MATERIALS TECHNOLOGY LABORATORY

REPORT MTL 2003-44(CF)

EVALUATION OF THE POTENTIAL USE OF THE SUPERPLASTICIZER ALPHALITH FT30LV WITH CANADIAN CEMENTS

by

P.C. Nkinambanzi and B. Fournier

SUMMARY

ECTechnology, Vancouver, via ECOSMART, provided CANMET-MTL's International Centre for Sustainable Development of Cement and Concrete (ICON) with a new superplasticizer (SP) produced by Rhein Chemotechnik GmbH, Breitscheid Germany, the Alphalith FT30LV. This superplasticizer meets the European standards and certifications for analyzing and testing of its suitability for the Canadian market, and it belongs to the family of naphthalene formaldehyde condensates-based superplasticizers. A commercially available and commonly used Canadian superplasticizer from this family that efficiently improves the flow of cement pastes was used in this study for comparison.

Tests were conducted on grout to determine the efficiency of both superplasticizers as dispersing admixtures of cement pastes. Tests on concrete were performed to ensure that this admixture does not induce bad side effects such as segregation, increasing air content of the concrete, setting retardation, etc.

The FT30LV is compatible with the Canadian cement used, but the dosage required to obtain a good fluidity of the cement pastes is higher than that of the Canadian superplasticizer. No retardation of setting was observed even when the superplasticizer content was fairly high. The one-day compressive strength of the concrete containing the FT30LV superplasticizer was similar to that obtained with the reference superplasticizer, indicating that this admixture does not negatively effect the hardening of the concrete.

LABORATOIRE DE LA TECHNOLOGIE DES MATÉRIAUX

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ÉVALUATION DE L'UTILISATION DU SUPERPLASTIFIANT ALPHALITH FT30LV AVEC LES CIMENTS CANADIENS

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RÉSUMÉ

La compagnie ECTechnology, Vancouver, via ECOSMART, a fourni à LTM-CANMET Centre international pour le développement durable du ciment et du béton (ICON) un nouveau superplastifiant pour béton, l'Alphalith FT30LV, produit par la compagnie allemande Rhein Chemotechnik GmbH, Breitscheid, pour analyse et essais de sa compatibilité avec le marché canadien. Le nouveau SP respecte les normes et certifications européennes. Ce produit est de la famille des superplastifiants à base de condensats de formaldéhyde et d'acide naphtalène sulfonique. Un superplastifiant canadien de cette même famille qui est couramment utilisé et connu pour être très efficace pour améliorer la fluidité des pâtes de ciment a été utilisé pour des besoins de comparaison.

Des essais ont été menés sur coulis pour déterminer l'efficacité des deux superplastifiants comme adjuvants dispersants des pâtes de ciments. Des essais sur béton ont été menés pour s'assurer que le nouvel adjuvant n'occasionne pas d'effets secondaires néfastes tels que la ségrégation, l'augmentation de la teneur en air du béton, le retard de la prise, etc.

Le FT30LV est compatible avec le ciment canadien utilisé mais son dosage pour obtenir une bonne fluidité des pâtes de ciment est plus élevé que celui du superplastifiant canadien. Aucun retard de la prise du béton n'est observé même si le dosage en superplastifiant est très élevé. La résistance à la compression à un jour du béton fait avec le FT30LV est similaire à celle obtenue avec le superplastifiant de référence, ce qui est une bonne indication du fait que ce superplastifiant n'a pas nui au durcissement du béton.

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OBJECTIVES

The objective of this study was to analyze and test the Alphalith FT30LV superplasticizer provided by ECTechnology with Canadian cement to determine its suitability for the Canadian market.

SCOPE OF THE STUDY

Laboratory tests on grouts and concrete were performed to identify the possible side effects of admixtures on the properties of fresh concrete, such as increasing setting time, modification of air content, hardening retardation, etc.

The efficiency of the superplasticizer was first determined on grouts by using two simple test methods: the Marsh cone and the mini-slump test (see Appendices 1 and 2 for sketches of the apparatus). As screening test procedures, these two tests are quick and require minimum amounts of materials compared to tests performed on concrete (1).

Grouts containing different amounts of superplasticizers were prepared, and their fluidity was monitored at regular intervals over a 90-min period using the mini-slump test. This test is, as its name implies, a small-scale version of the slump test commonly performed on concrete. For the Marsh cone test, 1.2 L of grout having a fixed W/C of 0.35 was allowed to flow through a cone with a funnel diameter of 5 mm. The flow time was measured 5 and 60 min after mixing the cement and water. Five to six dosages of superplasticizer are sufficient to obtain the so-called *saturation point* of the superplasticizer, i.e. the optimum amount beyond which no more gain in fluidity of the grout is obtained. The information obtained from these two screening tests minimizes the number of concrete mixtures needed to determine the optimum dosage of a superplasticizer which, actually, changes with the cement used.

