

TABLE 1

Mini Plant for manufacturing Type A or Type B
concrete superplasticisers

Capacity:	2 metric tonnes per month;
Time to commission plant from scratch:	10 weeks;
Skilled people needed (Supervisors):	2;
Unskilled people needed (Workers):	6;
Total area needed:	100 sq.m.;
Area for production plant:	50 sq.m.;
Investment in production plant and machinery:	Rs.50,000/- (US \$ 6,000/-);
Power requirement:	7 kW;
Power source:	Portable diesel generating set;
Water requirement:	5 cu.m. per month;
Pay-back period for plant:	12 months;

TABLE 2

Effect of superplasticiser on physical properties of cement

S.No.	Conc. % by wt. of cement	Normal consistency by %	Initial setting time, min.	Final setting time, min.	Soundness, mm.	Avg. comp. strength, kg/sq.cm.		
						3day	7day	28day
1	0.0	25.0	204	245	1.0	127	161	266
2	0.4	23.4	212	280	0.9	189	302	307
3	0.6	21.8	290	375	0.5	212	271	368
4	0.8	21.5	220	325	0.5	202	282	342
5	1.0	21.0	207	317	1.0	182	225	277

TABLE 3

Effect of Superplasticizer on Water Content and Compressive Strength

S.No.	W/C ratio	A/C ratio	Aggregate	Water content, lit/cu.m.	Conc. %	Slump mm.	Avg. comp. strength, kg/sq.cm.	
							7day	28day
1	0.55	5.5	33:27:40	195	0.0	25	107	179
2	0.47	5.5	33:27:40	171	0.4	25	156	242
3	0.46	5.5	33:27:40	165	0.6	25	151	242
4	0.44	5.5	33:27:40	161	0.8	25	148	250
5	0.42	5.5	33:27:40	155	1.0	25	183	264
6	0.40	5.5	33:27:40	149	1.2	25	181	271

TABLE 4

Effect of superplasticiser on cement content

S.No.	W/C ratio	A/C ratio	Aggregate	Cement content kg/cu.m.	Conc. %	Avg. comp. strength kg./sq. cm.	
						7day	28day
1	0.55	5.5	33:27:40	355	0.0	107	179
2	0.50	5.8	33:27:40	346	0.6	130	223
3	0.52	6.1	33:27:40	331	0.6	120	196
4	0.55	6.5	33:24:43	310	0.6	103	176
5	0.59	6.9	33:24:43	298	0.6	93	160
6	0.62	7.3	33:23:43	281	0.6	94	149
7	0.67	7.9	33:22:45	263	0.6	74	110

Note: Slump = 25 mm. in all mixes.

TABLE 5

Effect of superplasticiser on stiffening of fresh concrete

S.No.	W/C ratio	Conc. %	V.B. degree after			% Entrapped air content
			10min.	20min.	30min.	
1	0.55	0.0	11.5	17.5	21.5	1.5
2	0.47	0.4	10.0	18.0	22.0	1.1
3	0.45	0.6	10.5	18.0	21.0	1.2
4	0.44	0.8	31.0	33.0	38.0	1.2
5	0.42	1.0	40.0	42.0	48.0	1.5
6	0.40	1.2	19.5	22.0	25.0	1.2

Note: Aggregate (by weight): 33:27:40
 A/C ratio = 5.5
 Slump = 25 mm.

