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MONITORING MICROSTRUCTURE EVOLUTION USING LOW COST DIGITAL MICROSCOPE

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Abstract: Development of sustainable construction material relies on our ability to monitor the impact of change in material configuration on resulting microstructure and strength development. The wide ranging active and passive techniques available for microstructure characterization estimates composition at discrete time periods. This is because either the sample is destroyed during the test or is surface modified rendering it unsuitable for future use. Present study adopts optical microscope to monitor microstructure development in ordinary and pozzolana blended mortar samples. The non-intrusive character and ease of sample preparation allows for progressive mapping of target areas thus capturing information on microstructure evolution. To assess variation in microstructure evaluations, three different mortar compositions namely with ordinary portland cement (OPC), pozzolana portland cement (PPC) and OPC with fly ash C is considered. Cement to sand ratio for all compositions is chosen as 1:3 and constant water binder ratio of 0.48 is maintained. Samples are then subjected to curing and taken out at regular interval, air dried and imaged. In order to ensure reliable results, all images are taken at constant height and light intensity. The obtained images are then analyzed through image processing techniques for evolution of pixel intensities and its correlationship with microstructural features of the material.

1 INTRODUCTION

Hydration reaction and resulting microstructure development in concrete is an intricate process to measure. Established literature identifies Calcium hydroxide (CH) and Calcium silicate hydrate (CSH) as an important constituent making bulk of microstructure. Compositions of these along with other compounds and their extent, is influenced by the chemical composition of reacting medium namely cement and water. Addition of supplementary cementitious materials as reacting medium alters this composition thus impacting microstructure development. It is now well established that the reactive supplementary materials referred as pozzolanic materials, in presence of water can consume CH developed as a result of cement hydration to produce low density CSH [1-2]. This implies in comparison with ordinary concrete, pozzolana blended concrete shall have lower proportion of CH. Thus, the above premise have become the cornerstone for judging the pozzolanic capacity of the material. Several experimental techniques including X-Ray Diffraction (XRD), Thermo-gravimetry/Differential Thermal Analysis (TG/DTA),Scanning Electron Microscopy (SEM) imaging are thus used to generate inferences on the extent of CH formation in the material. It is reported that the estimate of CH as evaluated by XRD is different from that of chemical analysis with later method giving a conservative estimates. In comparison, estimates generated by XRD matches well with those by TG/DTA [3]. Thus, XRD and TG/DTA can be used to estimate the net pozzolanic activity based on the proportion of CH available. SEM imaging at the other end offers evidence on breakdown/conversion of added pozzolanic materials thus justifying the reactive characteristics of the material. It is also reported that the pozzolanic activity of the material is sensitive to various factors including alkalinity of pore water, chemical composition of pozzolanic materials, pH etc. As the local environmental conditions can strongly influence pozzolanic activity, the observed behaviour at a particular time instant may not reveal the complete reaction dynamics of the material [4-5]. Thus, any approach that permits non-destructive monitoring of microstructure shall be used in conjunction with the proven microstructure analysis techniques to develop complete understanding of the reaction process.

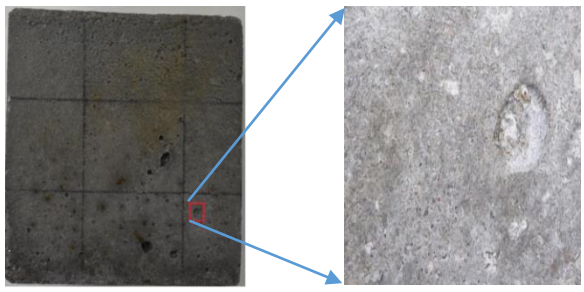
Optical microscopy (OM) on account of its simplicity and ability to capture microstructure without any pre-treatment can be considered as a favoured choice to study the evolution process. The observations by optical microscopy can then be correlated with those of XRD/TG-DTA [6]. The present study focusses on usage of optical microscopy to image cement microstructure. The obtained images are then converted to grey scale and processed through image processing tools to statistically analyse the distribution of intensities. The comparison of these intensities with the line histogram for sections passing through identified microstructure products are then used to predict the threshold values that enables discretization of intensity histogram to correlate the range with that of microstructure products. Thus, optical microscopy would offer the dual benefit of estimating the proportion of microstructure products developed together with the visual interpretation. The progressive mapping of set target area along with the analysis described thus enables tracking microstructure evolution process for the said material.