Concrete mixtures have been made to validate the results obtained on grouts. For comparison, control concrete mixtures used a commercially available superplasticizer of the same family.

MATERIALS USED

CEMENT

The cement used is a CSA Type 10 cement from Lafarge Canada, Montréal. This cement is commonly used at CANMET-MTL as the base cement to evaluate the performance of high-range water-reducing admixtures. The chemical composition, the Bogue composition and some physical properties of the cement are given in Table 1.

Table 1 – Chemical composition and physical properties of the cement.

CSA type-10 (ASTM type I)			
Chemical composition (%)		Bogue composition (%)	
SiO_2	20.33		
Al_2O_3	4.36	C_3S	59.9
Fe_2O_3	2.99		
CaO	62.92	C_2S	13.9
MgO	2.72		
Na_2O	0.27	C_3A	6.5
K_2O	0.83		
TiO_2	0.19	C_4AF	9.1
MnO	0.04		
SO_3	3.23		
LOI	2.32		
SiO_2	20.33		
Physical properties			
Passing 45 µm, %			93.2
Blaine, cm^2/g			402
Specific gravity			3.15
Compressive strength on cubes, MPa			
3 days			28.2
7 days			33.0
28 days			40.8

AGGREGATES

The coarse aggregate used in the concrete mixtures was a crushed limestone from the Ottawa area. The fine aggregate was a local natural sand derived from granite. The grading and physical properties of the coarse and fine aggregates used in the concrete mixtures are given in Tables 2 and 3, respectively.

SUPERPLASTICIZERS

Two superplasticizers were used in this study, i.e. the Alphalith FT30LV from Germany and a control superplasticizer, Disal (from Handy Chemical Inc.), from Canada. Both superplasticizers are solutions of sodium salts of naphthalene formaldehyde condensates having solids content of around 41%. The physico-chemical properties of the superplasticizers are given in Table 4.

Table 2 – Grading of aggregates.

Coarse aggregate			Fine aggregate		
Sieve size		Cumulative percentage retained	Sieve size		Cumulative percentage retained
mm	(in.)	%	mm		%
19.0	(3/4)	0	4.75	(No. 4)	0
12.7	(1/2)	30	2.36	(No. 8)	10.0
9.5	(3/8)	70	1.18	(No. 16)	32.5
4.75	(No. 4)	100	0.60	(No. 30)	57.5
			0.30	(No. 50)	80.0
			0.15	(No. 100)	94.0
			Pan		100.0

Table 3 – Physical properties of aggregates.

	Coarse aggregate	Fine aggregate
Specific gravity	2.72	2.70
Absorption, %	0.50	0.80

The solid content of both superplasticizers was determined by weighing a sample of 50 g of the chemical admixture before and after drying for 24 h in an oven at 110°C. The solid content is the difference between the 2 weights. The specific gravity was obtained by weighing 20 mL of the admixture in a gauged flask.

Table 4 – Some properties of the superplasticizers.

Superplasticizers	Na-PNS* (control)	Alphalith FT30LV
Chemical family	Na-PNS*	Na-PNS*
Solids content	41.2	41.1
pH	7.8	8 - 10
Specific gravity	1.21	1.17
% Sulphates	0.92	-

*Na-PNS = Naphthalene formaldehyde condensate sodium salt

MANUFACTURING OF THE GROUTS AND THE CONCRETE MIXTURES

GROUTS

Mini-Slump Test

The grouts were prepared using 300 g of cement and sufficient water to achieve the target W/C of 0.35 after adjusting for the water contributed by the superplasticizer solution. Dosages of superplasticizers ranging from 0.4% to 2.0%, expressed as percent of dry solids by the mass of the cement in the mixture, were used to prepare the grouts. The cement and the superplasticizer solution were first mixed by hand for 1 min in a 250 mL beaker using a stainless-steel spatula; this was followed by 2 min of mechanical mixing using an electric hand-blender (in this case a MR 400 Braun Multiquick, 200 W). The water used was cold enough so that the temperature of the grout at the end of the mixing was not higher than 23°C (1).

