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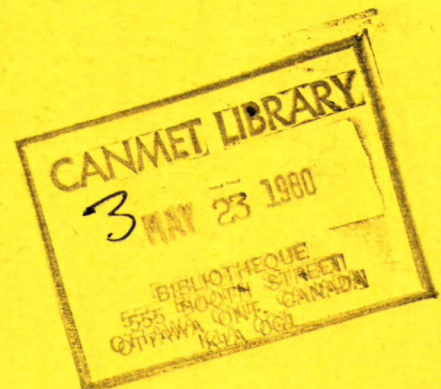
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SUPERPLASTICIZERS: THEIR EFFECT ON FRESH AND HARDENED CONCRETE

V.M. MALHOTRA



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SUPERPLASTICIZERS: THEIR EFFECT ON FRESH AND HARDENED CONCRETE

by

V.M. Malhotra*

SYNOPSIS

Superplasticizers are a new family of admixtures which can either be used as high-range water reducers or be incorporated into concrete to produce "flowing" concrete. They were introduced into North America in 1976 and since then a number of research laboratories have been developing data on their effect on the properties of fresh and hardened concrete. This paper reviews these developments. The properties of fresh concrete reviewed include bleeding and segregation, increases in slump and its subsequent loss with time, initial setting time, entrained air content, effect of repeated dosages, vibration requirements, and pumpability of superplasticized concrete. The properties of hardened concrete reviewed include accelerated strength, mechanical and elastic properties, freeze-thaw durability, resistance to salt scaling and sulphate attack.

The review indicates that incorporation of superplasticizers does not adversely affect the properties of fresh concrete, though superplasticized concretes lose slump rapidly with time and this is accentuated at elevated temperatures. All superplasticizers appear to have no significant retarding effect on the time of initial set of concrete and the rate of bleeding of fresh concrete is not affected.

When superplasticizers are added to concrete at the manufacturers' recommended dosage rates, the strength, shrinkage and creep properties of test specimens cast from the superplasticized concrete are comparable to the corresponding properties of specimens cast from the reference mix; the compressive strengths are comparable when the cylinders from the superplasticized concrete are cast without compaction by vibration and greater with vibration.

In the superplasticized concretes at the recommended dosage rates, the bubble spacing factor is generally greater than 200 μm , a value considered maximum for satisfactory freeze-thaw durability. In spite of increased bubble spacing factor the freeze-thaw durability of air-entrained superplasticized concrete compares favourably with that of air-entrained reference concrete.

The review indicates that these admixtures have opened up a new era in concrete technology and are already being used in the precast concrete industry. However, before their use becomes widespread for ready-mixed concrete, problems associated with slump loss would have to be overcome.

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SUPERPLASTIFIANTS: LEUR EFFET SUR LE BETON FRAIS ET DURCI

par

V.M. Malhotra*

SYNOPSIS

Les superplastifiants sont une nouvelle famille d'additifs qui peuvent soit être employés comme réducteurs de l'eau de grande envergure ou être incorporés dans le béton pour leur donner une meilleure propriété d'écoulement. Ils ont été introduits en Amérique du Nord en 1976. Depuis, plusieurs laboratoires de recherche ont accumulé des données sur leur effet sur les propriétés du béton frais et durci. Le présent rapport résume ces développements. Les propriétés du béton frais mentionnées comprennent le ressuage et la ségrégation, augmentations de l'affaissement et les pertes subséquentes dans le temps, le temps initial de prise, la teneur en air entraîné, l'effet de dosages répétés, la nécessité de vibration et la pompabilité du béton superplastifié. Les propriétés du béton durci que l'on mentionne dans ce rapport incluent la résistance accélérée, les propriétés mécaniques et élastiques, la durabilité au gel-dégel, la résistance à l'écaillage causé par le sel et l'attaque du sulfate.

Ce rapport démontre que l'incorporation de superplastifiants n'a causé aucun effet défavorable sur les propriétés du béton frais quoique les bétons superplastifiants perdent leur propriété d'affaissement rapidement selon le temps surtout à haute température. Tous les superplastifiants semblent n'avoir aucun effet retardataire sur le temps de prise initiale du béton et la vitesse de ressuage du béton frais n'est pas affectée.

Lorsque les superplastifiants sont ajoutés au béton selon la dose recommandée par le manufacturier, la résistance, les propriétés de retrait et du gonflement des échantillons coulés à partir de béton superplastifiants sont comparables aux propriétés correspondant aux échantillons coulés à partir d'un mélange de référence. Les résistances à la compression se comparent bien lorsque des cylindres provenant du béton superplastifiant sont coulés sans être tassés par la vibration mais sont plus grandes avec l'aide de vibration.

Dans les bétons superplastifiants, mélangés à la vitesse de dosage recommandée, le facteur d'espacement des bulles est généralement plus de 200 μm , valeur maximale pour donner une durabilité au gel-dégel satisfaisante. En dépit de l'augmentation du facteur d'espacement des bulles, la durabilité au gel-dégel du béton superplastifiant avec air entraîné se compare bien au béton de référence avec air entraîné.

Le rapport explique que ces additifs ont ouvert les portes à une nouvelle technologie du béton et sont déjà employés dans l'industrie de pièces pré-fabriquées en béton. Par contre, certains problèmes concernant la perte d'affaissement devront être surmontés avant que l'usage de ces additifs se soit répandu.

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INTRODUCTION

Superplasticizers are new types of admixtures that have only recently been introduced into North America, although they have been used in Japan since the 1960's, and in Europe since 1972 (1-4). In Japan, their use has primarily been to produce high strength concrete by making possible large reductions in water content whereas in Germany they were developed to produce "flowing" concretes, i.e., concretes having slump in excess of 200 mm. To achieve these properties, large doses of the new chemical admixtures are needed compared with the conventional water-reducing admixtures, and consequently the superplasticizers are relatively expensive.

The new admixtures are finding increasing acceptance in the precast concrete industry but ready-mixed concrete producers have been slow to accept them because of the associated slump loss and increased cost.

In 1978-79, three different international symposia were held in Canada, the U.S.A. and Mexico to discuss the usefulness, advantages and limitations of these new admixtures (5,6,7). This paper briefly describes the main types available in North America, discusses their mode of action, and describes their effect on the properties of fresh and hardened concrete. Although emphasis has been placed on the data available from North American sources, references are made to the original research reported in Japan, Germany and other countries.

TYPES OF SUPERPLASTICIZERS

The currently available superplasticizers in North America may be classified as:

- (i) sulphonated melamine formaldehyde condensates
- (ii) sulphonated naphthalene formaldehyde condensates
- (iii) modified lignosulphonates

Sulphonated Melamine Formaldehyde Condensates

These are primarily of German origin, the

best known being Melment L10. It is usually available as a 20% aqueous solution with a density of 1100 kg/m^3 and is limpid to slightly turbid or milky in appearance. The chloride content is 0.005%. Modified types have recently been introduced into North America.

Sulphonated Naphthalene Formaldehyde Condensates

These were first developed in Japan but lately a number of manufacturers in North America are producing various modified versions. The best known brand is Mighty 150 of Japanese origin, the others, Lomar D and Sikament, are of North American origin. Mighty 150 is available as a dark brown 42% aqueous solution with a density of 1200 kg/m^3 . The chloride content is negligible.

Modified Lignosulphonates

These are available from a number of sources, the best known being Mulcoplast of Canadian origin. It is usually available as a light brown 20% aqueous solution with a density of 1100 kg/m^3 . It contains no chloride.

All three superplasticizers are made from organic sulphonates of the Type RSO_3^- where R is a complex organic group frequently of high molecular weight (Fig. 1).

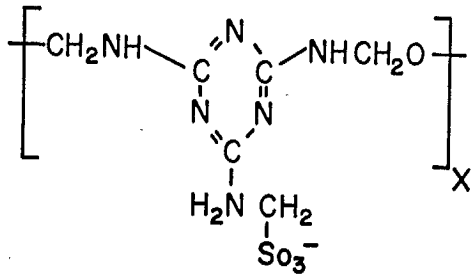
MODE OF ACTION OF SUPERPLASTICIZING ADMIXTURES

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):

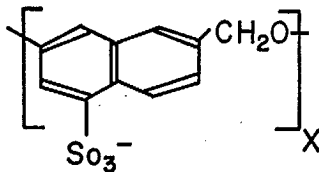
"These admixtures are thought to be adsorbed onto cement particles, causing them to become mutually repulsive as a result of the anionic nature of superplasticizers, which causes the cement particles to become negatively charged. In principle, this adsorption and dispersing effect is similar to that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

From their recent studies Daiman and Roy (8) have concluded that the water reducing effect of superplasticizers investigated (of the sulphonated melamine and naphthalene formaldehyde

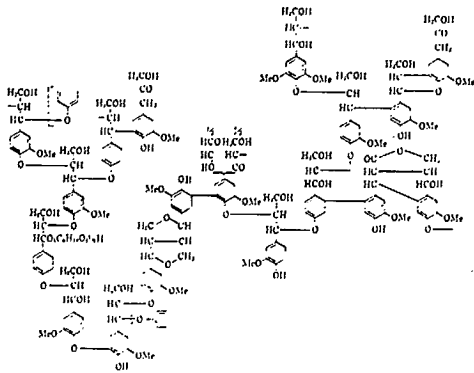
condensate types) is caused mainly by the change in zeta potential as a function of surfactant adsorption onto the cement surfaces.



