Mix Proportioning

The graded coarse and fine aggregates were weighed in the room-dry condition. The coarse aggregate was then immersed in water for 24 hours: the excess water was decanted and the water retained by the aggregate was determined by weight difference. A predetermined amount of water was added to the fine aggregate, which was then allowed to stand for 24 hours.

A standard mix with water-cement ratio of 0.42, aggregate-cement ratio of 4.77 and cement content of 639 lb/yd^3 (379 kg/m^3) was used. The dosage of air-entraining agent was constant but the type and dosage of superplasticizers were varied as shown in Table 4.

Properties of Fresh Concrete

The properties of the fresh concrete, i.e., temperature, slump, unit weight and air content, were determined after the initial mixing time of 6 minutes, and again after addition of the superplasticizers and further mixing for 2 minutes (Table 4). Also, measurements were taken frequently to determine the rate of loss of slump.

Initial Time of Set of Fresh Concrete

To determine whether the superplasticizers retarded the set of the concrete, initial times of set were determined in accordance with ASTM Standard C403-77.

PREPARATION AND CASTING OF TEST SPECIMENS

Concrete Mixes No. 1 to 12

Six 4 x 8-in. (102 x 203-mm) cylinders and six 3.5 x 4 x 16-in. (89 x 102 x 406-mm) prisms were cast from each mix. All test specimens were cast after adding superplasticizers except for for mixes 1 and 2, which were control mixes. Three cylinders were compacted using a vibrating table; the remaining three cylinders were not subjected to any vibration. The prisms were cast by filling brass moulds and compacting the moulds on a vibrating table. After casting, all the moulded specimens were covered with water-saturated burlap, and were left in the casting room at 75 ± 3°F (24 ± 1.3°C) and 50% relative humidity for 24 hours. They were then demoulded and transferred to the moist-curing room until required for testing.

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Concrete Mixes No. 13 to 15

Six 4 x 8-in. (102 x 203-mm) cylinders were cast from each of the three mixes: two cylinders were cast immediately after completion of initial mixing; two cylinders were cast after adding the superplasticizers and further mixing for two minutes; the remaining two cylinders were cast after the concrete had been allowed to stand in the mixer for 120 minutes. The cylinders were cast by filling steel moulds in two approximately equal layers, each compacted on a vibrating table.

TESTING OF SPECIMENS

Concrete Mixes No. 1 to 12

At 14 days, two prisms were removed from the moist-curing room and tested in flexure according to ASTM Standard C78-75, using a third point loading. At 28 days, both vibrated and non-vibrated cylinders from each mix were removed from the moist-curing room, capped with a sulphur and flint mixture and tested in compression on a 600,000-lb (272, 160-kg) testing machine.

Concrete Mixes No. 13 to 15

At 28 days, the three sets of cylinders from each mix were removed from the moist-curing room, capped with a sulphur and flint mixture, and tested in compression.

DURABILITY STUDIES

Although durability cannot be measured directly, prolonged exposure of concrete to repeated cycles of freezing and thawing produces measurable changes in test specimens that may indicate deterioration. Measurements made on the test specimens after freeze-thaw cycling provide data that can be used to evaluate the relative frost resistance or durability.

In this investigation, test prisms were exposed to repeated cycles of freezing in air and thawing in water according to ASTM Standard C666-75. The automatic freeze-thaw unit can perform eight cycles per day. One complete cycle from 40 ± 3°F to 0 ± 3°F (4.4 ± 1.7°C to -17.8 ± 1.7°C) and back to 40 ± 3°F (4.4 ± 1.7°C) requires about 3 hours. During this investigation the freeze-thaw unit did not fully meet the above temperature requirements, fluctuating between 5 and 11°F (-15 and -11.7°C) during freeze cycles.

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At the end of the initial moist-curing period of 14 days, the temperature of each set of prisms was reduced to a uniform 40 ± 3°F (4.4 ± 1.7°C) by placing in the freeze-thaw cabinet at the thawing phase for one hour. The initial and all subsequent measurements of the freeze-thaw and reference test specimens were made at this temperature. After initial measurements of the test prisms were taken, two test prisms were placed in the freeze-thaw cabinet and the two companion prisms placed in the moist-curing room for reference purposes.

The freeze-thaw test specimens were visually examined at the end of every 50-cycle interval. Their lengths were measured and they were weighed and tested by resonant frequency, and by the ultrasonic pulse method at approximately every 100-cycle interval. The freeze-thaw test was terminated at the end of 700 cylces in each case, when both the freeze-thaw and reference prisms were tested in flexure.

