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THE MICROSTRUCTURE OF DRY CONCRETE PRODUCTS

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ABSTRACT

In North America, the use of dry concrete products has significantly expanded over the past decades. Despite the growing interest in these materials, little is known of their microstructure. In order to obtain more information on the subject, the microstructure of various dry concretes was investigated by means of optical microscopy, scanning electron microscopy, and mercury intrusion porosimetry, and compared to that of ordinary concretes. The results indicate that the cement paste fraction of most dry concretes is generally much more heterogeneous than that of ordinary concretes. High-energy mixing and the use of mineral additives, such as silica fume and fly ash, were found to significantly enhance the homogeneity of the cement paste. Considerable improvements of the engineering properties of dry concretes can be achieved by using these additives.

Introduction

Nowadays, dry concrete technology is used to produce a broad range of concrete products designed for various applications. Although these products can sometimes differ in their constituent proportions and fabrication methods, they all share important common features. Generally speaking, dry concrete products can be defined as concretes with an initial consistency significantly higher than that of usual concrete mixtures. The dry concrete mixture must be stiff enough to allow an effective consolidation by rollers (as for roller-compacted concrete), or to allow immediate demoulding (as for most pre-cast elements), but wet enough to allow an adequate distribution of the paste throughout the mass of the element during the mixing and vibration operations. The high consistency required is normally achieved by reducing the amount of water or by lowering the paste fraction of the mixture.

Previous investigations have indicated that dry concretes are generally less resistant to deicer salt scaling than ordinary concretes of similar composition [1, 2]. In many cases, this reduction of the durability of dry concretes can not be solely explained by the presence of numerous macroscopic voids resulting from the compaction operations. It can thus be hypothesized that they result, at least in part, from a higher degree of heterogeneity of the dry concrete cement paste.

A literature survey indicated that only limited information was available on the microstructure of dry concretes. Thus, an investigation specifically devoted to the internal structure of various dry concrete products, as well as to the parameters that can affect it, was carried out. The objective was to help to find ways to improve the engineering properties of dry concretes. An overview of the main results obtained is presented in this paper, and ways of improving the microstructure of dry concretes are discussed.

Test Program

The microstructures of two different types of industrially-made dry concretes, namely pre-cast paving blocks and roller-compacted concrete (RCC) pavements, were investigated in this research. To determine the extent to which it was possible to produce representative specimens of dry concretes under controlled laboratory conditions, a series of dry concretes made in the laboratory was also studied. Since previous test results had indicated that the frost durability of dry concretes could be significantly enhanced by the use of mineral additives [3, 4], three different binders (a CSA type 10 cement, a blended silica fume cement, and a CSA type 10 cement + a class F fly ash) were used in the preparation of each type of dry concrete. In addition to the usual characterization tests (compressive strength and density measurements), the microstructure of these various dry concretes was investigated by means of optical microscopy, scanning electron microscopy, and mercury intrusion porosimetry.

Materials and Casting Procedures

Materials. As previously mentioned, the influence of three different binders was evaluated. For each type of concrete, one mixture was produced with an ordinary Portland cement (CSA type 10), another with a blended silica fume cement (containing approximately 8% of silica fume by mass of cement), and a third with the same ordinary Portland cement and a class F fly ash. The amount of fly ash was equal to 20% of the Portland cement mass. Fly ash was used, not as partial cement replacement, but as partial sand replacement. The chemical composition of the two binders and of the fly ash are given in Table 1. A lignosulfonate based water-reducer, acting also as a retarder, was added to all mixtures. No superplasticizer was used in the production of any of the nine mixtures prepared in this study.

For all mixtures, a natural granitic sand, having a fineness modulus of 2.5 and a water absorption after 24 hours of 0.6% was used. A very dense crushed limestone, containing approximately 20% of dolomite particles, was used as coarse aggregate. Pertinent data for the aggregates are given in Table 2.