R = MELAMINE-FORMALDEHYDE



R = NAPHTHALENE-FORMALDEHYDE



LIGNOSULPHONATE R = LIGNIN

METHODS OF USING SUPERPLASTICIZERS

There are three possible ways in which superplasticizers may be used in concrete:

- To produce concrete with very low water to cement ratio. To achieve high strength concrete, water content of the mix is reduced while maintaining the same cement content. The reduced workability is compensated for by incorporating superplasticizers. By this method water reductions of up to 30% can be achieved and concrete with water to cement ratios as low as 0.28 have been successfully placed. This is the most common method of using superplasticizers in Japan.
- To produce concretes with reduced cement content. Superplasticizers can be used to produce concretes with reduced cement contents while the water to cement ratio is maintained constant. As in method (a), the decrease in workability of concrete is achieved by incorporating superplasticizers. This method has found appeal in North America because of the desire to reduce cement content of the mixes in the context of energy conservation.
- To produce flowing concretes. Superplasticizers have been used to produce self-compacting, self-levelling flowing concretes. In this application no attempt is made to reduce either the water to cement ratio or the cement content. Instead, the aim is to increase the slump from, say, 75 to 200 mm without causing any segregation so that the concrete can be placed in heavily steel-reinforced sections.

MIX PROPORTIONS FOR SUPERPLASTICIZED CONCRETE

The use of superplasticizers as water reducers does not require any significant change in the mix proportioning and water reductions as high as 30% can be obtained. In the case of flowing concretes there appears to be a need for

Fig. 1 - R-organic group for naphthalene formaldehyde, melamine formaldehyde, and lignosulphonate; from reference 9

additional fine material to avoid segregation. This can be achieved by increasing the percentage of the finer fractions of sand by 4 to 5% or by the addition of pozzolanic materials such as fly ash. To arrive at the correct mix proportions laboratory trial mixes are necessary and additional fine material may also be required.

It has been observed that melamine and naphthalene-based superplasticizers cause some loss of entrained air whereas lignosulphonate based materials sometimes result in an increase in air content (9). The loss of air in flowing concretes can be explained by the lower viscosity of the superplasticized concrete, but a similar phenomenon has been noticed in water-reduced concretes (10). It is therefore important that correct quantities of air-entraining agents in superplasticized concrete be determined by trial mixes. It is possible that certain types of air-entraining agents may not be compatible with certain types of superplasticizers. This aspect should be taken into account when materials are being selected.

DOSAGE REQUIREMENTS

The dosage requirements of superplasticizers will depend upon whether flowing or water-reduced high strength concrete is required. Generally the dosage is relatively high when compared with that required for conventional water reducers and can vary from 0.5 to 3% by weight of cement. The need for high dosages makes the superplasticizers considerably more expensive than conventional admixtures.

It is generally recommended by manufacturers that the superplasticizers be added to the concrete mix just before its discharge from the mixer. This is because the increased slump due to the superplasticizers is maintained for only about 30 min. This loss of slump with time is a serious problem for ready-mixed concrete producers, which is further compounded by the fact that most North American specifications usually discourage the addition of admixtures to ready-mixed concrete at a job site. This explains the

slow acceptance of superplasticizers by this segment of the concrete industry.

In precast concrete plants, where the time lag between mixing and placing of concrete is only minutes, the superplasticizers can be added to the mix at the batch plant.

MEASUREMENT OF WORKABILITY OF SUPERPLASTICIZED CONCRETE

In North America, consistency of concrete is usually measured by the slump test. This test gives satisfactory results when slumps are below 150 mm, beyond which the use of the slump test can be questioned. In spite of this, researchers have used slump tests to characterize flowing concrete. The rationale, which has some merit, has been that the introduction of a new test to measure the flow of superplasticized concrete - itself a new concept - will only complicate matters. However, in Germany a "flow table" specified in German standard DIN 1048 (1972) is employed to measure the consistency of flowing concrete. Details of the flow table are given in Appendix A. A fixed amount of concrete is cast in a mould placed on a smooth surface and the mould is then jolted in a standard manner. After jolting, the radial spread of the concrete is measured.

It is doubtful if the flow table test is superior to the slump test and further research is indicated.

A relationship between the results of slump and flowtable tests is given in Fig. 2 (11).

EFFECTS OF SUPERPLASTICIZERS ON FRESH CONCRETE

A number of investigators have reported the effects of superplasticizers on the properties of fresh concrete. Some of the more significant data are presented below.

SEGREGATION AND BLEEDING

There is no undue segregation or bleeding of concrete when superplasticizers are incorporated into concrete at dosages recommended by manu-

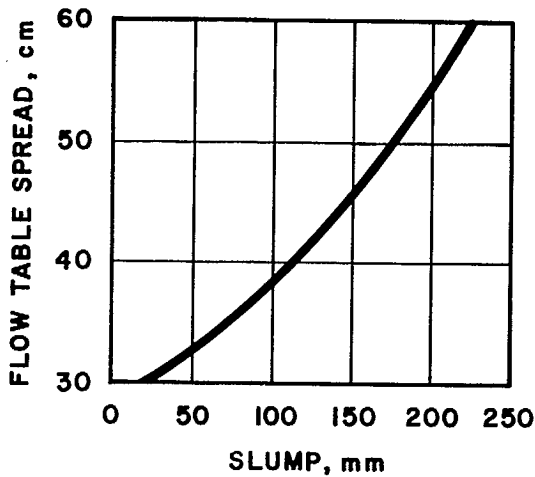


Fig. 2 - Relation between flowtable spread and slump; from reference 11

facturers. Data on bleeding for the three types of superplasticizers are presented in Fig. 3. Extra precautions may be needed if superplasticized concretes are to be placed using conveyor belts which may cause segregation of flowing concrete.

SETTING TIME

A number of reports have been published on the effects of superplasticizers on the setting time of fresh concrete (5,10). Figure 4 presents results of time-of-set studies for water-reduced superplasticized concretes made with different types of cements.

VIBRATION REQUIREMENTS

A number of studies show that when superplasticizers are added to concrete at the manufacturers' recommended dosages, the 28-day compressive strengths of test cylinders are equal to or greater than the corresponding strengths of cylinders cast from the reference mix (9). 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 resulting in considerable possible

savings of time and money. Compressive strength data for test cylinders cast with and without vibration are shown in Fig. 5.

It should be noted that concretes incorporating lignosulphonate-based superplasticizers show some loss in strength compared with test specimens cast from the control mix. This is probably due to the higher air content of the concrete mixes, which is a direct result of this type of superplasticizer.

AIR CONTENT OF FRESH CONCRETE

The entrained air content of fresh superplasticized concretes may show some decrease with time (Fig. 6). However, this may not be so for concretes superplasticized with lignosulphonate-based products (9). It is therefore imperative that air content of concrete be determined immediately after mixing and before casting test specimens.

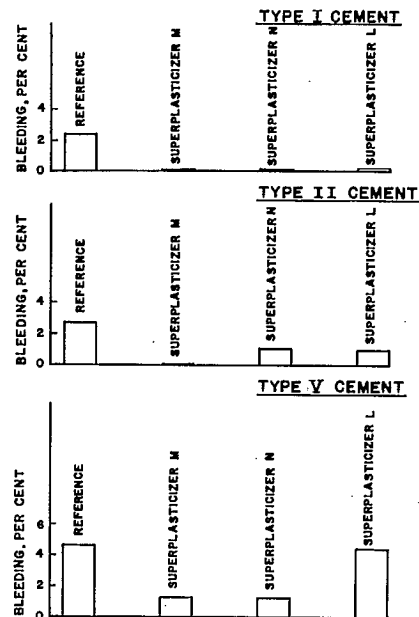


Fig. 3 - Bleeding characteristics of reference and superplasticized concretes; from reference 10

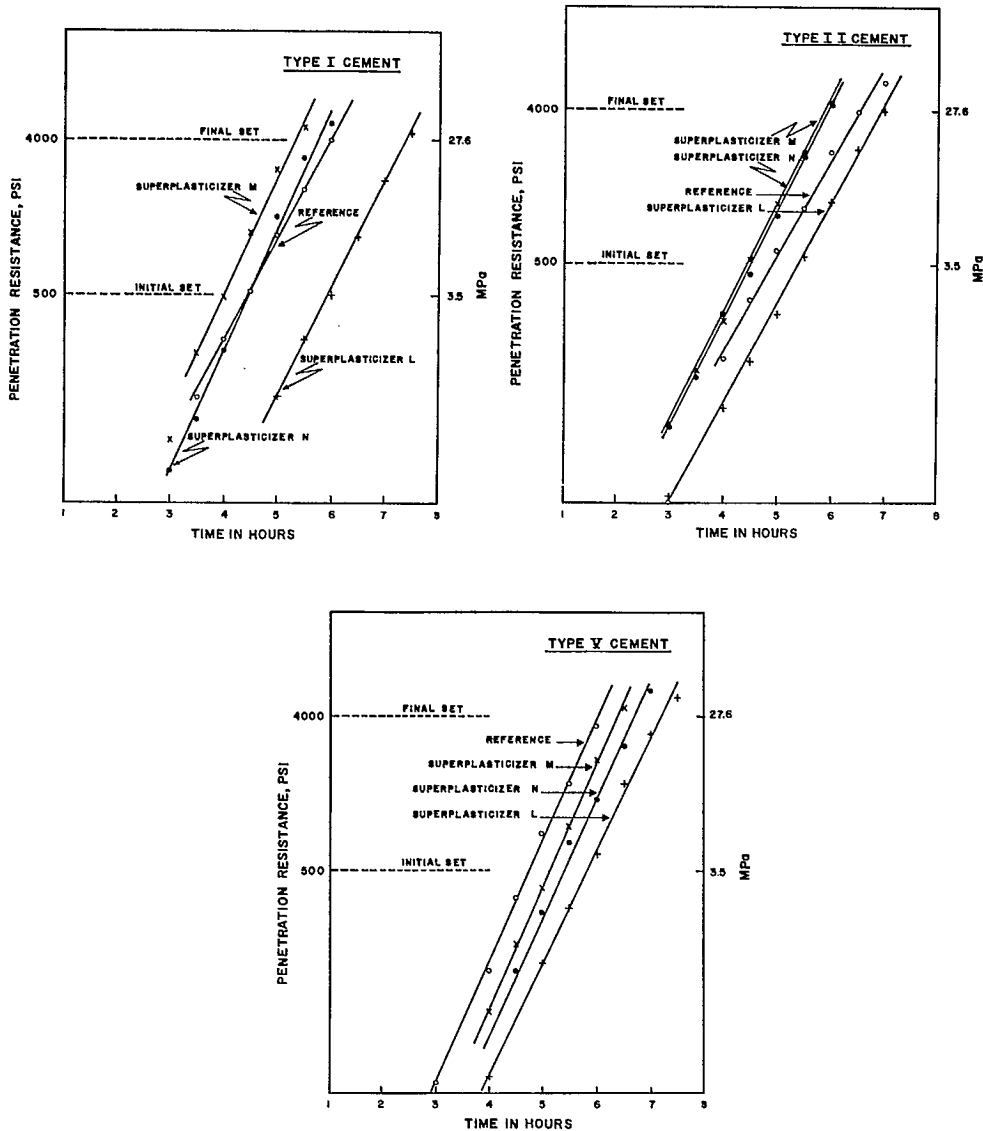


Fig. 4 - Setting time characteristics of reference and superplasticized concretes; from reference 10