Another useful index to determine the durability of concrete exposed to freeze-thaw cycling is the bubble spacing factor, an index related to the maximum distance in inches of any point in the cement paste from the periphery of an air void. The spacing factor for concrete under investigation was determined in accordance with ASTM Standard C457-71 using the modified point count method.

TEST RESULTS AND THEIR ANALYSIS

Ninety cylinders and seventy-two prisms were tested in this investigation. The densities of all specimens were taken at one day as shown in Table 5. The setting times of the concretes are shown in Figure 2 and the loss of slump with time is shown in Figures 3 to 7, with a view of a typical flowing concrete in Figure 8. A summary of the compressive and flexural strengths is given in Tables 6 to 8 and the data are illustrated in Figures 9 to 12. The ratio of flexural to compressive strength for the test data is shown in Figure 13. Figure 14 shows a comparison of test cylinders cast without compaction with those cast using external vibration.

Changes in weight, length, pulse velocity and resonant frequencies on reference prisms and prisms subjected to freeze-thaw cycling are shown in Tables 9 to 12. Photographs of typical test prisms before and after freeze-thaw cycling are shown in Figure 15.

Results of the air-void analyses of the hardened concrete test specimens are given in Table 13.

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DISCUSSION

Superplasticizers - Is this the correct term?

Since their introduction into North America the new admixtures have been variously called "superplasticizers", "super water reducers", "high-range water reducers", and "superfluidifiers". In Germany, they are called "superverflussiger" (1,2), the literal English translation of which is superfluidifiers. Before the technical literature becomes cluttered with these different names, it is important that a consensus be reached on the correct name.

Mode of Action of Superplasticizing Admixtures

Superplasticizing admixtures act by causing the cement agglomerates to disperse. According to a report by the Cement and Concrete Association, London, their mode of action is best described as follows (3):

> "These admixtures are thought to be adsorbed onto cement particles, causing them to become mutually repulsive as a result of the anionic nature of superplasticizers, which causes the cement particles to become negatively charged. In principle, this adsorption and dispersing effect is similar to that found for normal anionic plasticizers".

Initial Time of Set of Concrete

All superplasticizers investigated had a retarding effect on the time of initial set of concrete as measured by ASTM Standard C403-70. At the recommended dosage rates* of the superplasticizers, the time of initial set was least affected by the Melment L10, followed in turn by Mighty 150 and Mulcoplast CF (Figure 2). At higher dosages Mulcoplast CF retarded the initial set by about four hours with the initial set of the reference concrete occurring at 3 hours and 50 minutes. This was probably so because Mulcoplast CF is a lignin-based water reducer. The set retarding property of the superplasticizers can be either beneficial or detrimental, depending on the job application.

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*Melment L10 - 2% by weight of cement
Mighty 150 - 1% by weight of cement
Mulcoplast CF - 2% by weight of cement

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Segregation of Superplasticized Concrete

When examined visually, the superplasticized concretes did not show significant segregation even when used at the maximum recommended dosage. When Mighty 150 was used at 10% by weight of cement, there was complete segregation of coarse aggregate from the cement matrix, accompanied by a foaming action. The concrete also failed to set for a number of hours. Of all the superplasticizers investigated, concrete superplasticized with Mulcoplast CF appeared to be more cohesive. If superplasticized concrete is placed by buckets, segregation of concrete should pose no serious problems. However, if placed using a conveyor belt system, segregation may have to be watched closely.

Increases in Slump and Its Loss with Time

Superplasticized concretes exhibited very large increases in slump at the recommended dosages. The slumps reached 8 in. (206 mm) or more within minutes of adding the superplasticizers and the low-slump concretes became flowing concretes (Figures 3 to 8). Even at these high slumps there were no signs of serious segregation. The concretes maintained high slumps for the initial 5 to 10 minutes, following which there was rapid loss in slump. Concrete superplasticized with Melment L10 lost slump more rapidly than concrete with either of the other two agents tested. At recommended dosages, superplasticized concretes reverted to the original slump of about 2 in. (50 mm) in less than 90 minutes under laboratory temperature and humidity conditions. The rate of course varied with the dosage. At maximum recommended dosages*, the superplasticized concrete lost slump at a slower rate; at the elapsed time of two hours, the concretes superplasticized with Melment L10, Mighty 150 and Mulcoplast CF had residual slumps of 1 1/2, 2, and 5 1/2 in. (38, 50 and 140 mm) respectively.

To take advantage of these large increases in slump, concrete must be transported and placed quickly. This can easily be achieved in precast concrete plants but may create problems for cast-in-place concrete. In spite of these difficulties, superplasticizers offer opportunities for placing high-strength concrete in heavily reinforced sections without incurring segregation or honeycombing. This appears to be their principal advantage.