For the laboratory and for the RCC mixtures, the binder content was equal to 300 kg/m^3, and the water to binder ratio was fixed at 0.35. The water content of these mixtures corresponds roughly to the optimum water content as given by the Moisture Density Test (ASTM D1557 - Modified Proctor) originally developed for soils. To facilitate demoulding operations and to

TABLE 1

Chemical Composition of the Binders

Constituents	Cements		Class F fly ash
	CSA type 10 (%)	BlendedSF cement (%)	(%)
Slilcon dioxide	21.0	27.3	40.0
Aluminium oxide	4.1	3.9	20.0
Ferric oxide	3.1	3.0	26.0
Total calcium oxide	63.5	57.6	3.0
Free calcium oxide	0.7	0.5	—
Sulphur trioxide	3.4	3.4	1.0
Magnesium oxide	2.1	2.3	0.8
Alkalies (eq. Na_2O)	0.9	0.8	—
Loss on ignition	2.2	1.5	4.0
Insoluble residue	0.7	1.3	—
Bogue's Calculations			
C_3S	54.8	—	—
C_2S	18.9	—	—
C_3A	6.6	—	—
C_4AF	9.4	—	—

obtain a surface finish representative of that commonly found in the industry, the paving block mixture characteristics had to be modified. In this case, the binder content was equal to 422 kg/m^3 and the water to binder ratio was reduced to 0.28. The composition of all mixtures is given in Table 3.

TABLE 2

Aggregate Physical Properties

Physical Properties	Fine aggregate	Coarse aggregate
Density	2.70	2.73
Absorption (%)	0.60	0.66
Fin. Mod.	2.45	—

TABLE 3

Mixture Characteristics

Mixture	Binder kg/m³	Water kg/m³	Fine aggr. kg/m³	Coarse aggr. kg/m³	WRA* ml/m³
Pre-cast concrete paving blocks					
P-10	422	107	1064	794	1350
P-SF	422	105	1064	794	1350
P-CF	422 + 84	117	980	794	1350
Roller compacted concretes					
R-10	300	105	1060	1000	960
R-SF	300	105	1060	1000	960
R-CF	300 + 60	105	1000	1000	960
Laboratory concretes					
L-10	300	105	1060	1000	960
L-SF	300	105	1060	1000	960
L-CF	300 + 60	105	1000	1000	960

* Water reducing admixture

Casting procedures. All paving block mixtures were produced in a counter-current pan mixer. Paving blocks were cast using a Knauer multi-layer machine. Three layers of 60 paving blocks were produced from each mixture. They were air cured for a period of approximately 16 hours, and, after this initial period, the top layer from each mixture was packed and wrapped with cellophane on two skids and then shipped to the laboratory. After 28 days, the paving blocks were unwrapped and simply kept under laboratory conditions (i.e. 20°C and 50% R.H.) until testing. A detailed description of the casting procedure is given elsewhere [5].

All RCC mixtures were batched in a premix plant and transported to the construction site in dumptrucks. Concrete was placed on a compacted granular base with an ordinary asphalt paver, and later compacted with a dual steel-drum static roller. Final surface treatment was carried out by a rubber-tire roller. The test section was cured with a white membrane forming curing compound. Four weeks after casting, hardened pavement samples were obtained by cutting and kept at approximately 20° C and 50% R.H. Construction procedures are described in detail elsewhere [1, 3, 4].

All laboratory mixtures were prepared in a counter-current pan mixer. After a five-minute mixing period, a specific mass of concrete was placed in a 150 mm x 300 mm plastic cylinder and consolidated using a vibro-compacting machine. The mass of concrete was adjusted to obtain the same density than that of companion RCC mixtures. Cylinders were recovered with a wet burlap for the first seven days. At the end of this period, the wet burlap was removed and the samples were left in the plastic cylinders until 28 days. They were then unwrapped and kept at approximately 20° C and 50% R.H. until testing.