INCREASES IN SLUMP AND ITS LOSS WITH TIME

Superplasticized concretes exhibit large increases in slump. However, this increase is of short duration and within 30 to 60 min. the concrete reverts back to its original consistency (Fig. 7). The rate of loss of slump is dependent on the type of superplasticizer, its dosage rate, temperature of the concrete and on the type of cement. Figure 8 presents some of the data for

concrete having low and high water to cement ratios and made with different types of cement.

Mechanism of Slump Loss

The rapid loss of slump of superplasticized concrete with time is a serious disadvantage and research is being continued to find a solution to this problem. The reasons for loss of slump with time are not clear but may be attributed to

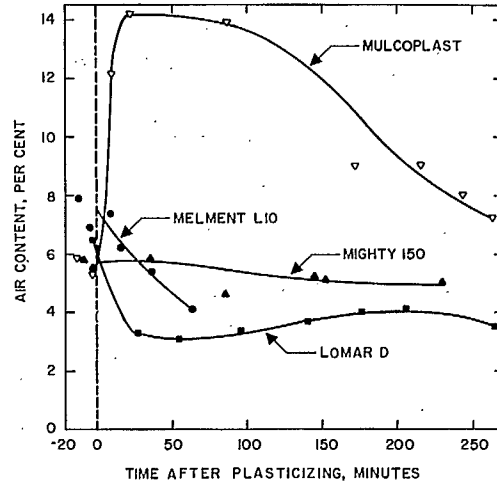
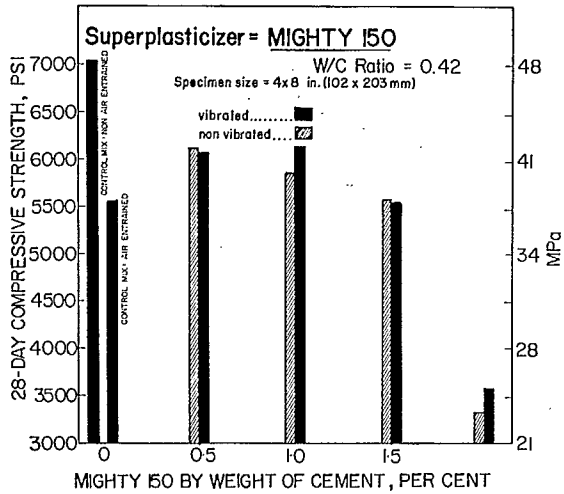


Fig. 6 - Change of air content with time after addition of admixtures; from reference 25

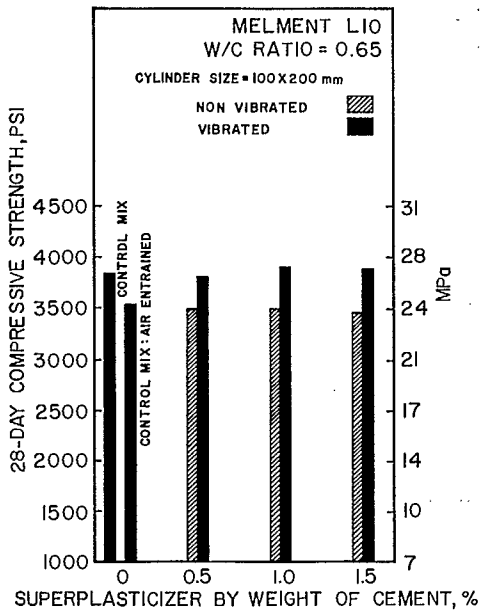


Fig. 5 - Compressive strengths of vibrated and non-vibrated cylinders at 28 days, Top W/C = 0.42, Bottom W/C = 0.65; from references 9, 31

chemical and physical changes in the cement paste of fresh concrete. Hattori has tried to explain the slump loss phenomenon as follows (12).

"...the coagulation of hydrated cement particles in the dormant stage in the paste is playing a more important role in the slump loss than the chemical bonding which may be

formed between the particles through hydration process. Agreement between the forms of equations partially based on Smoluchowski's theory and the corresponding experimental results are considered to support the above conclusion."

Effect of Repeated Dosage on Slump Loss

To overcome the problem of rapid slump loss with time, a number of investigators have shown that large increases in slump of superplasticized concretes can be maintained for several hours by the addition of a second dosage, the amount of second and third dosage being the same as the initial (12,13). In studies reported by Malhotra, the addition of a third dosage is not recommended because the concrete, although maintaining its consistency, loses its workability (Fig. 9) (13).

Hattori has studied the behaviour of cement paste after dosing it repeatedly with a naphthalene-based superplasticizer (14). His data show that the amount of adsorption and zeta potential increased at every redosing, and viscosity decreased at the same time (Fig. 10). The microphotographs also showed clearly the change from a coagulated to a redispersed state before and after redosing. It is evident from Fig. 10

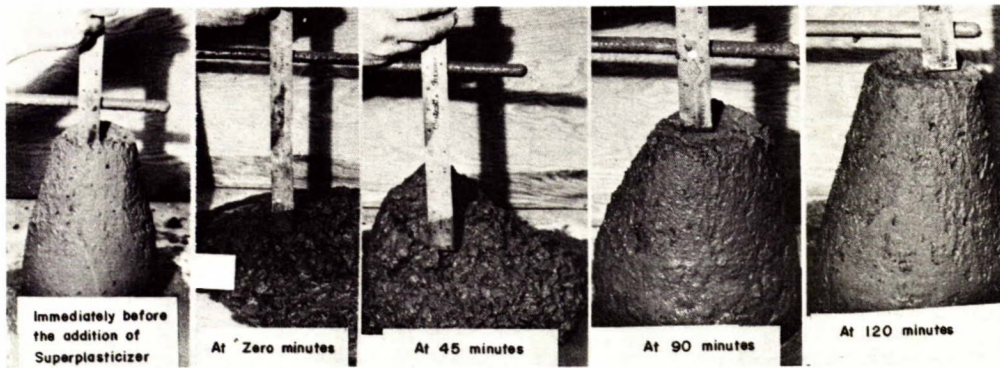


Fig. 7 - Slump tests after various intervals of time for concrete incorporating 3% Melment L10 by weight of cement; from reference 9

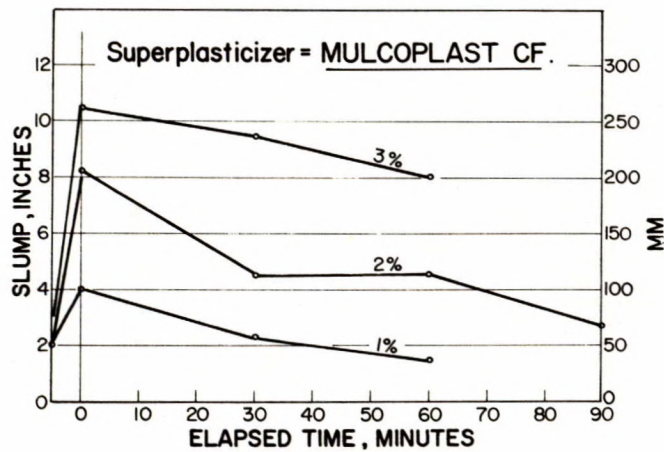
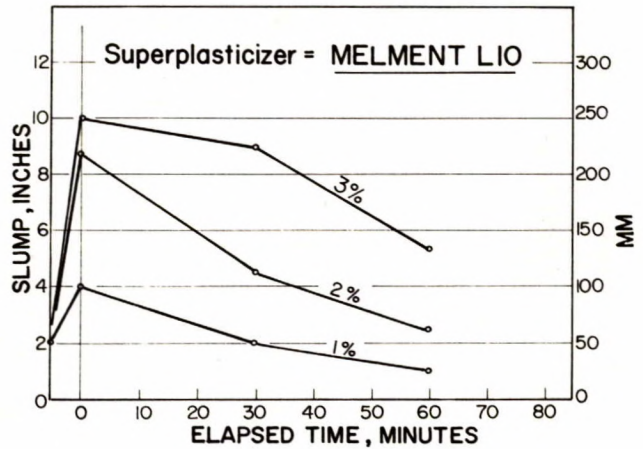
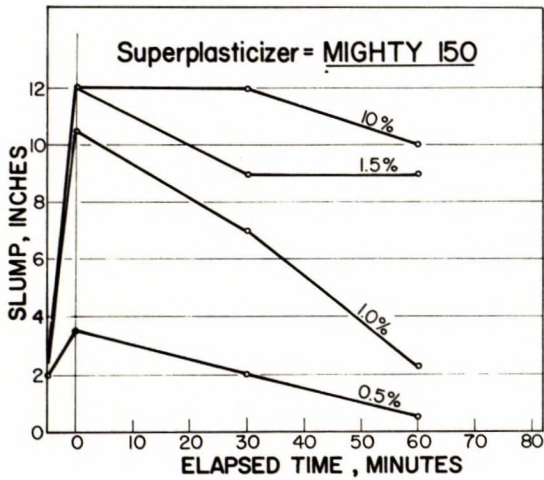


