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Effect of Sample Location and Curing on Pore Volume and Threshold Diameter of Cement Paste with and without Slag

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ABSTRACT

Slag is a by-product of the steel making industry. The re-use of industrial by-products in concrete applications to partially replace the cement is advantageous in that it reduces the amounts of cement used and also reduces the materials sent to landfill. In this paper, the porosity and threshold diameter of cement paste with and without slag is investigated. Specimens were subjected to elevated and normal temperature curing at different relative humidities. The results show that the inclusion of slag is beneficial when pastes are subjected to some initial moist curing. The influence of sample location within a paste specimen on porosity and pore size distribution is also considered. The intruded pore volume and threshold diameter vary depending upon the location within a cube relative to the casting position. Substantial difference in intruded pore volume exists between samples taken from the top (trowelled) face and the bottom face of paste cube of 100 mm in size regardless of the presence of slag. Samples taken from the middle of the side face show intermediate values compared with samples taken from the top and bottom of the cube.

INTRODUCTION

Water absorption by shallow immersion is generally measured by drying a specimen to constant mass, immersing it in water for a specific time and measuring its increase in mass as a percentage of the dry mass. Capillary water absorption of concrete, however, is the phenomenon by which water is absorbed into concrete by capillary action. It involves placing a sample with

one surface just in contact with water and the mass or height of water absorbed by capillary action is measured at different time intervals. The fineness of the capillary pores in concrete causes absorption of water by capillary action, hence a measure of the rate of absorption provide a useful indication of the pore structure of concrete. If water is absorbed rapidly the capillaries will be large and if the absorption is slow then the capillaries will be small. This phenomenon of water absorption by capillary action has been reported in the literature [RILEM 1980, Fagerlund 1981, Ho and Lewis, 1987, Hall 1989, McCarter et al 1996, Gopalan 1996, Khatib and Clay 2004, Khatib et al 2009].

The partial replacement of cement with cement replacement materials influences the properties of fresh and hardened concrete. Such hardened properties include water absorption and capillary water absorption [Kelham 1988, Parott 1992, Khatib and Mangat 1995].

The curing regime to which a concrete structure is subjected influences the absorption characteristics of the concrete. Moreover the location from which a sample within the concrete matrix is taken has a noticeable influence on the absorption. For example, a sample taken from the trowelled (top) surface of concrete will exhibit different absorption values of sample taken from the side surface (the side which is perpendicular to the trowelled face) or of sample taken from the bottom face of concrete [Senbetta and Scholar 1981, Parrot 1992, Khatib and Mangat 1995].

The porosity and pore size distribution of the binder phase (i.e. paste) in concrete have an influence on the absorption characteristics of concrete. The properties of the paste are also influenced by the paste location and curing regime. In this paper, the influence of initial curing and the influence of sample locations relative to casting position on porosity and pore size distribution of paste are considered. Samples were taken from different locations of the exposed surfaces (top, side and bottom) of paste cubes. Two pastes were prepared, the control paste (i.e. no cement replacement) and a paste where 40% of the cement was replaced with ground granulated blast furnace slag were considered. Specimens were subjected to curing at 45°C and 20°C and different relative humidities. High temperature curing (i.e. 45°C) was chosen to simulate concrete in hot environment. Similar work was conducted on pastes containing fly ash and silica fume and were reported in a previous investigation [Khatib and Mangat 2003].

EXPERMENTAL PROGRAMME

Mix Constituents and Materials

The cement used was Portland cement (PC) and the slag was ground granulated blastfurnace slag. Composition of PC and Slag is given in Table 1. The superplasticiser (SP) used was Naphthalene Sulphonate based liquid and conformed to Type F and G of ASTM C 494.

Two different pastes, representative of the hydrated binder phase of concrete mixes CM and SM were prepared to study their porosity and pore size. The control paste CP contained Portland cement (PC) and in the slag paste SP, the cement was partially replaced by 40% slag. Superplasticiser was added to both pastes. The water to binder ratio and the superplasticiser dosage were respectively 0.45 and 1.2% by mass of binder. Superplasticiser was added in order

to simulate the binder phase in the concrete mixes reported in a previous investigation [Khatib and Mangat 2003).

Curing

Paste cubes of 100mm in size were cast in steel moulds. Three different curing regimes were adopted. High temperature curing was chosen to simulate concrete in hot regions. The total curing period was 28 days. Details of the curing methods of concrete specimens are described below and summarised in Table 2.

		OPC	Slag
SiO ₂	%*	20.4	36.0
Al ₂ O ₃	%	4.89	9.0
Fe ₂ O ₃	%	3.18	1.0
CaO	%	64.02	43.0
MgO	%	2.56	7.0
SO ₃	%	2.85	_**
Na ₂ O	%	0.07	1.0
K ₂ O	%	0.53	-
Insoluble Residue	%	0.37	-
Loss on Ignition	%	0.98	-
Free Lime	%	2.37	-
Specific Surface Area	m²/kg	367.76	350

Table 1: Composition of the Cement and Slag

*Percentage by weight **Not carried out

Table 2: Details of paste mixes

	Paste Pro			
Paste	OPC	Slag	S*	water/binder
СР	100	0	1.2	0.45
SP	60	40	1.2	0.45

* superplasticiser, % by mass of binder

Sampling

After the predetermined curing period of 28 days, samples ranging from 0.8 to 2.0 g were taken from the middle of the side surface (S), the top trowelled surface (T), bottom surface (B) of the cubes. Further detail about the sampling procedure is given elsewhere [Khatib and Mangat 2003].