2 EXPERIMENTAL PROGRAM

Mortar sample with a binder filler ratio of 1:3

is prepared with Ordinary Portland Cement (OPC), Pozzolana Portland Cement (PPC) and 30 % OPC replaced with fly ash. Water binder ratio for all three set of samples is maintained constant at 0.48. As material ingredients, OPC/PPC 53 grade is chosen as cement and natural river sand passing through 2.36 mm sieve is used as fine aggregate. Mortar samples moulded into cubes with internal dimensions 70.6 mm × 70.6 mm × 70.6 mm is prepared with hand mixing conforming to IS: 4031(6)-1988 [7]. All samples are demoulded after 24 hours and cured for 56 days.

In order to investigate microstructure development, optical microscope is used to capture images at every 3 days till 56 days. To ensure minimum distraction due to external factors like lighting intensity, resolution etc, lighting, pixel intensity and focal length are kept constant for all images. The resolution chosen is such that each image represents a matrix of 640 x 480 pixels physically mapping an area of 90.2 mm^2. Thus, each pixel represents a physical dimension of 17.3µm and is considered as resolution of the image. The obtained resolution though coarser than other



(a) (b)

Figure.1: (a) Identification of target area on the sample (b) Microscopic image of the target area

imaging techniques used, is considered adequate to analyse formation of calcium hydroxide (CH), ettringite and large size pores [8]. Also, tracking of variation in formation, consumption of large size pores, reacted and un-reacted products can also be traced.

In order to investigate microstructure development, each cube face is divided into nine parts. The area of interest is then identified and the target area to be imaged is chosen. Identification of target area and the resulting microscopic image for a sample is as shown in Figure-1(a) and (b).

3. IMAGE PROCESSING:

The images obtained through OM represents a digital image and can be converted to grey scale image for further analysis. The grey scale image represents information available at a pixel in terms of an intensity value (correlated with the reflectance of the material) ranging from 0 to 255. Here, zero corresponds to black indicating no light is reflected and 255 indicating pure white that is complete reflection. As the reflectance characteristics of the material is influenced by shape, size and chemical composition, the obtained intensity values would reflect the resultant effect of all there parameters.

The standard algorithm is then used to convert captured colour images to its gray

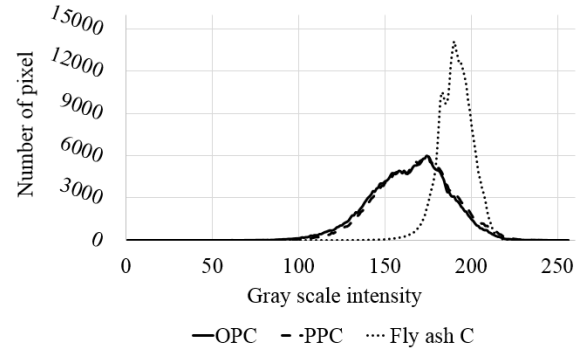


Figure.2 Intensity histogram of reference materials in dry state

scale. As the information available at grey scale is represented in terms of intensity values, it is now possible to aggregate/dispense pixels with desired

intensity values. Thus, identification of intensity range and the associated microstructure product would permit assessment of distribution of the said product in its target area. In order to predict average trend in microstructure development over target area, total number of pixels corresponding to each intensity values can be evaluated. This plot called intensity histogram represents intensity values on X-axis and number of pixels on Y-axis is shown in Figure.2

Plotted histogram represents the intensity values of reference materials in dry state. It can be observed from Figure.2 that OPC and PPC samples in its dry state represents an almost identical histogram whereas the Fly ash C considered has a very narrow histogram and is shifted towards right. Thus, it indicates OPC and PPC samples to have similar colour and particle size distribution whereas Fly ash C has a narrow band of particle size distribution and is also few shades lighter than OPC/PPC. Thus, the image histogram can be used to qualitatively interpret the microstructural variations in a given sample.