Fig. 8 - Loss of slump with time; from references 9, 31

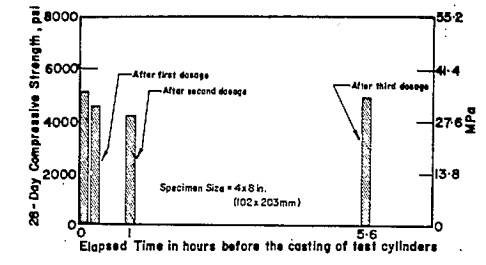
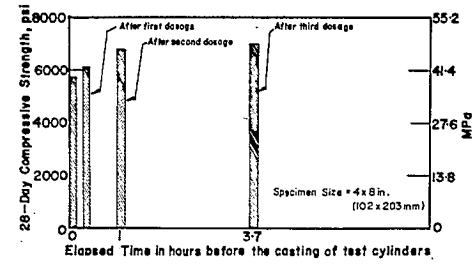
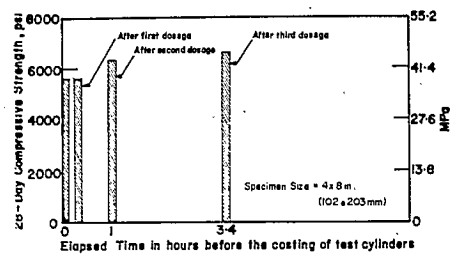
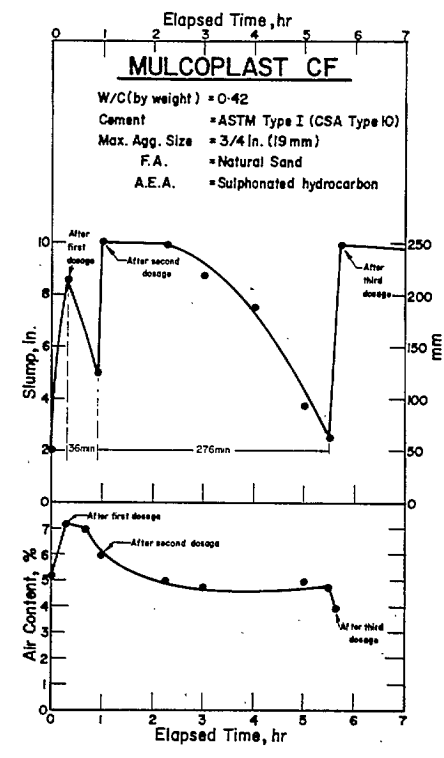
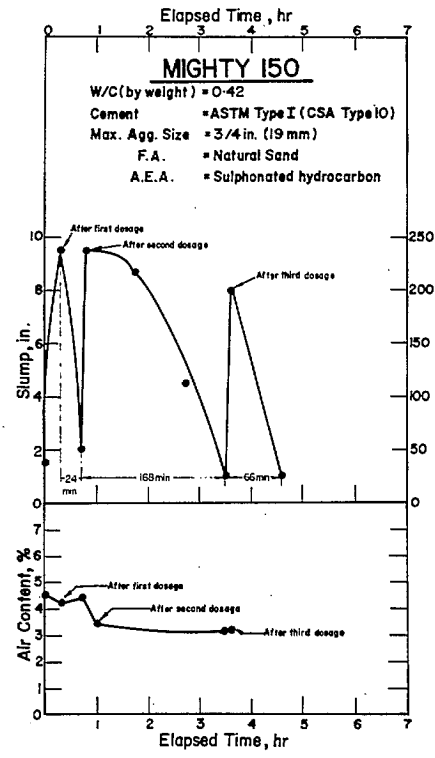
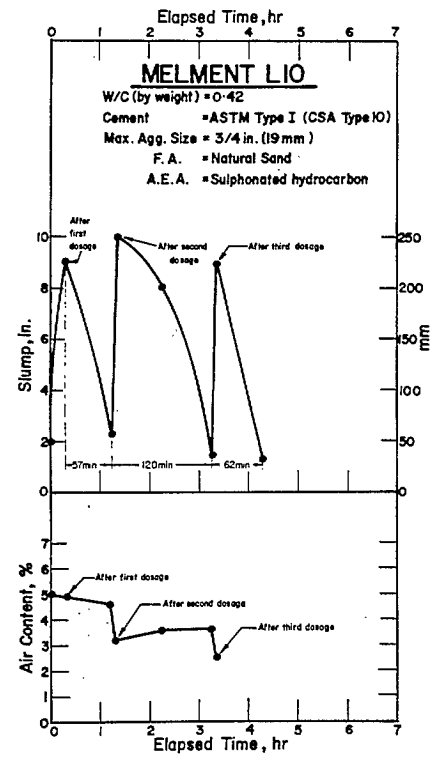


Fig. 9 - Effect of repeated dosages of superplasticizers on slump, air content and 28-d compressive strength of concrete; from reference 13

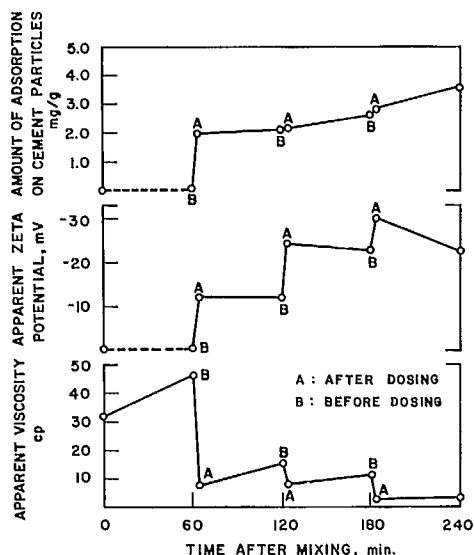


Fig. 10 - Mechanism of fluidizing of repeatedly dosed concrete; from reference 12

that higher zeta potential was required to redisperse the coagulated particles than for the previous dispersion. The effect of repeated dosing of concrete to maintain slump at about 200 mm is shown in Fig. 11. It should be noted that the repeated dosages used by Hattori were 20% of the initial dosage.

Slump Loss of Concrete Incorporating Fly Ash

Ryan and Munn have investigated concrete mixes in which all or part of the cement binder had been replaced by the same volume of fly ash to determine whether the fluidity of the superplasticized mix was primarily affected by physical or chemical changes in mix components (15). The test results indicated the rate of slump loss with time is not greatly affected when the binder is a mixture of cement and fly ash regardless of the ratio between the two. However, when all of the cement is replaced by fly ash, the addition of superplasticizer increases the slump of the mix in the usual manner but the rate loss with time is greatly reduced compared with mixes incorporating cement alone or a mixture of cement and fly ash (Fig. 12). Based upon the above, Ryan and Munn concluded that the initial superplasticizing

effect is a physical one, which is then subject to a chemical interaction between the cement (or lime) and superplasticizer resulting in the short "pot" life of the superplasticized concrete (15).

Effect of Sulphate Content of Cement on Slump Loss

Khalil and Ward have investigated the effect of sulphate content on slump loss of concretes containing superplasticizers (16). Their investigations indicate there is an optimum range of SO_3 content for which slump loss is minimized both at normal and elevated temperatures. Their data show that at $40^\circ C$, slump is relatively unaffected until 60 min. after mixing for the modified mixes, whereas at the as-received SO_3 content the concretes started to lose slump to a significant degree after 30 min. In addition, the short- and long-term compressive strengths showed an improvement of about 12% at the optimum sulphate content. No data, however, are provided for the durability of concrete containing the modified cement both in freeze-thaw tests and in soils containing sulphates.

Effect of Temperature on Slump Loss

Several investigators have reported the effect of temperature on slump loss of concrete (17,18). The increase in temperature from 15.5 to $32.2^\circ C$ dramatically increases the rate of slump loss. At $32.2^\circ C$ the superplasticized concrete

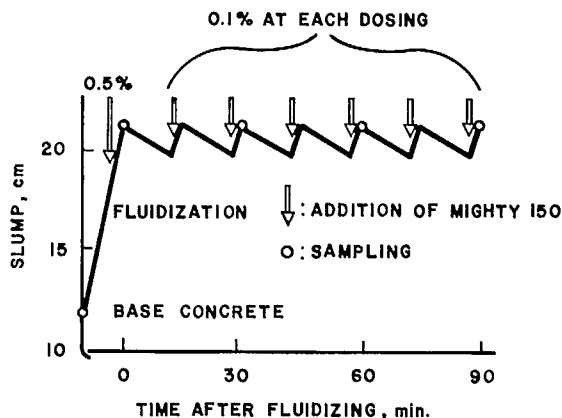


Fig. 11 - Repeated dosing of superplasticizer to control slump at 210 mm; from reference 12

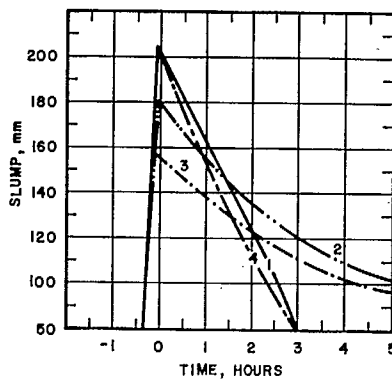
loses more than 50% of its slump in less than 30 min. (Fig. 13).