Drying of Specimens

After the sampling procedure, samples were dried by keeping them in the environmental chamber at 45°C and 25%RH for 4 hours to avoid sudden change in temperature, followed by oven drying to constant mass at 100 ± 5 °C. The effect of this drying method on the possible alterations of pore structure was not investigated. The total period of drying was about 24 hours for the paste samples. The specimens were then cooled in an air-tight container at 20 ± 1 °C prior to testing.

	After casting & before demoulding	After demoulding		
Curing	Up to 24 hours	After 24 hours and up to 14 days	After 14 days and up to 28 days	
A45	Exposed to air at 45°C & 25% RH	Exposed to air at 45°C & 25% RH	Exposed to air at 45°C & 25% RH	
MA45	Covered with plastic sheeting and wet burlap at 45°C&25%RH	Covered with plastic sheeting and wet burlap at 45°C&25%RH	Exposed to air at 45°C & 25% RH	
MA20	Covered with plastic sheeting and wet burlap at 20°C&55%RH	Covered with plastic sheeting and wet burlap at 20°C&55%RH	Exposed to air at 20°C&55% RH	

Table 3: Details of the curing methods

Testing

The porosity and pore size distribution of the paste samples were determined using the mercury intrusion porosimetry technique. The instrument had a pressure capacity of 200 MPa. The data were computed by using the Washburn equation as follows:

 $D = -\frac{4\delta\cos\theta}{P}$, where D is the pore diameter, P is the applied pressure, δ is the surface

tension which was taken as 484 dynes/cm and θ is the contact angle which was taken as 142°. A typical cumulative pore distribution plot is reported elsewhere [Khatib et al 2007].

RESULTS AND DISCUSSION

The intruded pore volume of the control paste (CP) and the slag paste (SP) cured in air at 45° C (A45) for sample taken from different locations of a cube specimen (top, side and bottom) is shown in Figures 1 and 2 respectively. The threshold diameter for the same pastes and same locations under A45 curing are respectively shown in Figures 3 and 4. The threshold diameter is

the diameter on the cumulative pore volume curve before which the pore volume rises sharply. Further details on the determination of the threshold diameter are reported elsewhere [Khatib and Wild 1996].

The intruded pore volume is higher for samples taken from the top part of the cube compared with samples taken from the bottom of the cube. Samples taken from the side show intermediate values between those taken from the top and bottom of the cube specimen. A difference in intruded pore volume between a sample taken from the top of the cube and a sample taken from the bottom part of the cube can be as large as 50%. The trend in the threshold diameter is similar to that of the intruded pore volume.

The 40% slag paste, under dry curing (i.e. air curing-A45), exhibits slightly larger pore volume and larger threshold diameter than the control paste, except for samples taken from the bottom part of the cube where lower value of pore volume is obtained. The slightly larger intruded pore volume in the slag paste is due to the rapid loss of water from the specimens under such dry curing. This water will not be available for the slag particles to hydrate further. A large threshold diameter indicates a coarser pore structure.



Fig. 1. Pore volume of control paste cured in air at $45^{\circ}C\&25\%RH$ (A45) and taken from various locations



Fig. 2. Pore volume of the slag paste cured in air at $45^{\circ}C\&25\%$ RH (A45) and taken from various locations

Figures 5 and 6 show respectively the intruded pore volume and threshold diameter for the control paste and the slag paste for samples subjected to moist/air curing at 45°C and 20°C and taken from the side surface. The partial replacement of cement with slag decreases the intruded pore volume and the threshold diameter for specimens subjected to initial moist curing (i.e. MA45 & MA20). Under these moist conditions, the threshold diameter also decreases in the presence of slag, indicating a finer pore structure.



Fig. 3. Threshold diameter of the control paste cured in air at 45°C&25%RH (A45) and taken from various locations



Fig. 4. Threshold diameter of the slag paste cured in air at 45°C&25%RH (A45) and taken from various locations

Comparing these results in Figures 5 and 6 with those reported in another investigation [Khatib et al 2009], it should be noted, however, that under MA20 curing the water absorption by shallow immersion and capillary action of concrete cube, having the same binder compositions, were larger in the slag mix due to crazing. This trend is not the same in the case of the intruded pore volume of paste, which is partly due to the size of sample in each case. A relatively large concrete specimen was tested to determine the water absorption, whereas a small paste sample was used in the present investigation to determine the pore volume and pore size distribution. The other reason might be that the cracks which were observed in the concrete specimens occurred in the interfacial zone of the binder and the aggregates. This does not arise in the case of pastes.



Fig. 5. Pore volume of pastes with and without slag subjected to moist/air curing at 45°C (MA45) and 20°C (MA20) taken from the side location



Fig. 6. Threshold diameter of pastes with and without slag subjected to moist/air curing at 45°C (MA45) and 20°C (MA20) taken from the side location

CONCLUSIONS

The intruded pore volume of pastes containing slag is lower that that of paste without slag when subjected to some initial moist curing. Under dry curing, the presence of slag slightly increases the intruded pore volume and the threshold diameter of paste. This may be due to the lack of water available for the late hydration of slag particles. Substantial difference in intruded pore volume of paste exists between samples taken from the top part and the bottom part of specimens owing to the bleeding effect. This difference can be as large as 50%. Pastes taken from the side face show intermediate values between those taken from the top and the bottom of the cube. This difference is exaggerated when slag is present in the paste.

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