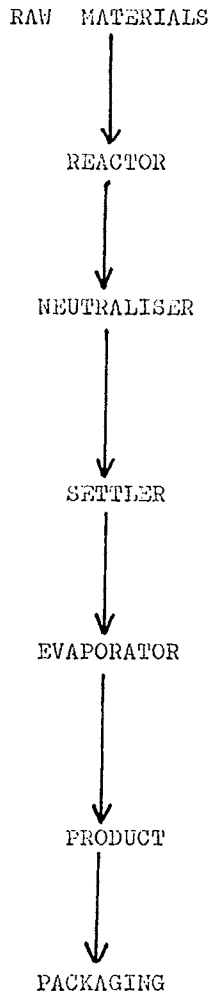


Fig.1: Outline of Manufacturing Process

Effect of Retarders/Water Reducers on Slump Loss in Superplasticized Concrete

By V. S. Ramachandran

Synopsis: Superplasticized concrete loses its workability within a few hours. The effect of different amounts of admixtures, such as Ca-lignosulfonate, sucrose, Na-gluconate, citric acid, salicylic acid, Na-heptonate and Na-boroheptonate, on the slump loss of concrete containing sulfonated melamine formaldehyde is reported. Of these admixtures, Na-gluconate proved to be the best retarder of slump loss; the influence on setting times and strength development in mortars was also examined.

Keywords: admixtures; calorimeters; compressive strength; concretes; hydration; mix proportioning; plasticizers; retarders; setting (hardening); slump tests; water-reducing agents; workability.

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The advent of superplasticizers has made it possible to produce concrete with high workability but no reduction in strength. Within a few minutes of the addition of a superplasticizer concrete begins to flow easily and becomes self-leveling, remains cohesive, and does not bleed or segregate. From an initial slump of about 50 mm, superplasticized concrete attains a slump (a measure of workability) in excess of about 200 mm. This increase is only transient, however, and is generally not maintained beyond a period of about 30 to 60 min. Consequently, there is a great reduction in the workability of concrete in the interval between mixing and placing. In ready-mix operations, therefore, it is suggested that the superplasticizer should be added at the point of discharge of concrete, although there are several practical problems associated with this procedure (1,2).

Factors that affect slump loss in concrete include initial slump value, type and amount of superplasticizer added, type and amount of cement, time of addition of superplasticizer, humidity, temperature, mixing criteria, and the presence of other admixtures in the mix (3-6). The rate of slump loss can be decreased by adding a higher than normal dosage of superplasticizer, by adding the superplasticizer at different times, or by including some type of retarder in the formulation. Inclusion of a retarder in small amounts seems to offer advantages such as economy and better control at the point of mixing.

This paper describes the effect of different amounts of retarders/water-reducing admixtures on the slump loss in concrete containing sulfonated melamine formaldehyde, and includes an assessment of the resulting rate of hydration, compressive strength, and setting properties of the mortars.

EXPERIMENTAL

Materials

Normal low-alkali portland cement with the following composition was used in the investigation:

<u>Chemical Analysis</u>	
<u>Constituent</u>	<u>% by weight</u>
SiO ₂	20.78
Al ₂ O ₃	6.20
Fe ₂ O ₃	2.22
CaO	64.83
MgO	1.84
SO ₃	3.17
Ignition Loss	0.50
Na ₂ O	0.05
K ₂ O	0.40
Insoluble	0.17
<u>Compound Composition</u>	
C ₃ S	51.4
C ₂ S	20.3
C ₃ A	12.7
C ₄ AF	6.7

It had a Blaine surface area of 299 m²/kg (2990 cm²/g) and showed 0.05% autoclave expansion.

The types of admixture that were used in two dosages in combination with 0.3% sulfonated melamine formaldehyde (SMF) are listed in Table 1.

Method

Compressive strengths of the mortar cubes (cement:sand ratio = 1:2.75 and w/c = 0.47), cured for 1, 3, 7, 28, and 90 days, were determined by ASTM 109-75 method.

Rate of heat development during hydration was determined by a conduction calorimeter, and rate of hydration was followed by a differential scanning calorimeter (DSC).

Time of initial setting of mortar mixes was determined according to ASTM test method C403-77, using the Proctor needle.

Slump values were determined using the ASTM C-143-78 method. Each 20.1 kg batch of concrete mix contained 3 kg cement, 6 kg sand, 9.6 kg graded coarse aggregate and 1.5 kg water.

