CHARACTERISATION OF PORE STRUCTURE OF HARDENED CEMENT PASTE IN CONCRETE USING BSE IMAGE ANALYSIS AND MERCURY INTRUSION POROSIMETRY

by

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cement and
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DEDICATED

TO

MY PARENTS

CERTIFICATE

This is to certify that the thesis titled "**CHARACTERISATION OF PORE STRUCTURE OF HARDENED CEMENT PASTE IN CONCRETE USING BSE IMAGE ANALYSIS AND MERCURY INTRUSION POROSIMETRY**" being submitted by **Mr. Manish Jain** to the Indian Institute of Technology Delhi, India, for the award of the degree of "**Doctor of Philosophy in Civil Engineering**" is a record of the original bonafide research work carried out by him under our guidance and supervision.

To the best of our knowledge, the thesis has reached the requisite standard. The material presented in this thesis has not been submitted in part or full to any other University or Institute for award of any degree/diploma.

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ABSTRACT

In the present study, the pore structure characteristics of the *same cement pastes* in various concretes, based on Backscatter Electron (BSE) Image Analysis and Mercury Intrusion Porosimetry (MIP), are compared. The fractions of the capillary porosity and the total porosity (capillary + gel) of the hardened cement paste (hcp) in concrete, which are measured by the two techniques, are also assessed.

The theoretical estimates of the capillary porosity and total porosity of the hcp in concrete, are based on Powers Model. For this purpose, the degree of hydration of cement in the hcp must be known. The degree of hydration of cement is obtained directly from the volume fraction of unhydrated cement in the hcp, which is determined by Image Analysis technique.

Concretes with water-cement ratios, 0.40, 0.50, and 0.60 are tested for pore structure characteristics and compressive strength at the age of 7, 28, and 180 days. Some concretes are wet cured continuously till the age at test, while others are wet cured for only 7 days and then stored in air till test. Concretes are made with normal-weight aggregates and ordinary portland cement (OPC). There are no admixtures used. Concrete made with portland pozzolana cement, w/c ratio equal to 0.50 and wet cured for 7 days, has also been tested at the age of 28 and 180 days for pore structure characteristics and compressive strength.

For all concrete mixes, the aggregate content is kept constant at 65% by volume; and the maximum size of aggregate and its grading are kept the same. The influence of aggregate content and grading on the pore structure characteristics is not studied.

The samples for pore structure characterisation are selected using the 'random systematic' sampling strategy. For each concrete, twenty BSE images are acquired from three polished sections, 25x30 mm size. The digitized BSE images are acquired at 512x512 pixels, and the resolution achieved is 0.199 μm at 1000x magnification. For MIP test six samples of each concrete, 6x8x25 mm size, and six aggregate samples are tested to obtain the average intrusion curves.

The pore structure characteristics evaluated are: porosity, pore specific surface area, mean pore diameter, pore size distribution, mean distribution pore radius, pore shape factor and threshold diameter.

Most of the porosity tallied by Image Analysis is in the pore size range 0.5 μm to 10 μm, whereas essentially all of the porosity tallied by MIP is represented as occurring in pore size range 0.002 μm to 0.20 μm.

The porosity of the hcp in concrete tallied by Image Analysis is found to be half of the porosity measured by MIP. Hence, Image Analysis reveals that a significant fraction of the porosity measured by MIP is due to the pores in the size range 0.5 μm to 10 μm. MIP measurements on the *same cement pastes* record almost all porosity as though it existed in sizes below about 0.20 μm, i.e. below the threshold or percolating diameter of the bulk pore system.

It is found that the porosity of the hcp in concrete based on Image Analysis is 0.55 to 0.65 times the estimated capillary porosity, whereas the porosity of the hcp tallied by MIP is 1.1 to 1.3 times the estimated capillary porosity of the cement paste based on Powers Model.

The MIP method measures only a fraction of the estimated total porosity of the cement paste based on Powers Model. The fraction of the total porosity, measured by MIP method, is smaller in cement pastes which are more dense, i.e. pastes with lower total porosity. It has been shown that hardened cement pastes with a lower total porosity have a greater fraction of gel porosity resulting in more segmented capillary pores. Hence, mercury intrusion is restricted in dense pastes and a smaller fraction of the total porosity is recorded by MIP test.

It is found that the mean pore diameter and the pore specific surface area increase with an increase in the porosity of the hcp in concrete. The pore shape factors, however, do not change with the porosity. The threshold diameter of an interconnected capillary network is found to lie in the range 0.10 μm to 0.20 μm for the hcp in various concretes. It is observed that the threshold diameter is smaller for concretes of low porosity which have a finer network of pores in the hcp.

It is shown that the mean pore diameter and the mean distribution pore radius, in fact, represent the same pore structure characteristic, although these are determined by two different approaches.

It is found that the experimental data in the present study fits well the strength –porosity relation proposed by Powers. Strength – porosity relation considering pore size distribution has been obtained based on Atzeni's Model.

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