Experimental Procedures

After one year, cores from each of the nine mixtures were taken for testing. These concretes had been submitted to no other curing treatments than those described in the previous section. In order to minimize wall-effect related perturbations, all samples for the microstructural analyses were taken from the central portion of the concrete cylinders.

Compressive strength measurements were performed in accordance with applicable sections of CSA/CAN3-A23.2-14C and CSA/CAN3-A231.2. For each of the paving block and RCC mixtures, five drilled cores were tested. For the laboratory mixtures, compressive strength was performed on four 75 mm x 150 mm cored cylinders. Dry density and water absorption measurements were made in accordance with ASTM C 642. In all cases, at least two representative concrete samples were tested.

Pore size distributions were obtained by mercury intrusion porosimetry. The equipment was capable of a minimum intruding pressure of 2.6 kPa and a maximum of 414 MPa. Each tested sample had a mass of approximately 6 grams, and had been oven dried at 110°C for 48 hours. The contact angle assumed for all samples was 116°. After mercury intrusion, the paste content of each sample was determined by maleic acid extraction, so that the pore volume could be calculated on a unit mass of dry paste basis. Such a calculation is considered reasonable, given the very small porosity of the aggregates used in this study. Maleic acid extractions were conducted in accordance with the procedure published by Tabikh et al. [6] and by Marusin [7].

Optical observations of the internal structure of each dry concrete mixture were performed on fluorescent thin sections. This technique is very useful to evaluate the homogeneity of the cement paste fraction of concrete [8, 9]. Thin sections, about 10 μm thick, were cut and ground from samples that had been previously vacuum-impregnated with a fluorescent epoxy resin. Observations were made at magnifications of 20X to 100X using a laboratory microscope in both the polarization and the fluorescent mode. For each concrete mixture, at least three 45 mm x 60 mm thin sections were examined. Scanning electron microscope (SEM) observations were carried out on both fractured and polished sections. For each mixture, at least five representative samples were examined.

Test Results

Physical properties and compressive strength results. The compressive strength test results are summarized in Table 4. The results from each concrete mixture were often quite scattered, but the values in Table 4 nevertheless indicate that good compressive strengths (all except one higher than 40 MPa) were generally obtained. Overall, it seems that both the use of the blended silica fume cement and the addition of fly ash had positive effects. The beneficial influence of silica fume on the mechanical properties of dry concrete is in accordance with previously published results [10, 11].

Density data and water absorption results are also shown in Table 4. It is interesting to note that the mixtures were found to have water absorptions below 5%, despite the presence of numerous compaction voids.

The characteristics of the air-void systems of these concretes were determined in accordance with the Modified Point Count Method of ASTM C 457. The air content was usually found to be in the 6% to 10% range [3, 5]. The results of the measurements also indicate that the air content was extremely variable from one sample to another of the same mixture.

TABLE 4

Compressive Strength Results and Physical Properties

Mixture	Comp. strength (Mpa)	ASTM C642 test results		
		Dry density	Water absorption (%)	Permeable void vol. (%)
Pre-cast concrete paving blocks				
P-10	37	2.27	4.2	15.5
P-SF	62	—	—	—
P-cf	63	—	—	—
Roller-compacted concretes				
R-10	53	2.32	3.6	10.2
R-SF	61	2.40	3.1	11.3
R-CF	61	2.40	4.0	11.1
Laboratory concretes				
L-10	41	2.41	3.7	9.3
L-SF	60	2.41	3.6	9.2
L-CF	50	2.39	3.9	10.8

Pore size distributions. As indicated earlier, pore size distributions of the concretes were attempted by mercury porosimetry, with the intruded volume normalized to reflect cm^3/g of paste, rather than of concrete. The paste content was determined separately in each case by maleic acid extraction.

