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PHASE I: ENERGY CONSERVATION POTENTIAL OF PORTLAND
CEMENT PARTICLE SIZE DISTRIBUTION CONTROL

Progress Report, May-July 1978

By
R. A. Helmuth

October 1978

Work Performed Under Contract No. EC-77-C-02-4269

Portland Cement Association
Construction Technology Laboratories
Skokie, Illinois

U. S. DEPARTMENT OF ENERGY

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Energy Conservation Potential
of Portland Cement
Particle Size Distribution Control

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Phase I Energy Conservation Potential of
Portland Cement Particle Size Distribution Control
ERDA Contract No.: EC-77-C-02-4269
PCA Contract No.: CR-7523-4330

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SUMMARY

The Series III preliminary tests were completed. These results more precisely define the amounts of fine particles required to obtain both adequate paste flow and strength development than did the Series II tests. Controlled particle size ranges with maximum sizes of 45, 20, and 14 microns were used with various amounts (0 to 45%) of 8 to 1 micron added fines. Better strength development was obtained with a maximum particle size of 20 microns than with 45 or 14 microns at equal finenesses, ($2,900-3,000 \text{ cm}^2/\text{g}$), or with normally ground cement of higher fineness ($3,655 \text{ cm}^2/\text{g}$). The flow of pastes made with this 20 micron cement was not quite as good as that of the 45 micron or normally ground cements. The data indicate that increasing the maximum particle size to 30 microns may be advantageous, and that the amounts of added fines for optimum performance lies between about 10 and 25%. Materials are being prepared for the Series III tests with maximum particle sizes of 30 and 20 microns with 10 to 25% fines additions.

Series III Preliminary Tests

The wide variations in paste properties found with the Series II blends made it difficult to make reasonable estimates

of the amounts of fines required for the blends to be used in Series III tests. Hence, a short series of preliminary tests was begun in the previous quarter to more narrowly define the amounts of fines required to obtain both adequate flow and strength development. These blends were prepared from excess materials remaining from the Series II preparations. The proportions of the primary size fractions for these blends were chosen to provide intermediate amounts of fines in blends otherwise similar to those used in the Series II tests. Primary particle size fractions of the low C_3A cement clinker MCC-274 with maximum particle sizes of 45, 20, and 14 microns were blended with various amounts of the 8 to 1 micron fraction and 3.78% gypsum to produce these cements. The proportions used are given in Table I (Table V of the previous report). Blaine specific surface (B.S.S.) values are also given.

The size ranges used in Series II were obtained by blend proportions that terminated rather sharply at each end. Hence, they were designated 45-2, 20-2 etc. microns. In the present series the termination of the fine end is not so sharp and the size ranges are designated 45-X, 45-Y etc. to reflect this fact. The actual particle size distribution curves are given in Figs. 1 through 3 for these blends and the corresponding blends from Series II.

Procedures used were the same as used in the Series II tests for materials preparation, mixing, mini-slump cone flow tests, and setting time. However, in these tests 1x2-inch

cylinders were cast for strength tests instead of the 1-inch cubes previously used.

The results of these tests are given in Table II. The strength data are given as strengths for 1-inch cubes calculated from the cylinder strengths for comparison with the Series II data. Since the ratio of cylinder strengths to cube strengths is about 0.8 (F.M. Lea, The Chemistry of Cement and Concrete, 3rd Edition, Chemical Publishing Company, 1971, p. 390), this factor was used to obtain the calculated cube strengths. This factor appears to yield reasonable strength relationships between these and the Series II test data, and will be verified for some of the mixes in Series III.

Most of the data are plotted in Figs. 4-10. These plots also include the Series II results for the controls (100-0 microns, ball-milled) and for the 45-2, 14-5, and 20-2 micron particle size cements at 3.78% gypsum. Figs. 4 and 5 show the mini-slump pat area data. They show the expected progressive increase in water requirement for flow with increasing content of added fines. To provide some basis for comparison it should be noted that previous work (on another project) has shown that to obtain workable concrete (2 to 3-inch slump) with normally ground cements at water-cement ratios of 0.41 to 0.43 the pastes should have pat areas of at least 5 in.² This requirement was met by the pastes made with the normally ground cement, but some of the other pastes had smaller pat areas and may not yield workable concretes (see later for further discussion).

Figures 6 and 7 show the bleeding water loss data. Since the method of measuring the bleeding is not very precise a few

points showing large deviations from the values indicated by interpolation between other points are omitted from the plots. These values have been replaced in the tables, as indicated, by estimated values obtained by interpolation from the remaining data. The bleeding data also show the expected progressive decrease in bleeding with increasing content of added fines.