Colleparidi and Corradi have referred to the development of a naphthalene-based superplasticizer that overcomes the slump loss problem to some degree and have published extensive data on the properties of concrete incorporating it (19, 20). Some results of changes in slump as affected by time and temperature are shown below (20).

Time (h)	Slump of superplasticized concrete (mm)		
	4°C	21°C	42°C
0	220	220	210
0.5	205	200	195
1	210	195	185
2	210	200	150
4	185	140	30

Effect of C₃A Content of Cement on Slump Loss

There is some evidence that concretes made with cements having moderate to high C₃A



MIX NO.	CEMENT INCLUDED	ADMIX. INCLUDED	INITIAL SLUMP
1.	100%	YES	80 mm
2.	NIL	YES	80 mm
3.	NIL	NO	165 mm
4.	40%	YES	80 mm

Fig. 12 - Rate of slump loss of "flowing" concrete with mixes containing fly ash; from reference 15

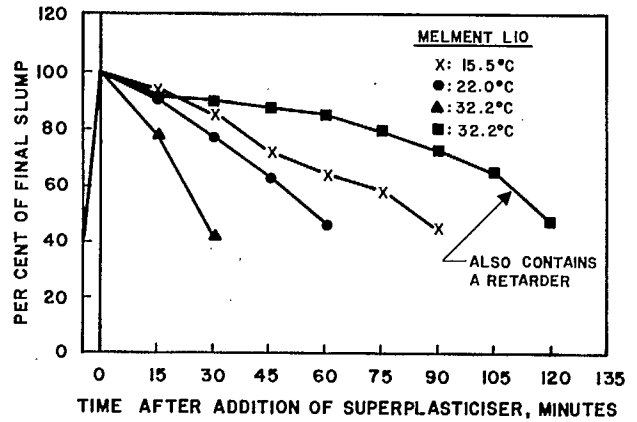


Fig. 13 - Effect of variation in temperature on slump loss; from reference 18

contents (C₃A > 9.0%) show increased slump loss over that of control concrete (21). The contrary appears to be true for concrete made with cements having a low C₃A content (C₃A < 5.0%). However, according to Perenchio et al., the use of low C₃A cement is no guarantee that slump loss will be reduced (21).

Mailvaganam has shown that concretes made with Type V cements (C₃A = 2.6%) show slightly lower rates of slump loss than those made with Type I cements having C₃A content varying from 6.9 to 12.6% (Fig. 14) (18).

PUMPABILITY OF SUPERPLASTICIZED CONCRETE

Kasami et al. have investigated the pumpability of superplasticized concrete under field conditions (22). In one experiment about 200 m³ of normal and lightweight aggregate concrete, involving 14 mixes with and without superplasticizers, was pumped horizontally. The pumping distance using an IHL-PTF 85 pump was 109 m and the line diameter was 125 mm. The dosage of the naphthalene-based superplasticizer was in the range of 0.4 to 0.7% by weight of cement and concrete mixing was done in ready-mixed agitator-type trucks. After the addition of the superplasticizer, the mixer was rapidly agitated for one minute. Following this, the concrete was pumped at the rates of 10, 20, 30, 40, 50 and 60 m³/h. Pump pressure and line pressure were measured at

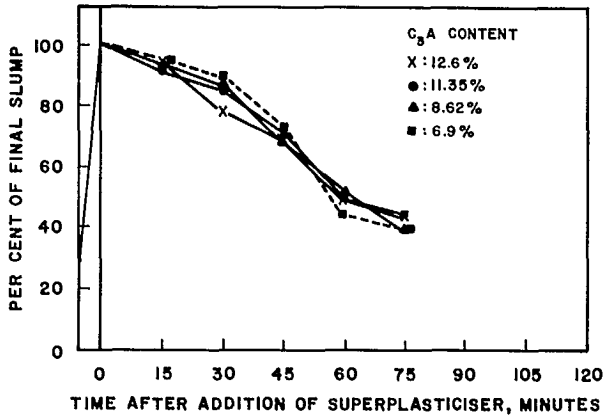


Fig. 14 - Effect of variation in C₃A content (Type I Cements) on slump loss; from reference 18

each pumping rate. The slump, air content and other properties of fresh and hardened concrete were measured on all reference superplasticized and pumped concretes. The test data indicated that pumping pressure and line pressure loss for normal weight concrete were reduced by about 30%, whereas those for lightweight concrete were reduced by no more than 10%. The increase in pumping resistance with the increase in pumping rate was less than that for conventional concrete. Data on slumps of conventional and superplasticized pumped concretes are shown in Fig. 15. Line pressure distribution is shown in Fig. 16.

EFFECT OF SUPERPLASTICIZERS ON THE PROPERTIES OF HARDENED CONCRETE

In North America, considerable data have been accumulated on the effect of superplasticizers on the properties of hardened concrete for use both in cast-in-place and precast construction. Because of severe climatic conditions in Canada and the northern United States, much attention has been devoted to the performance of superplasticized concrete under freeze-thaw conditions. Results of some of the significant studies are presented below.

ACCELERATED STRENGTH TESTING

Several studies have been reported dealing with the effect of superplasticizers on accelerated strength testing (23,24,25). It has been shown that their incorporation does not affect the relationship between the accelerated and 28-day compressive strengths (24)(Fig. 17). The modified boiling test, ASTM C684 (Procedure B), was the accelerated strength test used in the studies referred to above.

MECHANICAL PROPERTIES OF CONCRETE

It has been shown that water reductions of up to about 20% can be achieved in the manufacture of concrete when superplasticizers are incorporated in concrete as water reducers. The increase in mechanical properties, i.e., compressive and flexural strength and modulus of elasticity, is generally commensurate with reductions in water to cement ratio although some exceptions have been noted (25). The ability of superplasticizers to reduce water and achieve very high strengths is of special importance for the precast concrete industry where high early strengths are needed for rapid turnover of formwork. Table 1 presents compressive strength data for water-reduced concretes (9).

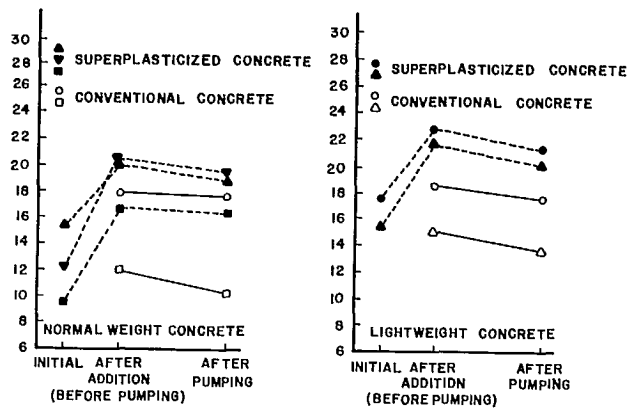


Fig. 15 - Slump of conventional and superplasticized pumped concretes; from reference 22

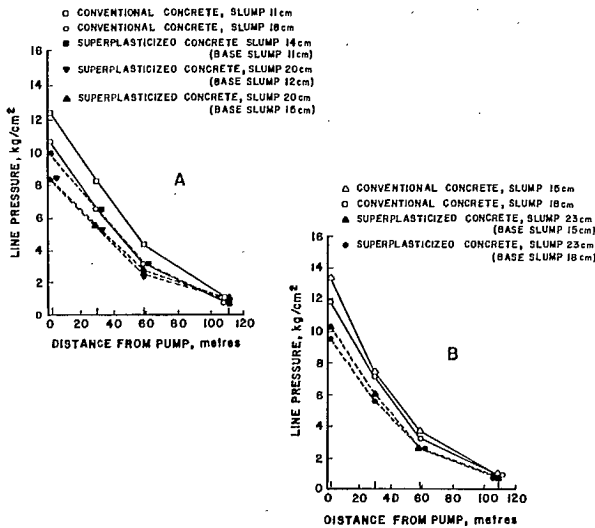


Fig. 16 - Line pressure distribution for normal and lightweight superplasticized concretes; A - normal weight concrete, B - lightweight concrete; from reference 22

In one study Johnston et al. reported that when the water to cement ratio was kept constant, the concretes incorporating superplasticizers had higher compressive strengths than the reference concrete (Table 2) (25). This led to the conclusion that the admixtures do indeed affect the hydration process. Further research is indicated to determine the effects of superplasticizers on the rate of hydration of cement pastes.

SHRINKAGE AND CREEP OF SUPERPLASTICIZED CONCRETE

Data on shrinkage and creep of concrete incorporating superplasticizers are rather limited. The available data indicate that the shrinkage compares with or is less than the shrinkage of reference concrete, though there are exceptions. Generally the shrinkage of test prisms is well below the maximum requirement of ASTM C494 (26). The relationship between moisture loss and shrinkage for water-reduced superplasticized concrete is shown in Fig. 18.

Data on creep of superplasticized concretes have been published by Ghosh and Malhotra, Johnston et al. and Brooks et al. (10,25,27). However, in each case, different concrete, loading and moisture conditions have been used, thus making direct comparison rather difficult. The general consensus appears to be that concretes made with superplasticizers have approximately the same creep as the reference concrete, though there are exceptions. However, under severe drying conditions superplasticized concretes may show higher creep and this may have to be taken into consideration when designing structures subject to very high stresses. Some creep data are shown in Table 3.