March Cone Test

The amounts of the ingredients required (water, cement and superplasticizer) were calculated using software developed for this purpose. Usually, the test is performed at a W/C ratio of 0.35 in order to test the cement and the superplasticizer under conditions similar to those of the paste of a high-performance concrete (1). The volume of the grout needed was 1.2 L, which is 6 times the material needed for the mini-slump test (around 1.8 kg of cement for the Marsh cone compared to 300 g for the mini-slump test). The grout was mixed in a kitchen blender with a 2-L container. The mixing water was weighed in the blender container, and the superplasticizer was then added to it. The mixing process was started, and the cement was introduced progressively in the blender over a period of 1 min 30 s. The mixing was stopped for 15 s in order to scrape the cement that had adhered to the sides of the container with a plastic spatula. The mixing continued for 60 s. The temperature of the grout was measured before measuring the flow-time of the grout.

CONCRETE MIXTURES

Non-air-entrained high-performance concrete mixtures having a W/C ratio of 0.30 were made using both superplasticizers at their saturation dosages. Conventional concrete mixtures having a W/C ratio of 0.42 were also made using lower superplasticizer dosages. The lower W/C of high-performance concrete is well suited to study the effectiveness of the dispersing action of the cement grains by superplasticizers. The proportions of the concrete mixture are given in Table 5. The relatively high cement content of the high-performance concrete was deliberately selected to enhance the interactions between the cement and the superplasticizer, allowing to easy detector of compatibility problems between cement/SP combinations (2).

The concrete mixtures were made in a counter-current-pan mixer. The materials preparation and the mixing sequence used were as follows:

- Coarse and fine aggregates were weighed in a dry state 24 h prior to mixing;
- Water was measured and added to the coarse and fine aggregates then allowed to stand for 24 h;
- Just prior to mixing, the water was drained from the coarse aggregate's container, and the coarse and fine aggregates in a saturated surface wet-state were weighed, the amount of mixing water required to achieve the desired W/C was then calculated;
- Coarse aggregate was loaded into the mixer and mixed for 5 s;
- Fine aggregate was then introduced into the mixer;
- Cement and half the amount of the mixing water were gradually added (simultaneously) to the mixture while the mixer is in motion (30 s);
- Remaining mixing water incorporating the superplasticizer was then added to the mixture was the mixing continued for an additional 2.5 min (principal mixing period). The mixture was then allowed to stand for 3 min in the mixer, covered with wet burlap. The concrete was then mixed for 2 min during which time the fluidity of the concrete was adjusted, as desired, by adding a small amount of superplasticizer.

Table 5 - Concrete mixture proportioning.

W/C		0.30	0.42
Water	kg/m^3	145	143
Cement		480	340
Sand		790	780
Coarse aggregate		1020	1180
Superplasticizer (L/m^3)	FT30LV	14.5	4.7
	Control	10.8	2.5

TESTING PROGRAM

TEST ON GROUTS

The two tests described below are commonly used to identify which combinations of cement and superplasticizers are compatible as well as the saturation point of superplasticizers.

Mini-Slump Test

Readings of mini-slump are taken at 10, 30, 45, 60 and 90 min after the contact between the cement and the mixing water. The mini-slump value of the grout was calculated using the spread diameter of the grout measured using the following procedures (1,3).

A precisely level Plexiglas plate was placed on a table in the laboratory. The mini-cone was then placed at the centre of the plate, and, after 15 s of hand mixing with a stainless steel spatula to homogenize the grout, the mini-cone was filled with the paste. Ten strokes were given on the top of the mini-cone before it was raised rapidly enabling the paste to spread on the Plexiglas plate. The diameter of the grout was then measured along two perpendicular directions and these two values were averaged. The paste was returned to the beaker, hand-mixed for 5 s, and the beaker was covered to avoid any desiccation. The Plexiglas plate and the cone were cleaned and dried for the next reading. The fluidity loss was thus monitored for increasing dosages of superplasticizer, and the saturation dosage was obtained by identifying the lowest dosage at which fluidity-loss of the grout was stabilized as a function of time.