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*Melment L10 - 3% by weight of cement
Mighty 150 - 1.5% by weight of cement
Mulcoplast CF - 3% by weight of cement

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Compressive Strength Development

Concrete Superplasticized with Melment L10

At the dosage rates investigated, the 28-day compressive strengths of cylinders compacted by vibration were about 10% higher than the strengths of cylinders cast from the air-entrained control mix (Figure 9). This was also true for test cylinders that were not vibrated, except that for dosages of 2.0 and 3.0% the difference was only about 5%. For 1.0% dosage, honeycombing caused the strengths of non-vibrated cylinders to be substantially lower than those of the control cylinders (Figure 14).

Concrete Superplasticized with Mighty 150

At the dosages investigated, with the exception of 10%, the compressive strengths of cylinders cast from the superplasticized concrete were equal to or higher than the strengths of cylinders cast from the air-entrained control mix (Figure 10). This was true for both vibrated and non-vibrated cylinders. At 0.5 and 1.0% dosage rates, the difference in strength was slightly more than 10%; at the 1.5% dosage rate, strength of the cylinders cast from the air-entrained control mix was equal to that of the superplasticized concrete.

Concrete Superplasticized with Mulcoplast CF

At dosage rates of 1 and 2%, the compressive strengths of cylinders were equal to or only slightly higher than the strengths of cylinders cast from the air-entrained control mix (Figure 11). However, at the 3% dosage rate, the strengths of cylinders were substantially lower than those of cylinders from the control mix. The difference was 680 psi (4.69 MPa) for cylinders cast without compaction by vibration, the compressive strength of the control cylinders being 5550 psi (38.27 MPa). The concrete mix superplasticized with Mulcoplast CF had entrained higher amounts of air than the control mix; this probably explains why the strengths of cylinders cast from the superplasticized mix failed to show any improvement compared with those of control cylinders, contrary to results using Melment L10 and Mighty 150.

At the 3% dosage rate, the higher strengths of non-vibrated cylinders compared with vibrated cylinders are unexplained.

Concrete Mixes No. 13 to 15

The test cylinders cast immediately before adding superplasticizers showed no significant difference in strength from those cast immediately after mixing in the additives for 2 minutes (Table 7). However, test cylinders cast 120 minutes after the addition of superplasticizers showed significantly higher strengths.

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Flexural Strengths

In general, at the recommended dosage rates, the 14-day flexural strengths of test prisms cast from the superplasticized concretes showed no significant change from the strengths of control prisms except for concretes superplasticized with Mulcoplast CF, in which case the strengths dropped by about 10% (Figure 12).

The prism specimens cast from concrete superplasticized with Melment L10 showed a steady increase in strength with increased dosage rate. At the 3% dosage rate, the flexural strength of the test prisms was 1050 psi (7.24 MPa) compared with 1000 psi (6.89 MPa) for the test prisms cast from the air-antrained control mix.

The prisms cast from concrete superplasticized with Mighty 150 showed slight increases in strength over those of the control prisms at dosage rates of 0.5 and 1.0%; at the 3% dosage rate the strength dropped, reaching a value of 970 psi (6.69 MPa) compared with 1000 psi (6.89 MPa) for the control prisms. This is not considered significant.

The prism specimens cast from concrete superplasticized with Mulcoplast CF showed slight increases in strength at a dosage rate of 1.0%, compared with the strengths of the control prisms. At 2 and 3% dosage rates, the strengths of the prisms dropped sharply. A value of 920 psi (6.34 MPa) was reached at 2%; this drop is unexplained because the compressive strengths of companion cylinders did not drop.

Durability of Concrete Prisms Exposed to Repeated Cycles of Freezing and Thawing

Durability of concrete prisms exposed to repeated cycles of freezing and thawing was determined by measuring weight, length, resonant frequency and pulse velocity of test prisms before and after exposure to freeze-thaw cycling and comparing these with corresponding values of reference prisms.

In general, there were no significant changes in the condition of the test prisms after about 700 cycles, when the freeze-thaw tests were discontinued (Tables 8-12). The changes in length of the prisms after 300 cycles of freezing and thawing were well within the limit of 0.07% set by Klieger for durable concrete (7). The only exceptions were the non-air-entrained control prisms and the prisms cast from concrete superplasticized with Mighty 150 at a dosage rate of 10%.