DURABILITY OF SUPERPLASTICIZED CONCRETE

Resistance to Freeze-Thaw Cycling

It is well known that for satisfactory freeze-thaw durability of concrete the cement paste should be protected with air bubbles, which result when adding an air-entraining agent. Adequate protection requires 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 200 μm. Investigations by leading research laboratories in North

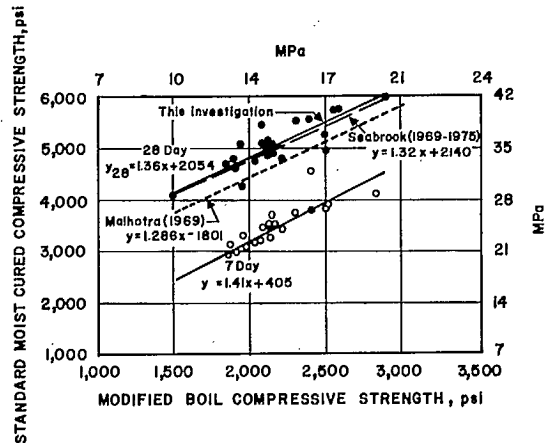


Fig. 17 - Relationship between accelerated, 7-day and 28-day compressive strengths of superplasticized concrete; from reference 24

Table 1 - Mix proportions and properties of fresh and hardened concrete - water reduced*

		Mix proportions and properties of fresh concrete					Properties of hardened concrete				
Mix series	Type of concrete	Water content kg/m ³	W/C ratio	Entrained air %	Slump mm	Density kg/m ³	Compressive strength of 150 x 300-mm cylinders			Flexural strength of 90 x 100 x 400-mm prisms MPa	Modulus of elasticity MPa x 10 ⁻⁴
							7 days	28 days	91 days		
A1	Reference (Type I cement)	147	0.49	5.2	75	2348	26.8	32.8	37.8	6.1	3.2
2	Type I cement & SP-M	120	0.40	5.6	80	2362	37.3	44.0	48.5	7.0	3.7
3	Type I cement & SP-N	120	0.40	6.0	70	2350	35.5	39.3	47.6	7.0	3.7
4	Type I cement & SP-L	120	0.40	5.6	80	2360	36.3	42.6	49.9	6.6	3.6
B5	Reference (Type II cement)	147	0.49	4.9	85	2354	25.6	36.6	42.4	6.0	3.2
6	Type II cement & SP-M	120	0.40	5.6	90	2362	36.3	47.6	55.0	6.9	3.7
7	Type II cement & SP-N	121	0.40	5.3	75	2377	36.9	47.6	55.8	7.2	3.7
8	Type II cement & SP-L	121	0.40	4.8	75	2385	35.0	47.6	55.8	7.3	3.6
C9	Reference (Type V cement)	144	0.48	5.4	90	2352	19.1	32.2	38.0	5.0	3.2
10	Type V cement & SP-M	117	0.38	5.4	75	2364	31.9	40.3	46.2	6.2	3.6
11	Type V cement & SP-N	118	0.38	5.3	80	2381	33.0	42.0	48.5	5.7	3.5
12	Type V cement & SP-L	118	0.38	5.2	85	2379	32.8	42.4	50.3	6.2	3.5

*From reference 10.

Notes: 1. Coarse aggregate was crushed limestone with a maximum size of 19 mm. Fine aggregate was natural sand. Air entraining agent was sulphonated hydrocarbon type. Size of cylinders was 150 x 300 mm.

2. M, N and L refer to melamine, naphthalene and lignosulphonate type superplasticizers (SP).

Table 2 - Percentage increase in compressive strength of superplasticized concrete relative to control mix for constant water/cement ratio*

Age days	Increase in strength relative to control mix, %			
	Concrete containing superplasticizer A	Concrete containing superplasticizer B	Concrete containing superplasticizer C	Concrete containing superplasticizer D
7	28.6	33.8	5.4	20.8
28	10.9	18.1	6.5	3.3
183	13.7	28.0	7.6	3.3
365	15.8	13.7	5.4	1.6

*From reference 25.

Notes: 1. All mixes including control had a water to cement ratio of 0.50, a slump of 100 ± 25 mm and an air content of 6 ± 1% immediately before the casting of test specimens.

2. Maximum size of gravel was 13 mm and cement was ASTM Type I.

3. The mix proportions for the control and superplasticized mixes were as follows:

	C.A.	F.A.	Cement	Water
(i) Control mix, kg/m ³	882	813	393	196
(ii) Superplasticized mix kg/m ³	872	982	290	145

4. Superplasticizer A refers to a melamine formaldehyde condensate.

Superplasticizer B refers to a high M.W. naphthalene condensate of Japanese origin.

Superplasticizer C refers to a sulphonated polymer of Canadian origin.

Superplasticizer D refers to a polymerized naphthalene condensate of U.S.A. origin.

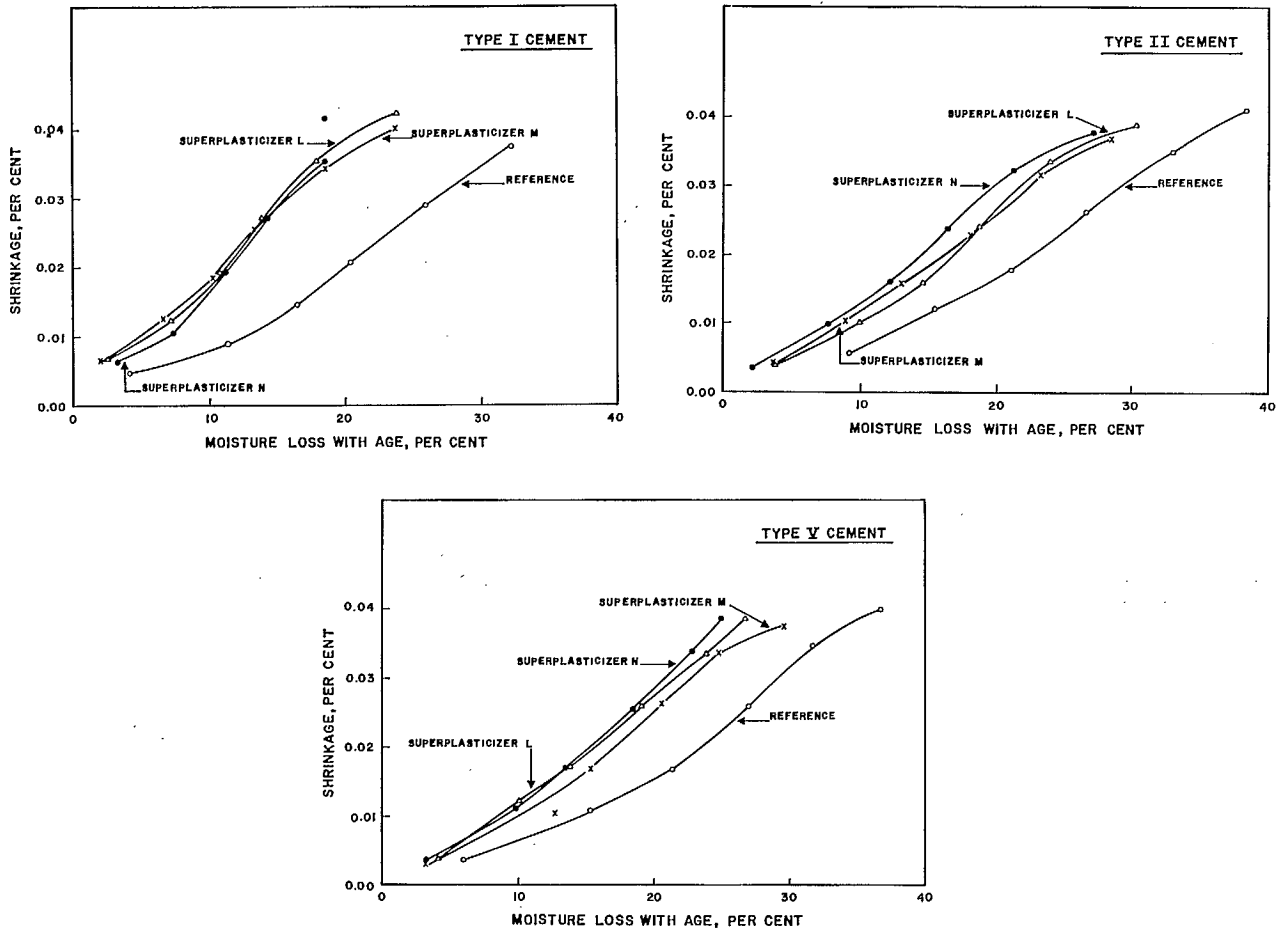


Fig. 18 - Shrinkage versus moisture loss with age for reference and superplasticized concretes; from reference 10

Table 3 - Data on creep measurements for control and water-reduced superplasticized concrete*

Mix series	Admixtures		Mix proportions			Properties of fresh concrete				Creep measurements on 150 x 300-mm cylinders				
	Superplasticizer type	Superplasticizer AEA** mL/kg of cement	W/C ratio by weight	Type I cement kg/m ³	F.A. kg/m ³	C.A. kg/m ³	Entrained air %	Slump mm	Density kg/m ³	f'c at 28 days MPa	f'c at 63 days MPa	Stress applied MPa	Stress/strength ratio	Total*** creep strain in./in. x 10 ⁶
1	Control	-	0.49	298	817	1082	5.3	80	2344	34.3	37.4	15.2	0.44	1101 at 338 d
2	Melamine	23.6	0.40	304	835	1106	5.4	75	2365	45.2	50.8	19.6	0.43	1085 at 324 d
3	Naphthalene	9.1	0.40	303	832	1102	6.0	80	2357	47.4	51.1	20.3	0.43	1107 at 345 d
4	Lignosulphonate	25.6	0.40	305	839	1111	5.4	40	2377	46.0	48.7	19.8	0.43	1157 at 339 d

*From reference 10.