Marsh Cone Test

The Marsh cone test is a dynamic measurement compared to the mini-slump test. In the Marsh cone test, 1.2 L of grout is allowed to freely flow through a cone with an aperture of 0.5 cm (see Appendix 2). The time required to fill a 1-L graduated cylinder is measured for each dosage of superplasticizer selected. Flow-time readings are taken at 5 and 60 min after the contact between the cement and the mixing water. The time elapsed after every 100 mL of flow of the grout is recorded until 1 L of grout has passed through the cone. Between these 5 and 60-min reading times, the grout in a 2-L plastic bottle is maintained mechanically in rotational motion to prevent coagulation of cement particles and segregation and also to simulate the concrete transportation (1). If less than 1L of grout can pass through the cone, the grout fails the test. The flow time for 700 mL of the grout passing through the Marsh cone is used to determine the saturation point of the superplasticizer under study by drawing a graph of the flow time of 700 mL of each grout as a function of the superplasticizer dosage[4]. The reason for using the volume of 700 mL of grout is that the flow-regime of the grout changes after the flow of 700 mL due to the decrease in the mass charge in the cone. A curve of the flow time as a function of the volume passing through the cone shows a deflection from the linearity at 700 mL of the volume passing through the cone. The dosage of the superplasticiser at the saturation point gives an approximation of the dosage needed to achieve the best rheological conditions for fresh concrete and mortar.

TESTS ON CONCRETE

Following the mixing of the concrete as described before, the slump of the fresh concrete was monitored at regular intervals over a 90-min period following the contact between the cement and the mixing water according to the ASTM C39 standard. Between each measurement, the mixer was covered with a plastic sheet to reduce the risk of water-loss through evaporation.

The properties of the freshly mixed concrete were determined according to ASTM standards [i.e. slump and slump loss (ASTM C 143), unit weight (ASTM C138), air-content (ASTM C 231) at 10 min, and setting time (ASTM C 403)]. Concrete cylinders, 100 by 200 mm in size, were cast for compressive-strength determination at 1, 7 and 28 days in accordance with the ASTM C39 standard.

RESULTS AND DISCUSSIONS

TESTS ON GROUTS

Mini-Slump Test

The fluidity of the cement pastes expressed in terms of spread diameter as a function of time is given in Fig. 1 for the control and FT30LV superplasticizers.

It can be seen that, for the control admixture, the spread diameter is higher than that given by the FT30LV admixture for all dosages tested in this study (Fig. 1). The initial mini-slump varies from 65 to 135 mm at the dosages of 0.4% and 1.2%, respectively, for the control superplasticizer, while it varies from 55 to 110 mm at the same dosages for the FT30LV superplasticizer. The saturation dosage determined by this test seems to be 1.0% for the control SP and more than 1.2% for the FT30LV SP. The trend of the fluidity loss of the control SP at a dosage of 0.8% is similar to that of the FT30LV SP at the dosage of 1.0%, and at the dosage of 1.2% the latter still loses its fluidity.

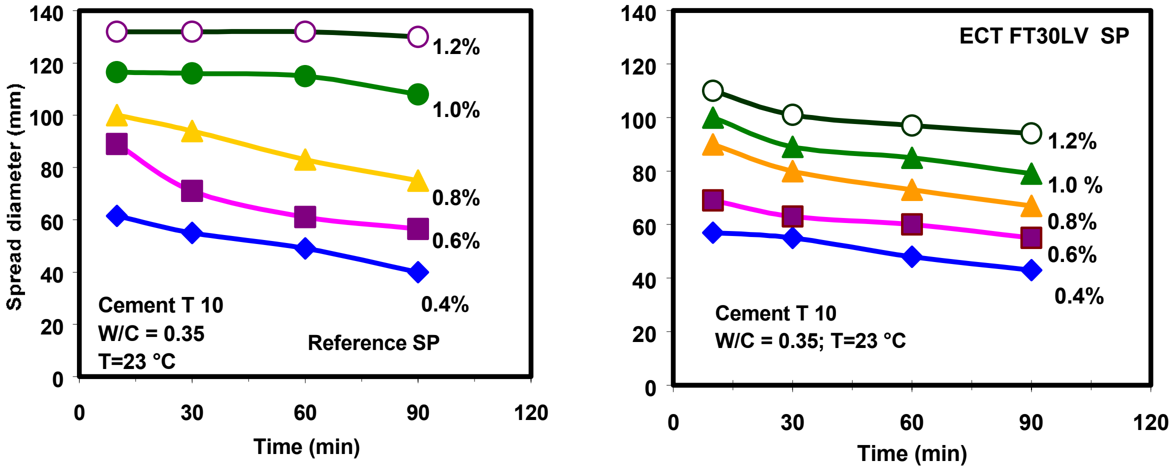

Fig. 1 - Results of the Mini-slump test expressed as the spread diameter of the grout as a function of time for the cement pastes made with the control and the FT30LV superplasticizers. The dosage of superplasticizer is expressed as percent of dry solids by the mass of the cement in mixture.

