. If we have established in the previous section that it is very unlikely that two images give exactly the same result upon analysis, then the images are different. For reasoning purposes, let's assume that we have taken four images. To quantify these relatively to each other we have: a dark image, a medium-dark image, a medium-light image and a light image. When all four images are analyzed we will know the exact distribution of the results. However, what would be the result if only two images are analyzed? Does this then depend completely random on which images we took first? It could be that the averaged values are on the darker side when by chance only the dark and medium-dark image are taken. It could also be that the average results are on the light side when again by chance the light and medium-light image are analyzed only.

To solve this problem the research and analysis were set up as follows. From a thin section with a known water-cement ratio a total of 15 locations of the microstructure were photographed and analyzed individually. Next, the individual results were (figuratively speaking) placed in a basket. From this basket, using a computer script, 50,000 times sets of images were drawn and analyzed for mean and standard deviation. The drawing of the samples was done in such a way that once an image was analyzed in a set, it could not be redrawn and reappear again in the same set during the same drawing sequence. Thus plots were made for analyzing sets of 3 images, sets of 4 images, all the way up to sets of 15 images. This was done for the range of reference samples of Portland cement with different water-cement ratios.

In Figure 7, as an example, the results are shown for a w/c ratio of 0.65. The results are plotted in series with different amount of images selected to be analyzed. So the series of 3 images is the result of 50,000 times drawing sets of 3 images out of the total of 15 images. From this graph it becomes clear that more images indeed do narrow down the precision of the methodology. Please also note with what accuracy the water-cement ratio can be analyzed using this technique; the axis representing the w/c ratio is drawn with marks of 0.01 difference.

In Figure 8 the results of the different w/c samples are plotted in one graph, using only three images to analyze the w/c ratio each time. As the plot shows, it is quite difficult based on such a low number of analyzes to determine the w/c ratio of the sample under investigation. There are large overlaps in the possible water-cement ratios. However, turning to Figure 9, it becomes clear that an increase in the number of images provides a much more accurate reliability on the w/c ratio of the sample under investigation.

Figure 9 also shows another interesting result. Even though the preparation of the reference samples has been done in laboratory conditions with the utmost precaution, the variation in the final microstructure is not everywhere identical. From the distribution in the results it becomes clear that the sample with w/c ratio of 0.61 is slightly more heterogeneous than the other samples presented in the graph. This is an observation that can only be quantified in numbers through the image analysis technique used here. It shows that even our preciously made reference samples show different local variation in their microstructure.

VIII. How many images should we analyze?

The easy answer to the question how many samples should be analyzed is: more is better. However, that is only in relation to collecting data. Equally important is the realization what is needed, or requested from the results of all the data analyses. If the request is that the outcome of the analysis should be an exact w/c ratio without any distribution, than the quest to determine this has failed before it has started. The microstructure of concrete is heterogeneous and not identical at each and every place. Because there is variation it is not possible to come up with an answer without any deviation from the mean. It is simply not present in the material.

Hence, the question should be: what is being done with the result of the analysis? And how accurate should this result be to be useful input for further processing. Now the answer has a relation with the next step in the process. That introduces a dependency, which could lead to a more demanding accuracy in one case (and hence possibly more images to be analyzed), while in another case the answer could leave room for more uncertainty resulting in a lower number of images to be analyzed.

Obviously in this process two other parameters play a very important factor: more images usually mean a higher price that needs to be paid. The other factor is the heterogeneity of the concrete itself. A more homogeneous microstructure requires less images to be analyzed to come to a certain