The compressive strength data are shown in Figs. 8, 9, and 10 for the values at 1 and 28 days as functions of the water-cement ratio corrected for bleeding.

Since our major concern here is the amount of fine material required to reach a satisfactory compromise between the flow and strength development these properties will be discussed together for each size range.

1. Figure 4 shows that for the cements with 45 micron maximum sized particles the addition of 8 to 1 micron fines to the 45 micron primary size fraction results in progressively higher water requirements for flow. With 10 and 15% of added fines the flow was greater than that of the controls; at 25 and 35% of added fines the flow was less than that of the controls. The 1 and 28 day strengths shown in Fig. 8 exceed those of the controls only when the added fines exceed 25%. Even when 35% of added fines are used the strengths are only slightly higher than those of the controls. This blend may yield workable concretes with this low C_3A content cement, but at 35% added fines the strengths are only slightly better than with 25%

fines and the B.S.S. ($3,316 \text{ cm}^2/\text{g}$) is approaching that of the control cement ($3,655 \text{ cm}^2/\text{g}$).

2. Similar tendencies are shown for the cements with 14 micron maximum sized particles with additions of the 8 to 1 micron fines to the 14 to 5 micron primary size fraction. In this case the B.S.S. values are all considerably less than that of the control cement. Fig. 5 shows that with 0% added fines the water requirement for flow is about the same as that of the controls, and that addition of up to 25% fines caused a progressive reduction of the flow. The strengths of the 0% and 10% added fines cements shown in Fig. 9 were less than the controls. At 25% added fines the strengths slightly exceeded those of the controls, as in the case of the <45 micron cements. However, these improved strengths were achieved at a lower B.S.S. ($2,930 \text{ cm}^2/\text{g}$) than with the <45 micron cements. In both cases the gains in strengths are achieved with some loss of flow.
3. The case of the 20 micron maximum particle size cement is interesting because the strengths, shown in Fig. 10, are significantly greater than those of the strongest < 14 micron cement at about the same B.S.S. ($2,970 \text{ cm}^2/\text{g}$ with 15% added fines) while the flow values shown in Fig. 4 are also slightly better. The bleeding was also less. Hence, some strength could be sacrificed to achieve better flow by some further reduction in the amount of added fines, to perhaps 10%, with some

further reduction of the Blaine fineness. Even with 15% added fines the Blaine fineness is significantly less than that of the control cement ($3,655 \text{ cm}^2/\text{g}$).

Since the strengths were increased by increasing the maximum size from 14 to 20 microns (at the same B.S.S.) it also seems possible that some further increase could be achieved by increasing the maximum size to perhaps 30 microns. At 45 microns, Fig. 8 shows that the strengths are again lower. Increasing the maximum particle size should also improve the paste flow as seen by the comparison of the <20 and <45 micron cements in Fig. 4. Then it may not be necessary to reduce the amounts of fines added, and to sacrifice strength. It may even be possible to increase the amounts of fines somewhat.

However, another factor to be considered concerning the effect of the maximum cement particle size on the flow is that in concretes the largest cement particles interfere with the flow of fine aggregate particles. This is discussed in Powers' book (Ref. 25, the proposal for this work) where it is noted that lower water-cement ratios can be used with finer cements in concretes although the paste itself may be of stiffer consistency. Hence it may be possible to use a cement like the 20-Y cement with even more than 15% of added fines at as low water-cement ratios in concrete as is possible with the control cement. Series IIIa will provide an opportunity to test for this effect.

The results of these tests have more narrowly defined the particle size distributions required for the Series III tests.

The data show that a maximum particle size of 20 microns is better than 14 microns, and that it may be advantageous to increase this to 30 microns, which would also be easier to achieve in practice. The amounts of fines to be added for optimum performance is still somewhat uncertain, but seems to lie between about 10 and 25%, depending on how sensitive the workability of the concrete is to variations of the maximum size of the cement particles.

Materials Preparations for Series III

On the basis of the foregoing results, preparation of the primary size fractions for the Series III blends was begun. The ball-milled materials were prepared first while data analysis continued. Maximum particle sizes of 30 and 20 microns were selected for the controlled size distributions and the Vortec milling of these fractions was initiated.

TABLE I

Pebble-Milled Blends of MCC-274
Cement Clinker Size Fractions and
T.A. Gypsum of 7,835 cm²/g Blaine Specific Surface (B.S.S.)