Marsh Cone Test

Figure 2 shows plots of the flow time at 5 and 60 min, for superplasticizer dosages ranging from 0.4% to 2.0% of the mass of the cement. For dosages up to 0.6%, there is no flow for the FT30LV superplasticizer (Fig. 2). For the dosages between 0.8 and 1.2%, the flow is good at

5 min, but the grout loses its fluidity between 5 and 60 min so that the flow time at 60 min is more than twice that at 5 min for the dosage of 1.2%. There is no flow at 60 min for dosages of 0.8 and 1.0% of FT30LV superplasticizer. The saturation dosage determined using the Marsh cone test is around 2.0% for this superplasticizer. For the control superplasticizer (Disal), the flow time is good at 5 min for dosages more than 0.6%. After 60 min, the dosage must be 1.0% to obtain a suitable flow time (less than 80 s). The saturation dosage is 1.0% for the control admixture which is half that needed for the FT30LV.

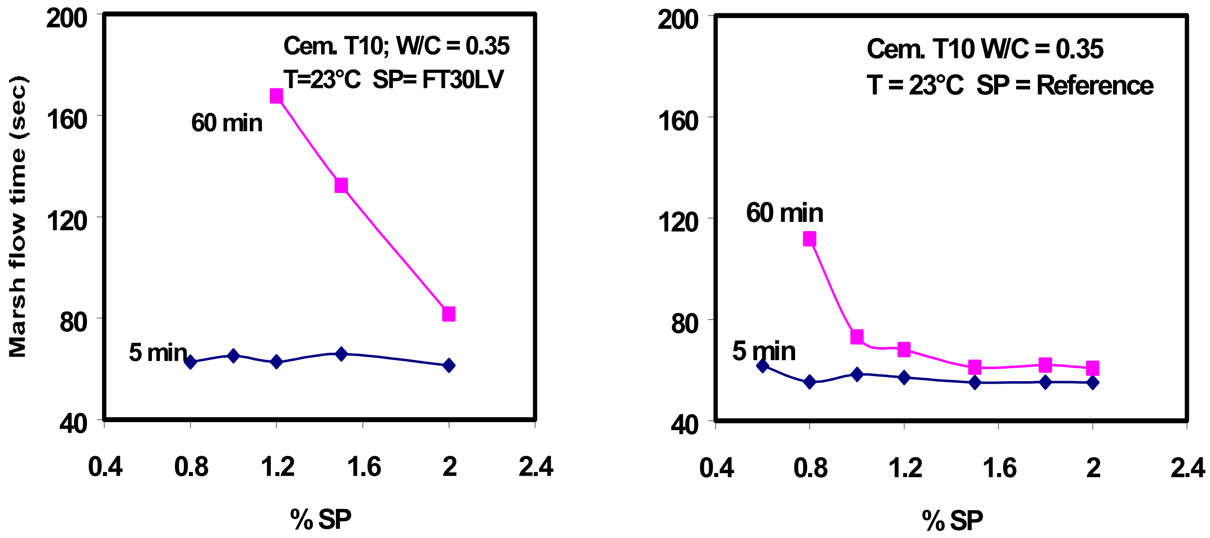

Fig. 2 - Saturation dosage of the FT30LV and of the control SPs with type-10 cement, as obtained by the Marsh cone test.

CONCLUSIONS FROM THE TESTS PERFORMED ON GROUTS

The mini-slump and the Marsh cone tests show that the FT30LV superplasticizer disperses the cement paste less effectively than the control superplasticizer. Molecular properties such as the degree of sulfonation, the degree of polymerization, the position where sulfonates group onto the aromatic rings of the molecule, the molecular weight distribution of the polymer, etc. could explain these differences (5). These results have to be confirmed by tests on concrete for more accurate conclusions.

TESTS ON CONCRETE

Properties of Fresh Concrete

Air Content and Slump

The slump and air content of the fresh concrete are presented in Table 6. The higher dosages of both superplasticizers used in the concrete mixtures with low water-to-cement ratio (i.e. W/C = 0.30) resulted in increased air contents in those concretes compared to the concretes with a W/C of 0.42. The air content was measured 10 min after the contact between the cement and the mixing water.

