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RHEOLOGICAL AND MECHANICAL BEHAVIOR OF HIGH VOLUME FLY ASH CEMENT GROUTS

by

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RESEARCH IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE
DEGREE OF MASTER OF ENGINEERING

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ABSTRACT

The society is becoming increasingly more energy and environmentally conscientious, which has forced development of construction materials, which are both inexpensive and require very little energy to produce. One way of producing such materials is to utilize by-products of industrial processes. High-volume fly ash cement based grouts are tributary of such developments.

This thesis outlines the results of a detailed study of the grain size distribution, three rheological properties and five mechanical properties of high-volume fly ash grouts (60 % cement replacement for fly ash by weight) with or without the use of superplasticizers and/or anti-washout agents. The rheological properties are reported for eight water/cement + fly ash (water/solids) ratios. The effect of superplasticizers and anti-washout agents on the flow time of low water/solids ratio grouts and the stability of high water/solids ratio grouts are investigated.

The study revealed that:

- The addition of fly ash reduces significantly the flow time of low water/solids ratio grouts (0.4 to 0.65).
- The stability of cement grout is improved by the addition of fly ash for water/solids ratios greater than or equal to 0.65.
- The use of fly ash reduces the early compressive and bond strength, when compared to the reference grout (pure cement). However, as they mature their strengths are closer to those of the pure cement grouts.
- Fly ash reduces the amount of drying shrinkage compared to pure cement grout with an equivalent water/solids ratio.

RÉSUMÉ

La conscientisation de la société face à l'environnement et aux ressources énergétiques a fait naître de nouveaux matériaux de construction à la fois économiques et faibles consommateurs d'énergie lors de leur production. Les coulis à base de ciment à haute teneur en cendres volantes sont tributaires de ces développements.

Les cendres volantes sont un sous-produit de la génération thermique de l'électricité. Quand elles sont combinées avec le ciment, elles contribuent aux propriétés mécaniques et rhéologiques des coulis.

Ce mémoire souligne les résultats d'une étude détaillée sur trois propriétés rhéologiques et cinq propriétés mécaniques de coulis à base de ciment à haute teneur en cendres volantes (remplacement de 60 % du ciment, par masse, pour des cendres volantes) avec ou sans l'utilisation de superplastifiant et/ou un agent anti-colloidal. Les propriétés rhéologiques ont été déterminées pour huit ratios eau/ciment+cendres volantes (eau/solides). Une enquête a été menée sur l'effet de superplastifiant et d'agent anti-colloidal sur le temps d'écoulement des coulis à bas ratio d'eau/solides et sur la stabilité des coulis à haut ratio d'eau/solides.

L'étude a révélé les points suivants :

- les cendres volantes réduisent le temps d'écoulement des coulis à bas ratio d'eau/solides (0,4 à 0,65);
- la stabilité des coulis à base de ciment est améliorée grâce à la présence des cendres volantes pour un ratio d'eau/solides plus grand ou égal à 0,65;
- les cendres volantes réduisent la résistance en compression ainsi que l'adhérence à bas âge (< 28 jours), comparativement à celles des coulis de référence (100 % ciment); par contre, cet écart diminue avec l'âge des échantillons;
- les cendres volantes réduisent le retrait des coulis à base de ciment.

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SYMBOLS

μ	Coefficient of Dynamic Viscosity
τ_0	Initial Yield Stress (Pa)
ν	Poisson's Ratio
τ_b	Shear Bond Strength (MPa)
$\dot{\gamma}$	Shear Rate (sec^{-1})
τ	Shear Stress (Pa)
E	Modulus of Elasticity (GPa)
f_c'	Compressive Strength (MPa)
n	Flow Behavior Index
η_p	Plastic Viscosity (cPs)
V_i	Volume of Sample at Beginning of Test
V_w	Volume of Decanted Bleed Water
W/C	Water to Cement Ratio
W/S	Water to Solids Ratio

ABBREVIATIONS

ACI	American Concrete Institute
ASTM	American Society for Testing and Materials
AWA	Anti-Washout Agent
C1	Sundance Fly Ash (Class C)
C2	Thunder Bay Fly Ash (Class C)
CLSM	Controlled Low Strength Material
CSF	Condensed Silica Fume
F	Point Tupper Fly Ash (Class F)
FA	Fly Ash
HVFA	High Volume Fly Ash
IDOT	Iowa Department of Transportation
PA	Preplaced Aggregates
PC	CSA Type 10 Portland Cement
PCA	Portland Cement Association
RHA	Rice Husk Ash
SCM	Supplementary Cementing Material
SP	Superplasticizer