Initial attempts using a standard procedure involving 21 pressure steps and an equilibrium time allowed of 15 seconds at each step gave unsatisfactory and non-reproducible results. The difficulty appeared to be associated with progressive but irregular intrusion of mercury into some of the large compaction voids. To facilitate more complete intrusion at each step, the number of pressure steps was increased to 50, and the equilibrium time allowed at each step was doubled at 30 seconds. The results were still unsatisfactory, as indicated below.

A typical mercury pore size distribution (obtained by the modified procedure) is shown in Figure 1 for a pre-cast paving block sample. For the sake of comparison, the pore size distribution of an ordinary concrete mixture (i.e. produced with a superplasticizer in order to have a 80-mm slump) is also given in Figure 1. Although the two concretes were prepared at a similar water/cement ratio and cement content, and with the same aggregates, the paving block concrete was found to have much greater intrudable porosity. Both showed apparent pore spaces which appeared to reflect late (i.e. high pressure) intrusion pore spaces which should have been intruded at much lower pressures. The net result is a measurement showing a significantly finer pore size distribution than appears to exist.

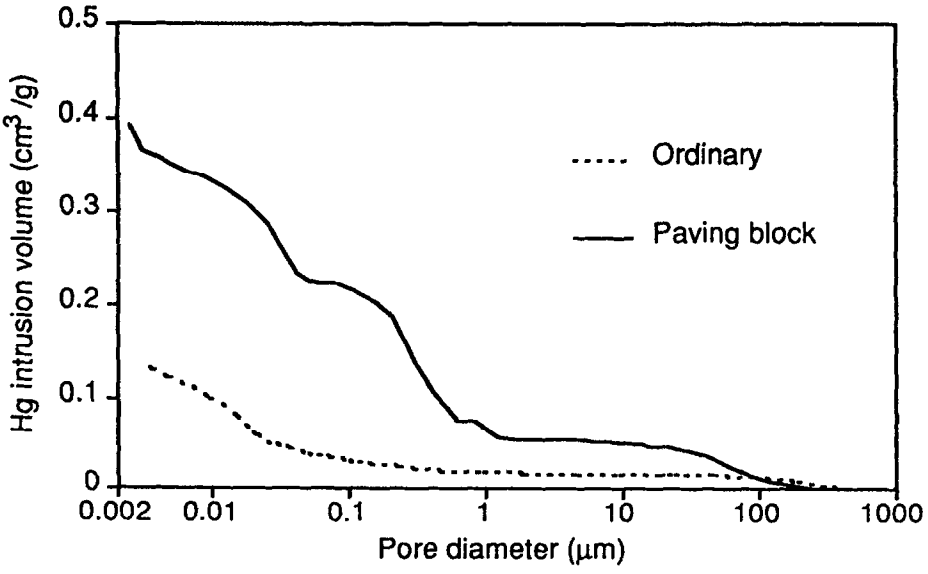

FIG. 1.
Typical pore size distributions of ordinary concrete and pre-cast paving block samples (type 10 cement).