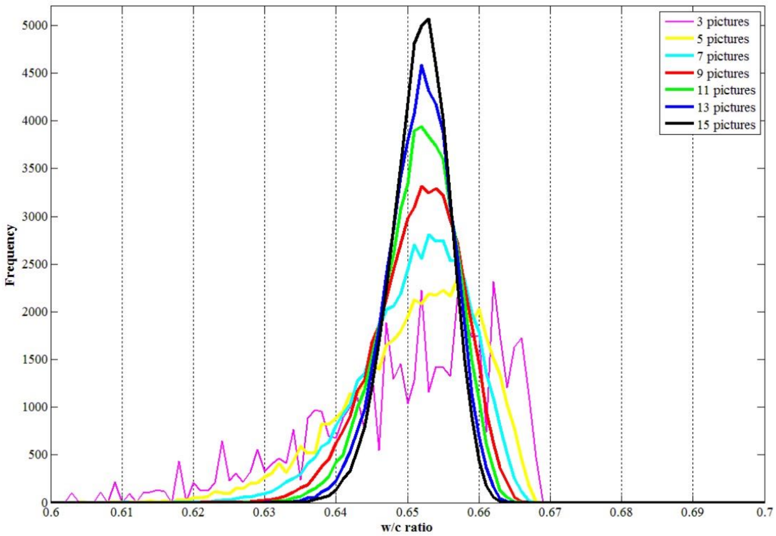

Figure 7: Histogram plot of 50,000 times selecting sets of 3, 5, 7, 9, 11, 13 or 15 images (see legend) out of a set of 15 images taken from a thin section with an original w/c ratio of 0.65 to determine the water-cement ratio.

precision than a very heterogeneous microstructure with large differences in the local microstructure.

To give some guidance in this process, the following comparison has been made. In the procedure described so far, random picks have been made out of a collection of 15 images per thin section sample. This provides us with a w/c ratio calculated from the average brightness value of the cement paste from the randomly picked set of specified number of images. By doing this 50,000 times a distribution in the w/c ratio is obtained. Using the standard deviation of this distribution, for instance the mean ± once the standard deviation (68% of all values) or mean ± twice the standard deviation (95% of all values) can be plotted in a graph to give an indication on the accuracy. This has been done in Figure 10 with the graphs labelled 'Pick'. The accuracy with which the w/c ratio can be determined is very precise.

Another option is to take just 3, 4, 5, or any of the numbers of pictures taken, and average the light intensities of these images. This then is being used to obtain a mean w/c ratio. In the process of obtaining an average light intensity also a standard deviation is obtained. Taking the mean brightness ± once or twice the standard deviation and using this to determine the corresponding w/c ratio provides a different way of obtaining w/c ratio distribution. These lines are marked 'All' in Figure 10. As can be seen, using this latter method to determine the w/c ratio distribution gives a much wider range of results.

Hence, to really characterize the distribution in the w/c ratio, or rather in the heterogeneity of the microstructure, the first method is strongly advised.

IX. The usefulness of these analysis

What can we do with all these analysis? First of all it provides us with a way to say something about

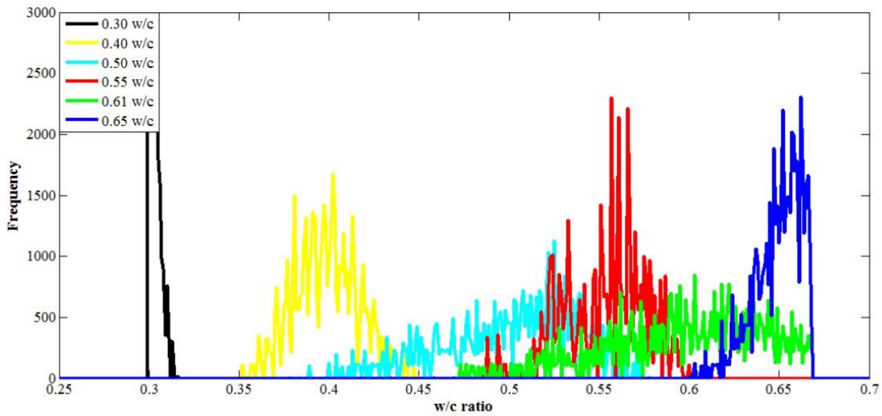

Figure 8: Distribution of the w/c ratios for the samples mentioned in the legend based on analyzing only 3 images