CHAPTER 1

INTRODUCTION

Fly ash is a by-product of combustion of pulverized coal in thermal power generation. Fly ash along with silica fume, slag, and rice-husk ash is considered a supplementary cementing material (SCM). Supplementary cementing materials are materials which, in combination with Portland cement, contribute to the various characteristics of freshly mixed and hardened water cement systems. These materials contribute to the characteristics of the hardened system either by hydraulic cementitious action and/or by pozzalanic action. A material that possesses pozzalanic properties is one that, in the presence of water, reacts chemically with calcium hydroxide released during the hydration of Portland cement to form compounds with cementitious properties [Kosmatka et al., 1991].

The world wide production of fly ash, in 1989, was 400 million metric tons annually [Malhotra, 1993]. In Canada, currently 4.5 million metric tons of fly ash are available (1992), of which about 10 percent is used in concrete [Mirza et al., 1991]. In general, 15 to 20 percent fly ash is incorporated in concrete. Due to the growing public concern for the environment, conventional disposal of waste and by-products are no longer acceptable. This is forcing electrical utilities to look at fly ash as a potential resource rather than a waste material. Cement-based grouts as well as the concrete industry are prime candidates for the consumption of large quantities of fly ash. However, to make the incorporation of fly ash profitable both environmentally and economically, the replacement of cement for fly ash must extend 15 to 20 percent.

A recent study by Morgan et al. [1992] was carried out on high-volume fly ash (HVFA) shotcrete. Shotcrete mixtures with 0, 60, and 75 percent by mass of the total cementing material were investigated. The addition of fly ash increased the workability of the mixture besides reducing the amount of bleeding, when compared to an equivalent mixture without fly ash. The results demonstrated low strength development at early ages, but within acceptable limits. However, excellent long-term strength and durability characteristics have been observed with HVFA mixtures.

Applications such as soil, rock, and oil well grouting all require large amounts of cement, and are therefore perfect examples where HVFA could partially replace cement to produce low cost and durable grouts. The replacement of cement by high volumes of fly ash are not only environmentally beneficial, but also reduce the cost of materials. Depending on the availability, demand, and type of fly ash, the price may range from a few dollars to about \$ 90/ton, compared to the price of cement which is approximately \$100/ton [Malhotra, 1993].

This study presents the results of an experimental study of rheological and mechanical behavior of cement-based grouts incorporating three different types of fly ashes.

CHAPTER 2

SCOPE OF STUDY

The main objectives of this research project are:

- To carry out a complete bibliographic study on the use of fly ash, tests performed, characteristics, case studies, etc.
- To study the rheological and mechanical characteristics of HVFA and cement grouts made with three types of Canadian fly ash and CSA Type 10 Portland cement for eight different water to solids ratios (0.4, 0.45, 0.5, 0.55, 0.65, 0.8, 1.0 and 1.3) and two fly ashes for cement replacement levels (0 and 60 percent). Test series include the determination of three rheological properties (flow time, bleeding and setting time) and five mechanical properties (uniaxial compression, bond strength, modulus of elasticity, Poisson's ratio and drying-shrinkage).
- To evaluate the performance and behavior of HVFA and cement grouts made from three Canadian fly ashes by analyzing the results of the different test series .
- To determine the influence of superplasticizer (SP) and/or anti-washout agent (AWA) on the rheological characteristics of cement and HVFA grouts at low and high water to solids ratios.

CHAPTER 3

BACKGROUND

3.1 Grouting

The term grouting applies to many fields of engineering, such as civil, environmental, geotechnical, and transportation. To a mining engineer, grouting concerns seepage and strength control of tunnels, shafts, and mines. For civil and geotechnical engineers, grouting is used for the construction and maintenance of subbase, for consolidating, compacting, and/or reducing the permeability of rock and soil foundations of dams, building, bridges and other structures. From the perspective of an environmental engineer, grouts may be used to construct low-permeability barriers around waste landfills to prevent the contamination of ground water supplies. Grouts are also used in the field of transportation engineering as backfill material for utility trenches. Finally, they can also be used to fill abandoned tunnels, sewers, and underground voids. Table 3-1 illustrates the various applications of grouts [Kosmatka, 1990].