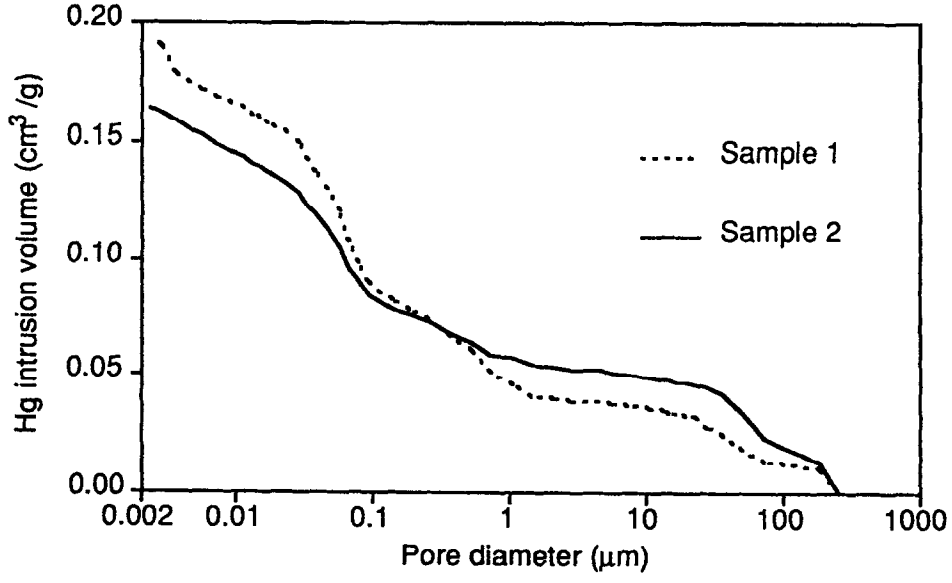

FIG. 2
Pore size distributions of two RCC samples from the same mixture (type 10 cement).

FIG. 3.
SEM micrograph: Typical zone of porous C-S-H in an RCC sample.

Furthermore, it appears that replicate pore size distribution curves for separate samples of the same concrete were not consistent. Figure 2 provides a typical example, showing replicate curves for two roller-compacted concrete specimens. The two distributions indicated are quite different from each other, and even the total intruded pore space is different. Such differences between the replicate specimens of the same concrete may reflect intrinsic heterogeneity in the pore system of the concrete.

Recently Diamond and Leeman [12] reported the results of paired studies of mercury intrusion pore size distributions and pore size distributions determined by SEM analysis on vacuum-mixed (no air voids) and otherwise identical air entrained pastes. The substantial volume of air voids of diameters in the range of 10 μm to 300 μm were correctly allocated to this range using microscopic image analysis, but were recorded by mercury intrusion only at high pressures. The air voids were allocated by mercury intrusion at less than 0.2 μm, about three orders of magnitude smaller than their actual diameters. It appears that mercury intrusion porosimetry is intrinsically inappropriate for measurements of the size distribution of air voids, and that this is likely holds for the large voids left by incomplete compaction in dry concretes.

Optical fluorescent thin-section observations. Contrary to mercury intrusion porosimetry, the observation of fluorescent thin-sections was found to be particularly well suited for the study of the microstructure of dry concrete products. The amount of fluorescent epoxy impregnated in a certain area is directly related to the capillary porosity of the cement paste. When observed in the fluorescent mode, areas with a low water to binder ratio appear much darker than those with a higher porosity. In addition to providing a rough estimate of the water to binder ratio, this method also gives a lot of information on the homogeneity of the sample. Unfortunately,

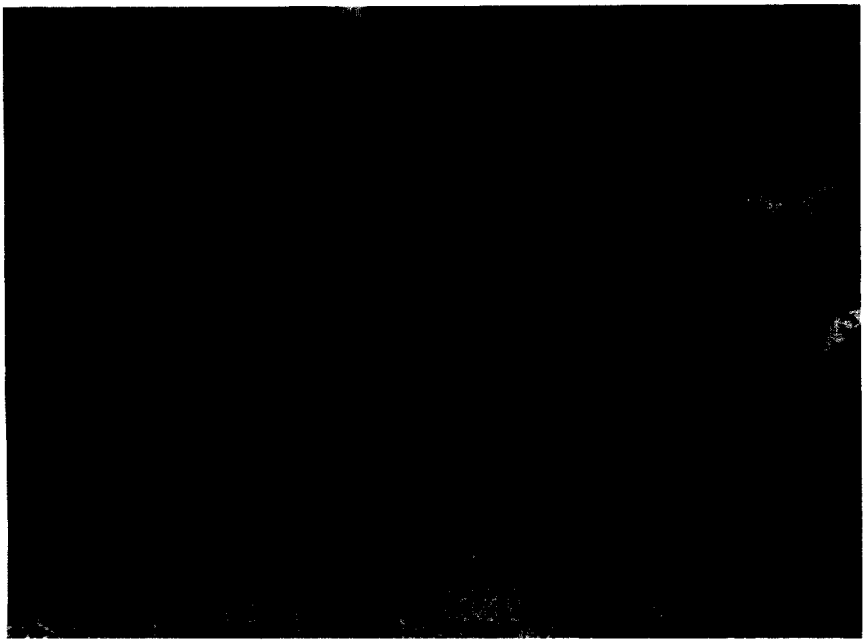

FIG. 4.
SEM micrograph: Typical paste-aggregate interface (Laboratory dry concrete - Blended silica fume cement).