In order to study microstructure evolution, the said process of capturing an image and analysing intensity histogram of target area over subsequent time periods reflect the net change in microstructure during the period of observation. Figure.3 represents a similar case with histogram comparison for OPC sample as observed at 7, 28 and 56 days.

As observed at 7 days, intensity histogram represents an approximate normal distribution with mean value just under 200. At 28 and 56 days, the histogram gets skewed and shift to right. It can be observed through Figure.3 that as time progress the histogram shifts to right and also number of pixels representing peak intensity drops. Intensity regions contributing to consumption and formation of pixels can be

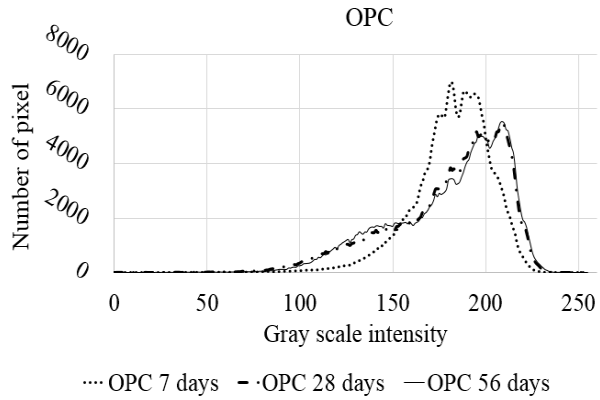
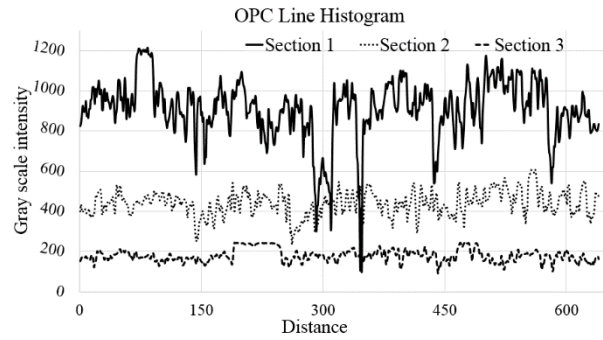


Figure.3 Evolution of intensity histogram for OPC samples

(a)



(b)

Figure.4: (a) OM image with identifiable microstructure features (b) Line histogram of indicated sections

easily observed for the period from 7 to 28 days. This change in histogram represents an underlying change in chemical compositions and physical characteristics, thus capturing the microstructure compositions. Also lack of activity in the period from 28 to 56 days is in

line with the hydration behaviour discussed in literature [9-11].

In addition to image histogram, a line histogram is also exploited to further understand distribution of intensities and its correlation with the identifiable microstructure products developed.

Figure.4a represents an OM image with easily identifiable features. Three distinct sections with first one intersecting CH, unfilled and partly filled voids, second passing through the bulk matrix and the third one intersecting CH and bulk of matrix. The intensity histogram for the chosen sections are shown in Figure 4b. To ensure clarity in representation, respective histograms for section 2 and 3 are shifted by a factor of 2.5 and 5. The comparison reveals that the line histogram clearly distinguishes presence of CH and unfilled voids as in section 1-1 and 3-3. However, partly filled voids are difficult to distinguish from rest of cement matrix. Further, the bulk of cement matrix represents a wide range of intensity values namely 120-190, the range that was dominant for histogram in dry state as discussed in Figure-2. Thus identifications of products in this range is a difficult task. This restricts the line histogram analysis to predict threshold values only for identification of CH.

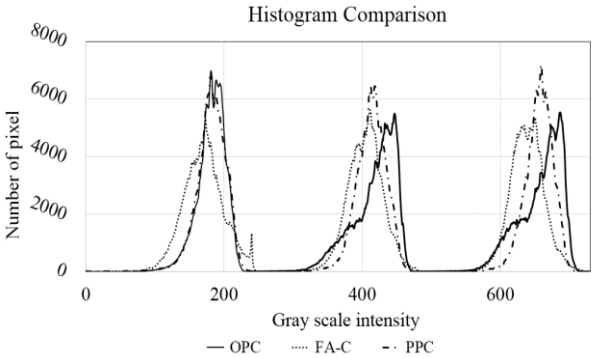


Figure 5. Histogram comparison for OPC, PPC and OPC-Flyash samples

Thus, the procedure outlined is used to study evolution of microstructural images, intensity histograms and the development of CH. Line histogram even with a limited capabilities of computing threshold values corresponding to CH thus plays a key enabler in evaluating the pozzolanic characteristics of the materials.