**Air-entraining agent.

***Total creep strain is obtained by subtracting shrinkage and elastic strain at loading from the strain readings.

America have indicated that in concretes incorporating superplasticizers, the above value is generally exceeded (9,10,21,25,26,28,29,30,31). However, in spite of the increased bubble spacing, the freeze-thaw durability of concrete when tested in accordance with ASTM Standard C666, Procedure A or B, is usually not impaired (Tables 4-7). Some exceptions have been reported (10,28). In one investigation in Canada, concrete incorporating a melamine-based superplasticizer and made with ASTM Type V cement performed rather poorly in a freeze-thaw test (10); in another investigation in the U.S.A., concrete incorporating the same type of superplasticizer and made with ASTM Type I cement failed to meet the ASTM freeze-thaw requirements (28). It appears that bubble spacing factor limitations stipulated for air-entrained concrete may or may not be valid for concretes incorporating superplasticizers. Research is needed to delineate how these parameters can be correlated with the performance of concrete under freeze-thaw cycling.

Resistance to Salt Scaling

The limited available data on resistance to salt scaling indicate that the performance of concrete slabs incorporating a melamine-based

superplasticizer compares well with that of the reference slabs. The maximum weight loss of 0.05 g/cm^2 shown in Fig. 19 is below the maximum allowed limit of 0.08 g/cm^2 (30).

Resistance to Sulphate Attack

Brooks et al. have investigated the performance of superplasticized concrete when exposed to magnesium sulphate solution. The concentration of the sulphate solution was maintained at a nominal 3.0% as SO_3 content (20,27). Brooks et al. used a naphthalene-based superplasticizer and the test specimens, 100 x 100 x 500-mm prisms, were exposed to the sulphate solution for 300 days (27). The durability was monitored by determining changes in weight, length and dynamic modulus of the specimens. The investigations indicated that the performance of the superplasticized concrete was no different from that of the control specimens. Similar results have been reported by Colleparidi et al. (20).

INFLUENCE OF SUPERPLASTICIZERS ON STEEL-CONCRETE BOND STRENGTH

Colleparidi and Corradi have published data on the influence of superplasticizers on steel-concrete bond strength (19). The incorpor-

Table 4 - Effect of superplasticizers on freezing and thawing of flowing concrete* - W/C ratio 0.42

Mix No.	Type of superplasticizer and dosage in % by weight of cement	Mix proportions			Properties of fresh concrete					Durability factor***	
		Water kg/m^3	Cement kg/m^3	W/C ratio	Slump mm	Air content %	Air void system**			% DF RDF	
							A, %	α, cm^{-1}	$\bar{L}, \mu\text{m}$		
1	Control, without AEA	169	402	0.42	45	2.1	-	-	-	-	-
2	Control, with AEA	170	405	0.42	45	4.8	4.9	340	150	100	
3	Melment L10 1%	158	376	0.42	100	5.2	5.8	246	180		100
4	2%	157	374	0.42	230	5.2	4.3	209	250		100
5	3%	158	376	0.42	260	4.8	3.5	346	150		100
6	Mighty 150 0.5%	159	379	0.42	90	5.0	4.4	243	200		>100
7	1.0%	158	376	0.42	260	4.8	4.1	226	230		100
8	1.5%	158	376	0.42	260	3.4	2.8	308	200		>100
9	Mulcoplast CF 1%	158	376	0.42	100	6.0	4.2	362	150		>100
10	2%	158	376	0.42	210	6.8	7.1	234	180		>100
11	3%	158	376	0.42	260	6.0	5.1	310	150		100

*From reference 9.

**Modified point count (ASTM 457).

***ASTM C666, Procedure B.

Table 5 - Effect of superplasticizers on freezing and thawing of flowing concrete* - W/C = 0.65

Mix No.	Type of superplasticizer and dosage in % by weight of cement	Mix proportions			Properties of fresh concrete					Durability factor***	
		Cement	Water	W/C ratio	Slump mm	Air content %	Air void system**			%	
						A, %	α , cm^{-1}	\bar{L} , μm		DF	RDF
1	Control, without AEA	180	275	0.65	50	2.8	2.2	55	127		-
2	Control, with AEA	162	248	0.65	50	5.0	6.5	166	250	95.6	
3	Melment L10, 1%	162	248	0.65	90	4.5	5.2	173	250		103
4	2%	162	244	0.65	210	3.5	4.9	128	360		101
5	3%	162	248	0.65	250	3.5	3.6	125	430		103
6	Mighty 150 0.5%	161	246	0.65	100	3.9	4.8	143	330		103
7	1.0%	161	246	0.65	210	3.4	3.0	141	410		102
8	1.5%	161	246	0.65	250	2.6	3.3	166	330		93
9	Mulcoplast CF 1%	162	248	0.65	80	5.8	5.6	137	310		104
10	2%	162	248	0.65	200	4.8	4.8	166	280		104
11	3%	162	248	0.65	250	4.6	5.0	150	310		104

*From reference 31.

**Modified point count (ASTM 457).

***ASTM C666, Procedure B.

Table 6 - Effect of melamine-based superplasticizers on freezing and thawing resistance of air-entrained concrete*

Series No.	Dosage of admixtures per 100 kg of cement		Cement (ASTM Type I) kg/m^3	Water kg/m^3	Entrained air %	Slump mm	Air void systems***			Durability factor****	
	Melamine-based superplasticizer	AEA** mL					A, %	α , cm^{-1}	\bar{L} , μm	DF	RDF
A	4564 mL	60.6	308	118	5.5	64	4.45	142	323	90	99
	0.667 kg	57.4	309	138	5.4	57	5.13	185	241	91	100
B	4564 mL	75.0	306	121	8.1	118	6.41	176	193	91	101
	0.667 kg	69.1	306	148	7.7	197	7.40	210	157	90	100

*Adopted from reference 29.

**Air-entraining agent.

***Modified point count (ASTM C457).

****ASTM C666, Procedure A.

ation of a superplasticizer into concrete improves the adhesion between steel and concrete for both normal and lightweight concretes (Table 8). For example, for normal concrete the addition of the superplasticizer raised the steel-concrete bond strength at 7 days from 1.2 to 3.5 MPa for smooth bars and from 15.0 to 27.5 MPa for twisted bars. Similar improvements were observed for lightweight concrete (Table 8).

CORROSION OF REINFORCEMENT IN SUPERPLASTICIZED CONCRETE

Hattori has published data showing that the incorporation of a naphthalene-based superplasticizer in concrete does not lead to any significant rust formation on reinforcement (12). In his investigations a series of reinforced concrete piles of 300-mm outer diameter, 70-mm wall thickness and 2300 mm in length was prepared. The

Table 7 - Freeze-thaw durability of superplasticized flowing concrete incorporating fly ash*

Durability factor** %		Relative durability factor, %
Control	Incorporating superplasticizer	
94.4	92.7	98

*Adopted from reference 26.

**All superplasticized specimens withstood 300 cycles of freezing and thawing in water in accordance with ASTM C666, Procedure A.

NOTE: The proportions for the control mix were:

- cement = 127 kg/m³
- fly ash = 104 kg/m³
- water = 109 kg/m³
- fine aggregate = 601 kg/m³
- coarse aggregate (19 mm) = 832 kg/m³

A standard water-reducing agent was used in the control mixture. An air-entraining agent was used for both control and mixes incorporating superplasticizers.

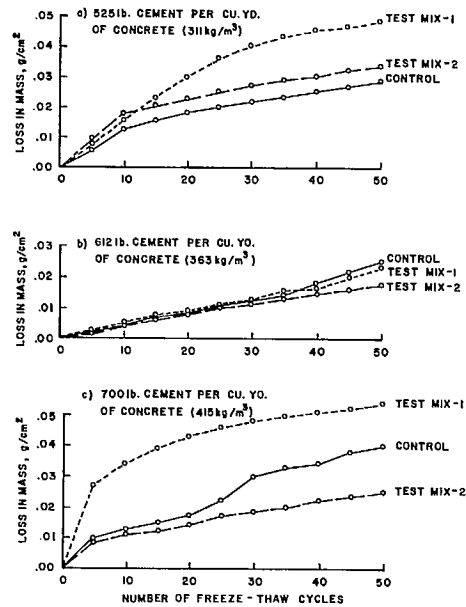


Fig. 19 - Summary of salt scaling test results, from reference 30

Table 8 - Data on steel-concrete bond strength*

Type of concrete	Type of admixture	Cement brand	Cement content	Slump	Bond strength, kg/m ²			
					7-day		28-day	
					Smooth bars	Twisted bars	Smooth bars	Twisted bars
Normal weight	None	Type I, Brand 1	400	100	12	150	13	152
Normal weight	Superplasticizer	Type I, Brand 1	400	220	35	275	40	285
Lightweight (1800 kg/m ³)	None	Type I, Brand 2	500	100	4	66	6	92
Lightweight (1800 kg/m ³)	Superplasticizer	Type I, Brand 2	500	210	9	142	21	210

*From reference 19.

effect of three types of admixtures, including a superplasticizer, was investigated. The reinforcement consisted of four steel bars. The piles were made by centrifugal forming and prestressing followed by curing in steam and autoclave. Following this, the piles were exposed to water for one year and left outdoors for four years. At the end of this period the reinforcements were taken out for evaluation of rusting. The test data are shown in Table 9.

It is seen from the table that, whereas reinforcement in superplasticized concrete showed only traces of rust, the reinforcement in concrete incorporating calcium chloride has a very high percentage of rust.

USE OF SUPERPLASTICIZERS IN PRECAST CONCRETE PRODUCTS

The superplasticizers are being increasingly used by the precast concrete industry. Principal reasons appear to be:

- To produce concrete products that achieve compressive strengths of the order of 40 MPa in 8 to 18 h.
- To reduce fuel costs associated with the use of heat in accelerated curing of precast products.
- To reduce the high cement contents prevalent in the precast industry.