The reference concrete was made by mixing it for 3 min, filling the cone, and determining the slump, 5 min in all. Determination of slump for concrete containing a retarder also took 5 min; the aqueous solution replaced the water used for making

the reference concrete. Concrete containing SMF was made as follows: initially, the concrete mix was made with water (mixed for 3 min) and let stand for 3 min more; SMF was added and the concrete mixed for 1 min and then poured into the cone. This operation took 9 min. Concrete containing SMF and retarder was made by initially adding the aqueous solution of the retarder and repeating the operation as described for that containing SMF.

RESULTS AND DISCUSSION

Sulfonated Melamine Formaldehyde (SMF)

The influence of SMF on the hydration of cement can be followed by a conduction calorimeter (Fig. 1). A broad hump with a maximum intensity at about 7 h for the reference cement paste (paste containing no SMF) represents hydration of the C_3S component of portland cement. After an induction or dormant period of about 2 h, hydration of C_3S is initiated and continues beyond 20 h, as is evident from the non-return of the curve to the base line; addition of 0.3 to 2% SMF does not seem to influence this dormant period. The rate of hydration of C_3S , however, is affected, as can be seen from the lower rates of heat development (Fig. 1). At 2% addition the rate of heat development at 7 h is decreased by about 50%. Adsorption-desorption isotherms of SMF on hydrating C_3S and portland cement show that SMF is irreversibly adsorbed to different extents (7). An adsorption complex (formed by the reaction of C_3S or cement with SMF and H_2O) formed on the unhydrated surfaces is capable of retarding hydration.

The retarding action of SMF is reflected in slightly delayed setting times of mortars. The reference mortar exhibits an initial setting time of $4\frac{1}{2}$ h, which is extended to $4\frac{3}{4}$ and $6\frac{1}{2}$ h in mortars containing 0.3 and 0.6% SMF, respectively (Fig. 2). DSC curves of initially set mortars show small endothermic peaks at about $100^\circ C$, representing C-S-H and/or ettringite phases, followed by another endothermic effect of larger intensity with a peak between 450 and $500^\circ C$ (Fig. 3). This peak is due to decomposition of $Ca(OH)_2$ formed by the hydration of C_3S . Both the reference mortar and that containing SMF show endothermic effects in this region, indicating some hydration of C_3S at the time of setting of the cement paste. Setting represents a matrix having a particular value of resistance (arbitrary) to needle penetration. The endothermic effect of the sample containing 0.3% SMF is less intense than that of the reference mortar (Fig. 3). In other words, a smaller amount of hydrated product formed in the presence of SMF offers the same resistance to the Proctor needle as does the reference mortar containing about twice the amount of hydrated C_3S . This indicates that mortar containing SMF is better dispersed and forms a more compact network.

Addition of 0.3% SMF to cement increases mortar strength substantially at all periods up to 90 days (Table 2). For example, at 28 and 90 days the percentage increase in strength of mortars containing SMF over that of the reference mortar is about 26 and 22%, respectively. These higher strengths are attributed to good mixing and better compaction.

The slump values of the reference concrete and that containing 0.3% SMF are shown in Fig. 4. The reference concrete has an initial value of about 57 mm, gradually decreasing to about 13 mm in 2 h. The slump of concrete to which SMF has been added with the mixing water is increased to about 89 mm, but it is reduced to about 32 mm in 2 h. The slump value can be as high as 178 mm for concrete to which SMF has been added a few minutes after mixing with water, but this high value decreases rapidly to about 51 mm in 120 min. Increase in slump value can be explained by adsorption of SMF by hydrating cement particles, followed by mutual repulsion similar to the phenomenon occurring in cements containing anionic admixtures. Addition of SMF with the mixing water does not promote high workability because most of the superplasticizer reacts chemically with hydrating aluminate components of cement (7). Thus, only a negligible amount of SMF is left in the aqueous phase to disperse the silicate phase in cement. The addition of SMF a few minutes after mixing with water allows rapid hydration of the aluminate phases of cement and hydrated products. Adsorbing less SMF than the unhydrated phase, the hydrated products leave a substantial amount of free SMF in the aqueous phase to disperse the calcium silicate phase. Similar observations have been made in cements containing lignosulfonates (8-10).