4 RESULTS AND DISCUSSIONS

The procedure discussed above is used to study time evolution of microstructure for OPC, PPC and OPC+FA samples. This time evolution is first analysed through intensity histogram which in second stage is discretized into two parts namely CH and rest of matrix. This allows one to understand average change in microstructure and distribution of CH, a constituent whose composition would point to pozzolanic behaviour of the materials.

Figure.5 represents comparison of intensity histogram for all samples considered at 7, 28 and 56 days. For ease of representation and understanding, intensity histogram for 28 and 56 days are shifted rightwards by 200 and 400 respectively. At 7 days, histogram for OPC and PPC samples are almost identical whereas fly ash blended samples exhibit lower peak and shift towards left indicating lower reactivity. For fly ash based sample, histogram shifts towards left could be attributed to lower cement content, and lower reactivity of fly ash added. At 28 days, activity in flyash mixed samples increases and is now comparable with that of PPC samples. During the said period, histogram for OPC samples exhibit shift towards right indicating higher generation of CH. The proportion of CH generated in each sample at a given time period can be computed based on the threshold values as discussed in last section. Further, at 56 days, histogram movement is seen only in PPC and fly ash blended samples. In contrast to 28 days,

histogram for fly ash once again shifts to left indicating part consumption of CH as witnessed at 28 days.

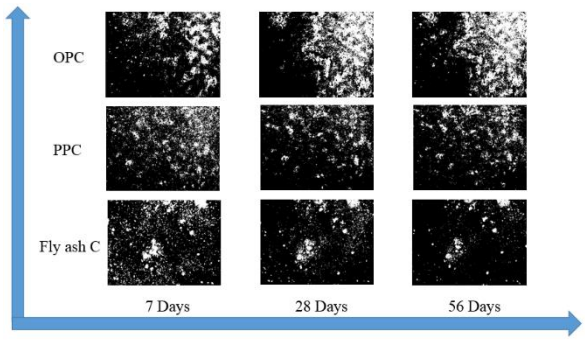


Figure.6 Distribution of calcium hydroxide

The obtained histograms at different time periods along with intensity values corresponding to CH can now be used to estimate the formation of CH at different time periods. Based on the threshold values corresponding to CH as obtained in last section, intensity histogram for samples is discretized into two parts namely CH that represents intensity values exceeding threshold and rest of cement matrix that treats all intensity values lower than threshold as equal to zero. Comparison of CH developed for samples at the considered time period is shown in Figure 6. As shown in Figure.6, it can be observed that both formation and distribution of CH is higher for PPC and OPC-flyash samples at 7 days. However, distribution of CH is more uniform for fly ash based samples as compared to PPC and OPC samples. Further at 28 days, though the density of CH increases for OPC, the proportion exhibits decrease for PPC and OPC+fly ash samples. At 56 days analogous to behaviour observed in Figure 5, proportion of CH decreases only for fly ash samples whereas the other two exhibits near stagnant response. This behaviour for OPC and PPC samples is well acknowledge and reported in literature [12-13].

Thus the evolution of CH studied over three different samples defines the effectiveness of OM in exhibiting the nature and extent of pozzolanic behaviour by supplementary materials.

5 CONCLUSIONS

Optical microscope has been used to study microstructure evolution for the samples considered. As PPC and Flyash is well acknowledged for its pozzolanic action, the same is demonstrated through usage of optical microscope and the image processing techniques. The microstructural images captured and its intensity histogram are correlated with the physical behaviour reported. Further, the estimation of threshold intensity values corresponding to CH and its monitoring and comparison amongst samples at different time instants enables estimation of pozzolanic activity of the material. Thus, the proposed method of OP coupled with available image processing tools can be tailored to monitor progress in microstructure evolution for blended concrete samples. To compute threshold values corresponding to micro structure products (say CH) validating intensity histogram and its assessment, the reference samples at discrete time periods can be analysed through established techniques like XRD, TG/DTA and the results compared.

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