Blend No.	Size Range (microns)	B.S.S. cm ² /g	Gypsum Percent	L.O.I. Percent	Clinker Size Fraction Percentages			
					<45 Microns	<20 Microns	14-5 Microns	8-1 Microns
PM 17	45-2	3,316	3.78	1.40	65	0	0	35
PM 31	45-X	3,084	3.78		75	0	0	25
PM 32	45-Y	2,686	3.78		85	0	0	15
PM 33	45-Z	2,533	3.78		90	0	0	10
PM 34	14-X	2,930	3.78		0	0	75	25
PM 35	14-Y	2,687	3.78		0	0	85	15
PM 36	14-Z	2,561	3.78		0	0	90	10
PM 23	14-5	2,310	3.78	1.32			100	0
PM 26	20-2	3,671	3.78	1.47		55		45
PM 37	20-Y	2,970	3.78		0	85	0	15

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TABLE II

Properties of Cement Pastes Made From
Various Particle Size Ranges of MCC-274
with 3.78% Gypsum

Blend - w/c			Size Range (Microns)	B.S.S. cm ² /g	Pat Area in. ²	Time of Set hrs:min.	Final w/c (Corrected for Bleeding)	1-inch Paste Cube Compressive Strength ^a (psi)		
								1d	7d	28d
PM 31	0.35	45-X	3,084	2.32	4:46	0.34 ^b	3,636	10,070	12,647	
PM 31	0.50	45-X	3,084	8.08	5:55	0.44	1,424	4,492	7,606	
PM 31	0.65	45-X	3,084	13.24	7:05	0.53	885	2,596	4,127	
PM 32	0.35	45-Y	2,686	2.36	4:45	0.32	2,385	9,475	14,120	
PM 32	0.50	45-Y	2,686	9.83	6:09	0.41	1,010	4,232	7,006	
PM 32	0.65	45-Y	2,686	21.39	7:10	0.51	708	2,270	4,210	
PM 33	0.35	45-Z	2,533	2.32	5:00	0.32	1,661	9,175	12,912	
PM 33	0.50	45-Z	2,533	11.59	6:00	0.38	930	3,311	8,470	
PM 33	0.65	45-Z	2,533	22.25	7:28	0.45	580	3,183	5,957	
PM 34	0.35	14-X	2,930	--	3:35	0.32	4,507	11,795	14,851	
PM 34	0.50	14-X	2,930	5.50	5:40	0.41	1,727	5,749	10,222	
PM 34	0.65	14-X	2,930	10.27	6:15	0.51	1,131	3,381	5,315	
PM 35	0.35	14-Y	2,687	--	4:00	0.32	3,422	14,986	15,195	
PM 35	0.50	14-Y	2,687	6.40	6:11	0.40 ^b	1,886	6,376	11,395	
PM 35	0.65	14-Y	2,687	12.52	6:56	0.47	1,057	3,239	5,795	

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TABLE II (Continued)

Properties of Cement Pastes Made From
Various Particle Size Ranges of MCC-274
with 3.78% Gypsum

Blend - w/c	Size Range (Microns)	B.S.S. cm ² /g	Pat Area in. ²	Time of Set hrs:min.	Final w/c (Corrected for Bleeding)	1-inch Paste Cube Compressive Strength ^a		
						1d	7d	28d
PM 36 0.35	14-Z	2,561	--	3:55	0.32	3,332	11,311	12,615
PM 36 0.50	14-Z	2,561	7.19	6:10	0.39 ^b	1,332	6,349	10,447
PM 36 0.65	14-Z	2,561	14.15	7:20	0.45 ^b	937	3,072	5,719
PM 37 0.35	20-Y	2,970	--	3:50	0.35	4,256	11,885	14,370
PM 37 0.50	20-Y	2,970	5.60	6:00	0.44	1,837	5,840	11,210
PM 37 0.65	20-Y	2,970	10.60	6:25	0.53	1,237	3,068	6,004

^aCalculated from 1x2-inch cylinder strengths.

^bEstimated by interpolation.

PARTICLE SIZE DISTRIBUTION

SAMPLE IDENTIFICATION

DATE

Density g/cc LIQUID

Density g/cc Viscosity cp

BY

Preparation

TEMPERATURE °C

RATE START DIA. μ m

Fig. 1

PARTICLE SIZE DISTRIBUTION

SAMPLE IDENTIFICATION

DATE

Density g/cc LIQUID

Density g/cc Viscosity cp

BY

Preparation

TEMPERATURE °C

RATE START DIA. μ m

Fig. 2

PARTICLE SIZE DISTRIBUTION

SAMPLE IDENTIFICATION

DATE

Density g/cc LIQUID

Density g/cc Viscosity cp

BY

Preparation

TEMPERATURE °C

RATE START DIA. μ m

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8

Fig. 9

Fig. 10