The slump of the concrete was determined at 10 and 60 min after the contact between the water and the cement. The value of the slump at 10 min (initial slump) gives the relative efficiency of the admixtures to disperse the cement grains and to enhance the workability of the concrete. The value of the slump at 60 min gives the slump retention of the superplasticized concrete or the ability of the superplasticizer to maintain a good workability concrete with low W/C (1,6). According to the data shown in Table 6, the initial slump was similar for both superplasticizers at the same dosage when the W/C is high (0.42). For low W/C concretes, the control superplasticizer gave a slightly higher initial slump and a slightly better slump retention, even when the dosage of the control admixture was significantly lower than that of the FT30LV admixture. This is in agreement with what has been seen with grouts and mortars. The FT30LV superplasticizer could be used with CSA Type-10 cement, but a higher dosage is required to obtain the same performance in low W/C concretes than the control superplasticizer.

Table 6. Properties of fresh and hardened concrete.

SP	Dosage L/m^3	W/C	Air %	Slump (mm)		Setting time (h)		Compressive strength (MPa)		
				10 min	60 min	Initial	Final	1 d	7 d	28 d
Control	2.5	0.42	1.7	140	-	4.20	5.60	30.8	39.8	48.9
	10.8	0.30	3.0	180	100	5.50	6.70	43.8	54.9	62.8
FT30LV	4.7	0.42	2.0	160	-	4.75	5.90	32.4	41.8	49.5
	14.5	0.30	3.6	150	80	5.85	7.25	43.7	56.7	63.6

Setting Time

For the concrete mixtures with a W/C of 0.42, the initial and final setting times were 4.75 and 5.90 h, respectively, for the concrete made with the FT30LV, and 4.20 and 5.60 h, respectively, for the “control” concrete (Table 6). The difference of 30 min between the performances of the two admixtures is not considered significant.

For the concrete mixtures with a W/C of 0.30, the difference between the initial final setting times for the two superplasticizers remained low; i.e. 5.50 and 6.70 h for the control admixture and 5.85 and 7.25 for the FT30LV admixture, respectively. The final setting time for the FT30LV was slightly higher (1 h) because the dosage was higher for this admixture. But this difference is not dramatic.

Mechanical Behaviour of Hardened Concrete

The compressive strengths of the two brands of concrete mixtures (i.e. W/C of 0.42 and 0.30) made with the two superplasticizers were similar at both early and later stages (Table 6). No side effect on the strength development was observed for the FT30LV superplasticizer. These results suggest that it could be used successfully with Canadian CSA Type-10 and similar cements, but trial mixtures on grouts would be good practice to avoid unexpected behaviors.

CONCLUDING REMARKS

The FT30LV superplasticizer could be used with the Canadian cement tested in this study, [a general-use portland cement (T10)]. The saturation dosage of this superplasticizer is somewhat higher than the one used with the control SP, and that is broadly used in North America and is from the same naphthalene SP family. Although a higher dosage of the FT30LV SP is necessary to achieve good fluidity in concrete, this did not result in excessive set retardation or excessive air content in the concrete. The compressive strengths of concretes made with this SP are similar to that made with the control SP.

More tests should be performed including testing of air-entrained concrete to evaluate the compatibility of the FT30LV superplasticizer with air-entraining admixtures and the effect it has on the durability of concrete. Tests with concrete incorporating fly ash and other cementitious materials should be added to the program.

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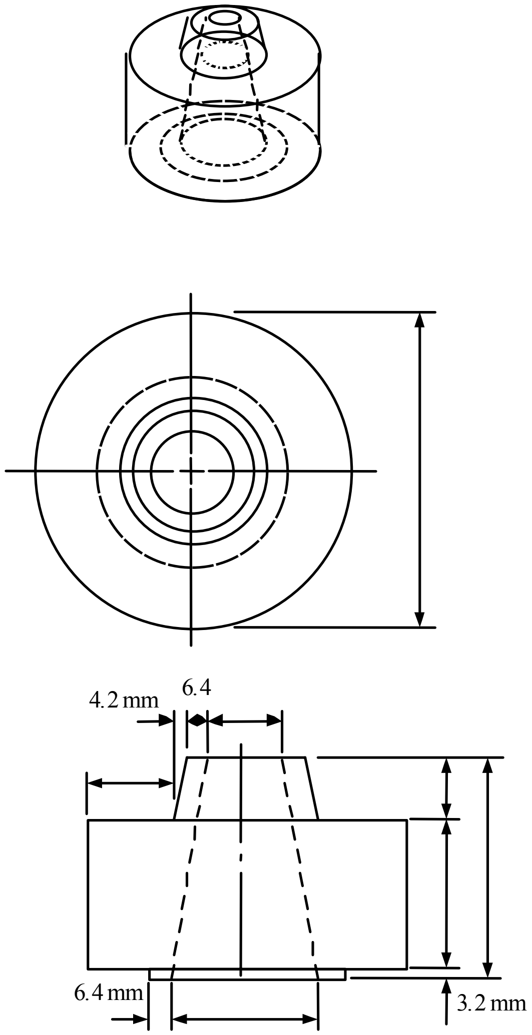
ANNEXE 1A



Schematic representation of a mini-cone*.