Retarders/Water-Reducing Admixtures

Representative data are given regarding the influence of retarders and water-reducing admixtures on slump loss, setting, hydration and strength development in concrete and mortar. Figures 5 to 8 show the influence of sucrose, citric acid, Na-heptonate, Na-boroheptonate, Ca-lignosulfonate and Na-gluconate on the slump values of concrete. Addition of sucrose or citric acid does not result in an increase in the initial slump of concrete (Figs. 4 and 5). Incorporation of Na-heptonate or Na-boroheptonate is effective in increasing the initial slump value from 57 mm (Fig. 4) to about 89 to 95 mm (Fig. 6). The addition of Ca-lignosulfonate and Na-gluconate gives substantially higher values of slump, 127 mm (Fig. 7) and 140 mm (Fig. 8), respectively. Although these values are more than double that of the reference concrete, they are less than those obtained with SMF.

Irrespective of the type of retarding admixture, all slump values decrease to about 38 to 51 mm in about 2 h. Although it is recognized that complicated factors are involved in the slump loss phenomena, accelerated formation of ettringite by the reaction of C_3A with gypsum is an important cause (11).

Initial setting times of mortars containing various admixtures are shown in Fig. 2. All increase the setting times, the more effective admixtures being Ca-lignosulfonate (0.4%), sucrose (0.05-0.1%), Na-gluconate (0.1-0.2%) and citric acid (0.1%). The most efficient, however, is Na-gluconate (0.2%), which increases setting time from 4½ to 15¼ h. This is also reflected in the effect of Na-gluconate on the hydration of cement (Fig. 9). Addition of Na-gluconate not only increases the induction period for the hydration of C₃S but also shifts the maximum rate of heat development for C₃S from 7 to 12 h. The DSC curves of the mortars containing various admixtures show that all cements have hydrated to some extent at the time of initial set (Fig. 3). Just as with SMF, the peak intensity for Ca(OH)₂ is reduced in the presence of retarding admixtures. Such reduction is particularly significant in cements containing 0.2% Na-gluconate.

Development of compressive strength in mortars containing various admixtures is indicated in Table 2. The one-day strength of mortars containing the retarding admixtures is generally lower than that of the reference specimen, the least strength developed by the mortar containing 0.2% Na-gluconate. This is to be expected because it is the best retarder of rate of hydration. As hydration progresses, strength of mortars containing these admixtures generally exceeds that of the reference mortar, but at 90 days those containing Ca-lignosulfonate and citric acid exhibit much lower strengths. Lower strengths in the presence of lignosulfonate may be due to entrainment of air, which causes higher porosity. The presence of Na-gluconate (0.1 to 0.2%), however, results in about 15% higher strength than that of the reference specimen. Generally, retarders are known to impart low early strength and higher strength at 28 days; at longer periods of hydration there is a possibility that hydration products are formed by slower rates of diffusion and precipitation and that this results in their relatively more uniform distribution in the interstitial spaces among the cement grains. An increase in the total interparticle bond area may thus be responsible for better strengths.

Sulfonated Melamine Formaldehyde + Retarders/Water-Reducing Admixtures

Figures 5 to 8 show the slump values of concrete containing SMF admixed with sucrose, citric acid, Na-heptonate, Na-boroheptonate, calcium lignosulfonate and Na-gluconate. Concrete with a combination of sucrose + SMF has the same initial slump as that containing SMF alone, but it seems to yield lower slump values at 2 h (Fig. 5). Concrete with citric acid + SMF not only has a higher initial slump value than the sucrose + SMF mixture but also retains it at 2 h (Fig. 5). Both Na-heptonate and Na-boroheptonate in combination with SMF increase initial slump and exhibit slightly higher slump at 2 h than concrete with SMF alone. The calcium lignosulfonate + SMF mixture shows similar behaviour (Fig. 7); but the Na-gluconate + SMF mixture is different from the others in that