The non air-entrained control prisms had shown a relative length change of 0.36% at 100 freeze-thaw cycles; the relative losses in the longitudinal resonant frequency and the ultrasonic pulse velocity were 35.7 and 27.6% respectively. The prisms were damaged

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to such an extent at the end of 100 freeze-thaw cycles that no flexural tests were possible.

The prisms cast from concrete superplasticized with Mighty 150 at a dosage rate of 10% had completely disintegrated at the end of 60 freeze-thaw cycles, thus preventing determination of resonant frequency, pulse velocity and relative length changes.

The freeze-thaw tests were performed using ASTM Standard C666-76 and employing Procedure B "Rapid Freezing in Air and Thawing in Water". ASTM Standard C494-71, Chemical Admixtures, specifies the use of Procedure A, "Rapid Freezing and Thawing in Water" for evaluating concrete incorporating chemical admixtures. Nevertheless, the reported freeze-thaw data are considered valid because the freeze-thaw test is a comparative test carried out with the specimens cast from the control mixes; for the investigation reported herein, the test prisms cast from the non-air-entrained control mix had disintegrated at 100 freeze-thaw cycles. Limited published data by Mukherjee and Chojnacki indicate that superplasticized concrete prisms perform satisfactorily when exposed to rapid freezing and thawing in water in accordance with Procedure A of ASTM Standard C494-71 (8).

Air Void Determination of Hardened Concrete

The microscopical determination of air void content, and parameters of the air void system in hardened concrete, were determined according to ASTM Standard C457-71. It has been found that for satisfactory durability, the cement paste should be protected with air bubbles. Adequate protection required that the spacing factor, an index related to the maximum distance of any point in the cement paste from the periphery of an air void, not exceed 0.008 in. (0.20 mm) (9). In the superplasticized concretes under investigation, the bubble spacing factor varies between 0.006 and 0.01 compared with 0.006 for the control air-entrained concrete (Table 13). In spite of the increased bubble spacing in some cases the durability of the concrete test specimens is not impaired. This is of considerable significance and investigations are needed to explain this phenomenon.

Elastic Properties of Superplasticized Concrete

No tests were performed to determine the elastic properties of the superplasticized concretes in this investigation; however, subsequent investigations at CANMET indicate that Young's modulus of elasticity is not affected by the incorporation of superplasticizers (10). Research is currently underway to determine the long-term creep characteristics of the superplasticized concrete. Limited published data on the subject are inconclusive (3,4).

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Superplasticized Concrete and Accelerated Strength Testing

Limited accelerated strength tests performed on test specimens prepared from superplasticized concrete show compressive strength development identical to that of test cylinders prepared from the reference concrete (11). Additional investigations are being done to cover a wide range of mix proportions and different types of superplasticizers using the modified boiling method.

GENERAL COMMENTS AND CONCLUSIONS

Superplasticized concretes show very large increases in slump with no significant segregation apparent in laboratory investigations. Thus, by incorporating superplasticizers, high-strength concretes can be placed in heavily reinforced and inaccessible areas. Superplasticized concretes do lose slump with time and this is one of the serious limitations of the new water reducers. However, with judicious selection of the dosages and by adding superplasticizers to concrete at a job site the above problems can be considerably reduced, if not eliminated altogether.

All superplasticizers investigated had a retarding effect on the time of initial set. This can be either beneficial or detrimental depending on the nature of the job on which they are used.

When superplasticizers are added to concrete at the manufacturers' recommended dosages, the 28-day compressive strengths of test cylinders cast from the superplasticized concrete are equal to or greater than the corresponding strengths of cylinders cast from the reference mix. This is true for cylinders cast with and without compaction by vibration, implying that high-strength concretes incorporating superplasticizers can be placed in forms without the need for mechanical compaction, which can result in considerable savings of time and money.

Limited available data on the elastic properties of superplasticized concretes indicate that Young's modulus of elasticity is not adversely affected by the use of superplasticizers. Further research is indicated in this direction.

The freeze-thaw durability of test specimens cast from superplasticized concretes compares favourably with those cast from the control mix regardless of the value of the bubble spacing factor in the hardened concrete.

There were some differences in performance of the three superplasticizers with regard to the compressive and flexural strengths of concretes and their loss of slump with time. Because the data are limited, no conclusions can be drawn concerning the relative performance of the three superplasticizers used.

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The results presented in this report were obtained for concrete having a water-cement ratio of 0.42 and made with ASTM Type 1 cement. The superplasticizers may or may not perform as reported in concretes made with other water-cement ratios and with different types of cements and aggregates.

Superplasticizers are more expensive than ordinary water reducers and are thus not economical for use in every-day concrete; they are ideal where flowing concretes with very low water-cement ratios are required.

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