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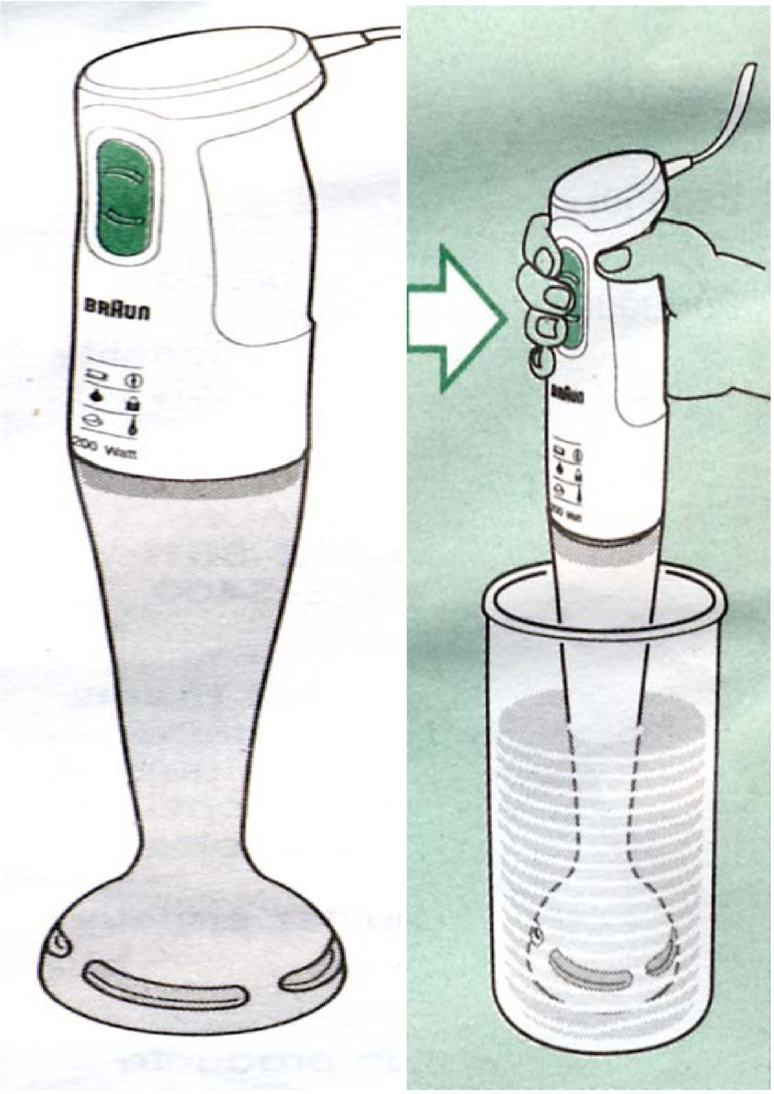
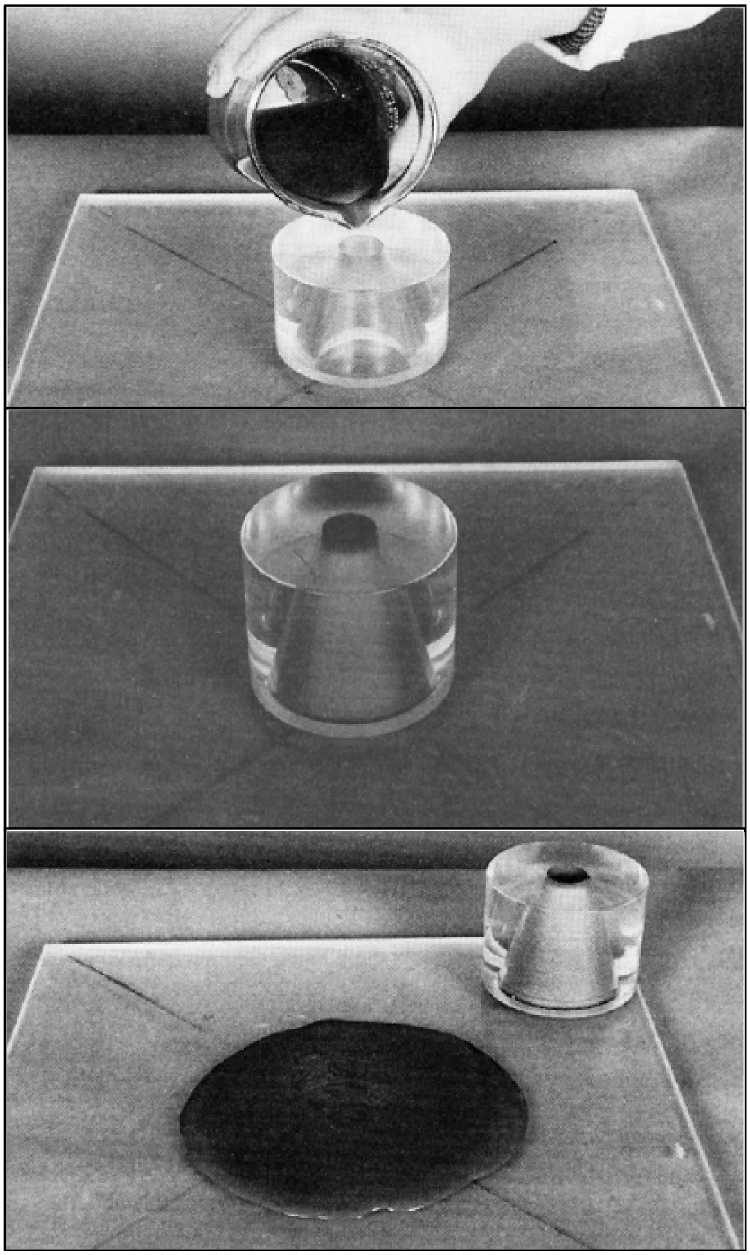
*(D.L. Kantro, cem.concr.agrgr., CCAGDP, Vol. 2, No. 2, Winter 1980, pp. 95-102).