color pictures can not be reproduced in this paper, and the reader will not be able to appreciate the full potential of this method.

When compared to that of ordinary concretes (i.e. concrete mixtures prepared with a superplasticizer), the paste fraction of most dry concretes appears to be much more heterogeneous. In ordinary concretes, variations in the fluorescent epoxy resin impregnation were only observable at higher magnifications (100X), and were mainly visible at the paste-aggregate interface. On the other hand, for most dry concretes, the dispersion of water appeared much more uneven, and variations of fluorescent color were clearly visible even at low magnifications (20X). These variations were not limited to the vicinity of the aggregates but were often located in the bulk of the cement paste.

The degree of heterogeneity was found to vary considerably with the type and composition of the dry concrete. The pre-cast paving block and the laboratory dry-concrete samples had quite similar features, and were found to be generally more homogeneous than the RCC samples. The heterogeneity of the pre-cast paving block concrete and of the laboratory concretes was generally limited to small areas.

No clear distinction could be made between the concretes made with different binders. In the present study, RCC samples made with the type 10 cement appeared to be the most heterogeneous. Interestingly, the use of silica fume and fly ash appeared to significantly enhance the homogeneity of the cement paste in the RCC mixtures. It seems that the addition of these mineral additives allowed a better dispersion of paste in the limited content of water provided.

SEM observations. The SEM observations made on polished sections at low magnifications underline another difference between the RCC samples and the two other types of dry concretes. Regardless of the type of cement, RCC compaction voids were always found to be irregularly shaped. On the other hand, the compaction voids of paving blocks and laboratory concretes appear to be more spherical.

These differences can perhaps be explained by the nature of the compaction operations. The RCC pavements were consolidated by static and vibratory rollers. The lack of any lateral restraint does not impede the horizontal movement of the various grains. The laboratory concretes and the paving blocks were cast in rigid molds, and were therefore submitted to a tri-axial pressure.

SEM observations also confirmed the higher degree of heterogeneity of the RCC samples. During the examination of the RCC samples made with the type 10 cement, it was frequent to encounter large zones of very porous C-S-H as shown in Figure 3. These areas were usually surrounded by more compact C-S-H, or by a loose arrangement of coarse aggregates.

The use of mineral additives appeared to enhance the homogeneity of RCC. Although small pockets of porous C-S-H were still visible, their number was more limited.

Observations at higher magnifications (1000X to 10000X) revealed small differences in the microstructural features between the ordinary concretes and those made with silica fume and fly ash. The C-S-H of all type 10 concretes had a typical layered structure, and were generally characterized by a microporosity. On the other hand, the C-S-H from the silica fume cement and from the type 10 cement with fly ash appeared more compact.

As it was the case with the optical microscope observations, it was extremely difficult to differentiate between the laboratory dry concretes and the paving blocks by SEM appearances. Both series of samples appeared to be dense and homogeneous. In both cases, the addition of fly ash or silica fume seemed to have improved the microstructure of the hydrated cement paste. The hydrates appeared more compact. The effect of the mineral additives, however, was much less obvious than in the case of the RCC samples.