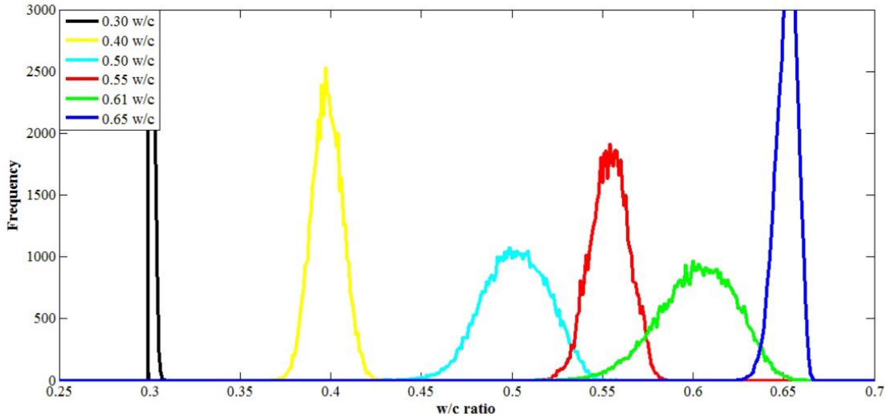

Figure 9: Distribution of the w/c ratios for the samples mentioned in the legend based on analyzing 9 images per thin section of a specific w/c ratio mentioned in the legend

the local variation in capillary porosity. Hence, all properties that are influenced by this local variation of capillary porosity can now be studied at a microstructure level in more detail through the use of optical microscopy. Obviously it would be nice to be able to link the variation in strength to the variation in local microstructure. However, much more interesting would be the characterization of the local microstructure in terms of transport properties. Can we relate the local variation to values and distribution of transport parameters so we can start to understand and model the ingress patterns that we see with for example Rapid Chloride Migration or carbonation, see e.g. Figure 11. We know these ingress patterns are not straight lines into the material as our current models assume. However, is it possible with this new local variability to predict the local variation in ingress that we see at a higher scale. If so, then we are making progress if only by understanding that our concrete material is so much more than just a recipe with emphasis on the water-cement ratio. A possible way to do so could be by using the width of the w/c ratio distribution, or rather the variation in the capillary porosity as a measure for the variation in the ingress rate of substances. This would open up possibilities to calculate ingress based on probability distributions rather than as a pure deterministic diffusion alone.

X. Conclusion

This article has taken a closer look at the determination of the w/c ratio through the analysis of capillary porosity by using image analysis. It has shown that accurate analysis does not shown identical results in the w/c ratio even for standard reference samples with known w/c ratio. This is not because the techniques we use to determine the w/c ratios

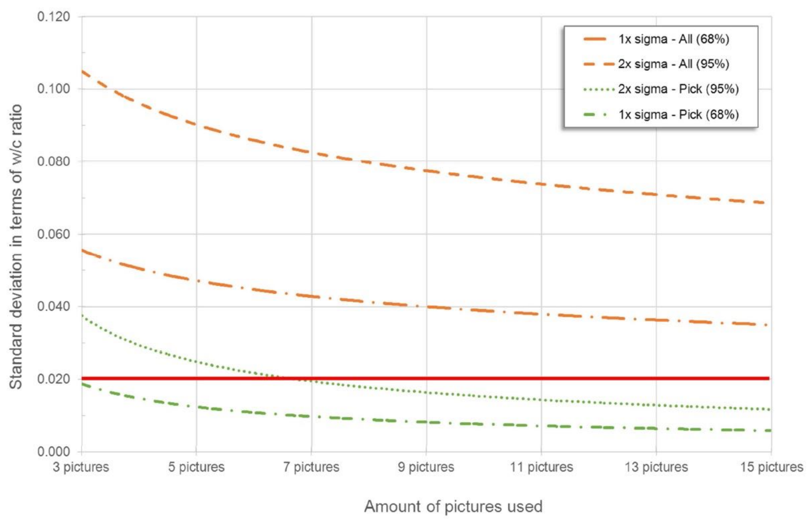

Figure 10: Plot to indicate the possible accuracy for determining w/c ratio. The lines marked 'All' are based on just averaging over the collected images. The lines marked 'Pick' use a random picking of the indicated number of pictures out of a set of 15 images for 50,000 times.

Figure 11: Ingress pattern of chlorides into a concrete sample after a Rapid Chloride Migration test

are not accurate enough, but much more because of the local variation in the microstructure.

For the first time the attention has shifted through this article from the analysis technique we use to what we actually observe in the microstructure. Hopefully this change in thought about the distribution in local microstructure variation will help to set off more research towards the capturing and modelling of these microstructure variation into consequences at a more structural level.

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