ANNEXE 1B



Handblender used for preparation of grout mixtures — Braun MR 400 Multiquick, 200 W.

ANNEXE 1C

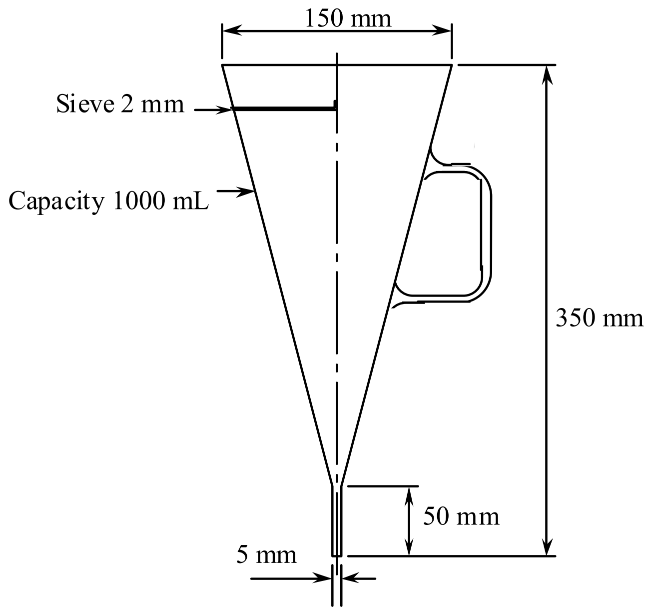


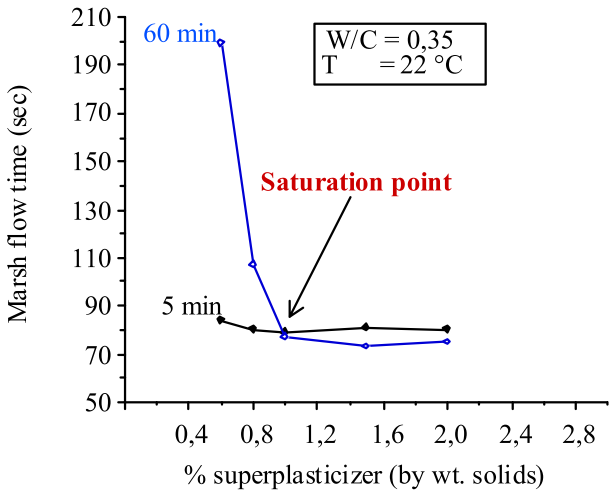
Mini slump test of cement grout (from Ref. 1).

ANNEXE 2A

SUPERPLASTICIZER SATURATION POINT

Marsh cone





ANNEXE 2B

Mixing device and measurement of the flow-time (Marsh Cone) (from Ref. 1).