The observations of the paste-aggregate interfaces revealed that they were systematically less dense than the bulk of the paste, and that they were generally microcracked. These observations were made for all types of dry concrete mixtures. As can be seen in Figure 4, the interfaces were often extremely porous, this higher porosity extending on a very significant distance from the interface. Curiously, in this respect, no significant differences between the various concretes observed were found. Neither the type of mixing nor the type of cement seemed to have influenced the poor quality of the paste-aggregate bond in these dry concretes. Given the good compressive strengths measured, the presence of these porous interfacial zones were quite unexpected.

Discussion

The microstructural differences that were observed between the RCC samples and the two other types of dry concrete are thought to be due to differences in the mixing operations. Both the laboratory and the paving block concretes were batched in counter-current pan mixers known for their very energetic mixing. On the other hand, the RCC mixtures were produced in an industrial pre-mix plant in which the mixing energy is significantly lower.

It is evident that the mixing energy required to obtain a proper water dispersion in dry concretes is significantly higher than that needed for ordinary "fluid" concretes. Dry concrete

mixtures are characterized by their stiffness and by the high viscosity of their cement paste. It thus probable that the energy in the pre-mix unit was insufficient to properly homogenize the cement paste. Similar conclusions have been reached concerning air entrainment in dry concretes [3, 4]. In this latter case, the use of a pan mixer was found to significantly facilitate the entrainment of air bubbles in dry concretes. It thus appears that these concretes are much more sensitive than ordinary concretes to the type of mixing. This information should be of interest to RCC producers that have to choose between a pre-mix plant and a continuous pug-mill mixer. The latter is known for its high energy and should therefore allow a more homogeneous dispersion of the mixing water throughout the paste.

The beneficial influence of silica fume and fly ash is considered by the authors to be due to a combination of two effects, one more or less macroscopic, the other microscopic. The first effect, limited to dry concretes, is related to the proper distribution of water during mixing. The addition of spherical fines to the mixture probably reduces the internal friction of the paste and helps to obtain a more uniform distribution of water. The authors' experience with RCC clearly indicates that silica fume mixtures are more "plastic" and easier to compact than ordinary Portland cement (OPC) mixtures. This is probably why the RCC mixtures containing fly ash or silica fume were found to be less heterogeneous than the OPC mixtures. Silica fume and fly ash were also found to improve the microstructure of the cement paste of dry concretes, notably that of the RCC mixtures. This effect is similar to that reported for ordinary concretes. The better spatial distribution of the hydrates and the consumption of portlandite by the pozzolanic reactions yields a denser matrix.

The presence of numerous porous and cracked interfacial zones in all types of concrete is hard to explain. It is possible that the high stiffness of these mixtures favors the formation of these porous interfaces. Due to this high stiffness, paste movements around the aggregate particles are limited during mixing. The water film at the wet aggregate surface can not adequately be distributed throughout the mixture, thus increasing the porosity at the interface. Even high-energy mixers are simply not efficient enough to remove the water film at the aggregate surface. Further research is certainly needed to confirm our observations and understand this phenomenon.

Conclusions

1. As shown by the optical microscope fluorescent thin-section and SEM observations, the cement paste fraction of dry concretes is often significantly less homogeneous than that of ordinary concretes. The cement paste of the RCC mixtures, produced in an industrial pre-mix plant, was found to be particularly heterogeneous.
2. High-energy mixing and the addition of mineral supplementary cementing materials, such as silica fume and fly ash, appear to enhance the homogeneity of the cement paste in dry concrete. Mineral supplementary cementing materials densify the microstructure of the cement paste considerably. Both effects can help to increase the mechanical strength and improve the frost durability.
3. The internal structure of the laboratory concretes and of the pre-cast paving blocks were found to be quite similar. However, both are significantly different, at both the macroscopic and the microscopic levels, from the RCC pavement mixture. This difference can be explained on the basis of the different production techniques, and indications concerning the performance of RCC obtained from laboratory-produced mixtures should therefore be considered with caution.

4. The application of mercury intrusion porosimetry to dry concretes yielded very variable results. This is most probably related to the internal structure of these concretes and more particularly to the presence of numerous compaction voids.

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