Table 9 - Data on rusting of reinforcement embedded for five years in concrete piles*

Type of admixture	Dosage % by weight of cement	Rust coverage %
None	-	0.25
Mighty 150	0.6	trace
Lignosulphonate	0.25	0.1
CaCl ₂	0.05	0.3
CaCl ₂	0.5	0.6
CaCl ₂	1.0	11.7
CaCl ₂	2.0	19.6
CaCl ₂	4.0	75.0

*From reference 12.

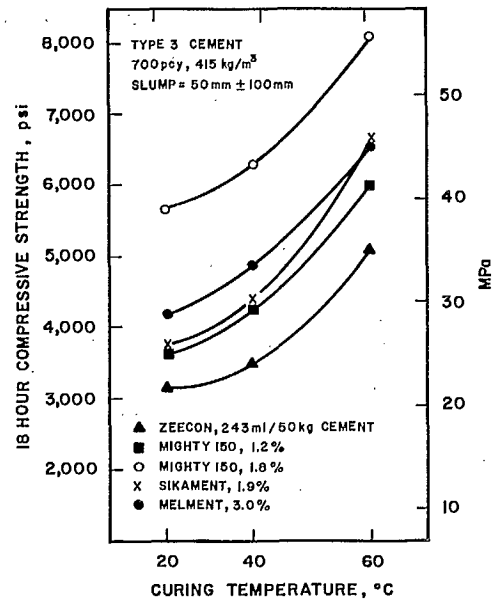


Fig. 20 - Effect of curing temperature on 18-h compressive strength of superplasticized concrete; from reference 32

Figure 20 shows the effect of superplasticizers on the strength of concrete at early ages (32).

DOSAGES OF SUPERPLASTICIZERS FOR BLAST FURNACE SLAG/PORTLAND CEMENT CONCRETES

Recent studies at CANMET have shown that blast furnace slag/portland cement concretes require about 10% less superplasticizer than reference portland cement concretes to attain the same workability (33,34). The reasons for the reduced requirement are not clear.

INCORPORATION OF SUPERPLASTICIZERS IN HIGH ALUMINA CEMENT CONCRETE

Limited investigations at CANMET have indicated that the addition of a low dosage of superplasticizer to high alumina cement concrete does not result in increased slump (34,35). However, at high dosage rates there is an increase in slump but the concrete does not flow; furthermore, the increase in slump disappears in less than 5 min. Additional research is indicated.

BIOLOGICAL SAFETY OF SUPERPLASTICIZERS

Limited or no data have been published on the biological safety of superplasticizers. Hattori has referred to some work being performed in Japan (12). No adverse effects from superplasticizers have been reported to date.

CONCLUSIONS

The North American concrete industry is generally conservative and is slow to accept new concepts and ideas, especially those developed elsewhere. The superplasticizers were developed in Japan and Germany, and have been only recently introduced into North America. They have already found acceptance in the precast concrete industry and research by various leading laboratories in North America may eventually result in their acceptance by the ready-mixed concrete industry.

Investigations performed in North America generally confirm the results published in Japan

and Germany. It is indicated that the slump loss problem must be overcome if the superplasticizers are to be accepted by the ready-mixed concrete industry because most North American specifications usually discourage the addition of admixtures to ready-mixed concrete at a job site.

The problem of freeze-thaw durability of concrete incorporating superplasticizers is not as serious as once thought, though research is needed to develop new limits for air void parameters. The problem of compatibility between different types of cements and superplasticizers also needs research.

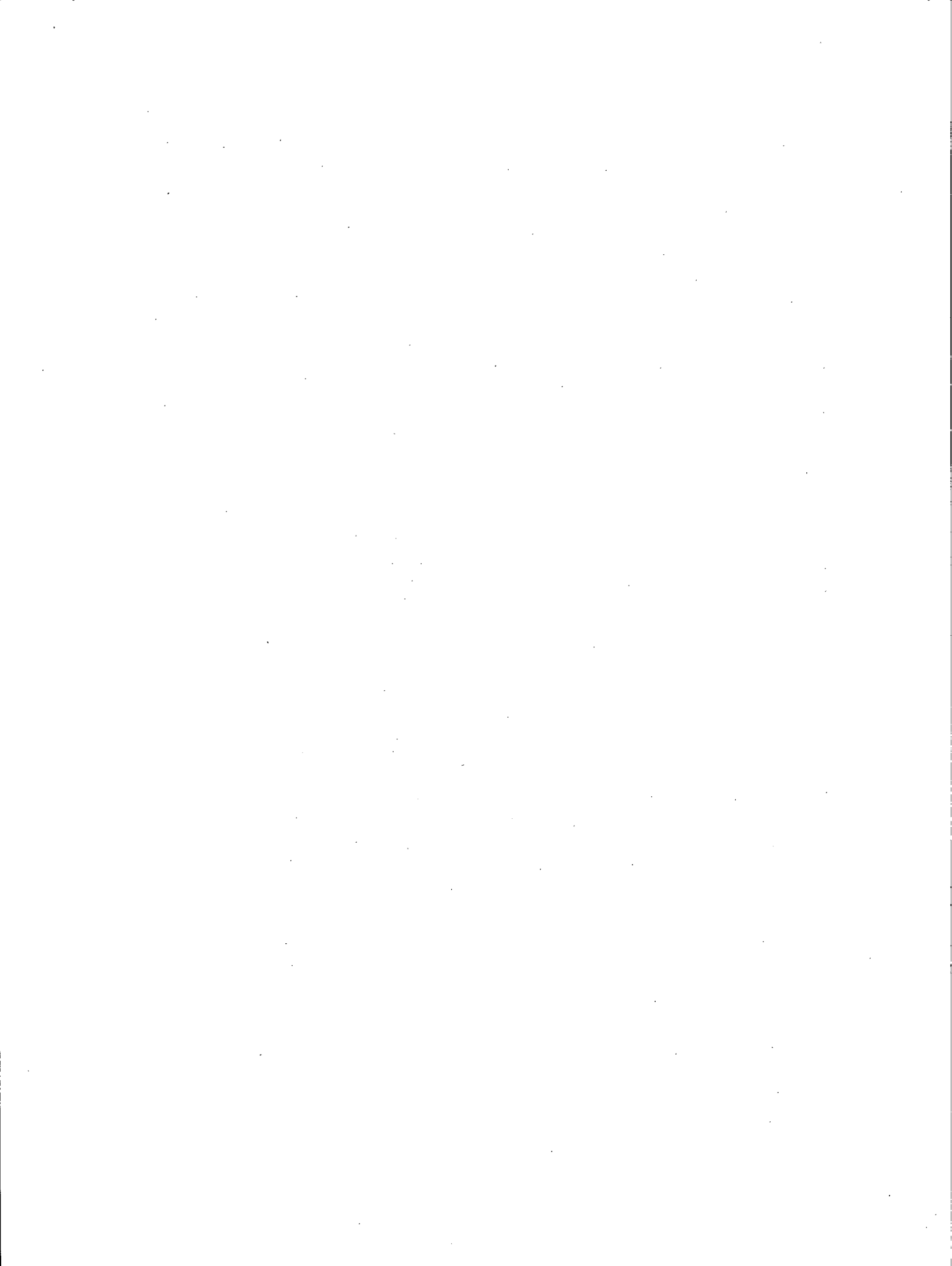
There have been very few major developments in concrete technology in recent years. The concept of air entrainment in the 1940's was one - it revolutionized concrete technology in North America. It is believed that development of superplasticizers is another major breakthrough which will have a very significant effect on the production and use of concrete in years to come.

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APPENDIX A

FLOWTABLE

APPARATUS

The flowtable comprises a board measuring 700 x 700 mm covered by a flat steel plate 2 mm thick (Fig. A-1). The centre of this plate, O, is marked by a cross parallel to the sides of the

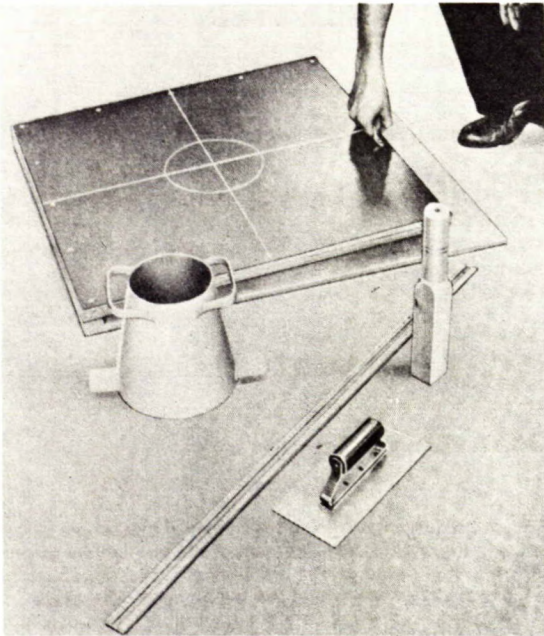


Fig. A-1 - Flow test apparatus; from reference 3

table and by a 20-cm diam circle, C. The hinged, H, top section, TS, weighs 16 kg, and vertical movement is limited to 40 mm by a stop, S. The mould for fresh concrete consists of a special slump cone 200 mm high, having internal diameters of 130 mm at the top and 200 mm at the bottom.

METHOD OF USE

The flowtable should be horizontal and steady. Before the test begins, the surface of the table and inside of the cone should be wetted.

The cone is placed at the centre of the table and concrete is added in two equal layers. Each layer is slightly tapped with 10 strokes of a wooden tamper with a base of 40 x 40 mm. Once the cone is full, the concrete is struck off with a steel straight-edge and the remaining concrete removed from the plate. Half a minute after striking off, the cone is removed by lifting slowly and vertically. After this, the table is lifted 15 times without striking it heavily and each time released and allowed to fall of its own accord. The entire operation takes about 15 s.

The two opposite diameters of the concrete are then measured. The spread is taken as the arithmetic mean of the two diameters, expressed to the nearest centimetre.

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