Grouting materials can be separated into two classes; suspension type, and solution type. The suspension type grouts include cement, clay, and asphalt, while the solution types include a wide variety of chemicals such as epoxies, phenoplast, aminoplast, etc. The difference between these two types of grouts lies in their rheological and mechanical characteristics.

The required rheological and mechanical properties of grouts vary with the designated application. For example, grouts with very low viscosity, high strength, and good durability are used in the consolidation of cracked concrete. On the other hand, high viscosity grouts with very low compressive strengths (<8.3 MPa), i.e. controlled low strength materials (CLSM), are often used to fill underground voids or as backfill around culverts and pipes.

3.1.1 Types of grouts

The following section presents a brief description of the most commonly used grouts. Chapter four and five will present a more detailed description of cement grout materials and their characteristics.

Table 3-1 Cementitious grout applications [Kosmatka, 1990]

Anchor bolts
Ballast grouting
Bonding grout
Ceramic tile
Column base plates
Dam foundations
Demolition
Flowable fill
Foundation grouting (stabilization)
Foundation jacking (lifting)
Ground anchors
Ground water control
Grout cleandown
Joints between precast units
Machine bases
Masonry walls
Oil wells
Postplaced-aggregate concrete
Post-tensioning ducts
Preplaced-aggregate
Railroad track stabilization
Reinforced masonry walls
Repair
Rock grouting
Slabjacking
Slurry-trench cutoff walls
Soil grouting
Stone-masonry restoration
Structural repairs
Subsealing (undersealing)
Toppings
Underlayments
Waterproofing of inground structures

3.1.1.1 Cement grouts

Cement grouts are fluid mixtures which contain water, cement, and in some cases chemical admixtures, fine sand, and/or supplementary cementing materials (SCMs). They are used for a wide variety of applications including the injection of cracks in concrete and rock masses, as well as filling large underground voids. Cement-based grouts are the most widely used type of grout. The advantage of using these grouts compared to chemical grouts are their high strength, low permeability, good durability and low cost.

The following section presents the terms used to describe the rheological and physical characteristics of grouts

- ***Consistency***

Consistency defines the ability of the grout to flow. Grout consistency can range from very fluid (i.e. like water) to thick, depending on the nature of the application. Admixtures may be used to vary the consistency of the grout without changing the water content.

- ***Workability***

Workability defines the energy required to place, handle, and consolidate a grout. An easily workable grout is one which requires very little energy to place. Working time refers to the amount of time during which the grout remains workable.

- ***Bleeding***

Bleeding refers to the upward migration of water. The result is the formation of a layer of water at the surface of the freshly placed grout.

- ***Setting time***

Setting defines the time when the grout has acquired a given consistency as a result of the chemical reaction between cement and water. The initial setting time is normally associated with the time when the grout is no longer workable, and the final setting time when the grout behaves as a rigid mass.

- ***Strength***

The compressive, bond and tensile strengths of a grout are related to the amount of cementitious material, the water content, and the degree of hydration. The strength (compressive and tensile) of a grout increases with a decrease in the water/cement ratio. However, the bond strength is related to the grout consistency. Fluid grouts normally have a better bond strength than thick grouts, since grouts with a fluid consistency easily penetrate the surface pores of concrete, thus providing a better bond.

• *Durability*

Durability defines the ability of a grout to withstand deterioration when subjected to such environmental conditions as chlorides, sulfates, erosion, corrosion, freezing and thawing, drying-wetting, alkali aggregate reactivity, etc.

The rheological as well as the mechanical properties of cement-based grouts are a function of :

- the quantity and type of cement used;
- the quantity of water added to the mixture;
- the presence of chemical admixtures such as:
 - water reducers (superplasticizers),
 - accelerators,
 - anti-washout agents (AWA), etc.
- the addition of supplementary cementing materials such as:
 - fly ash,
 - rice-husk ash,
 - blast-furnace slag,
 - silica fume,
 - natural pozzolans, etc.

The different types of cement, supplementary cementing materials (SCM), and chemical admixtures available on the market today are identified in detail in Chapter 4. In Chapter 5, the effects of these materials on the rheological and mechanical properties of grouts are investigated.

3.1.1.2 Clay grouts

These are made of clay and water, and in some cases an additive such as sodium oxalate or calcium chloride. The bonds formed in clay grouts possess very little strength. Their effectiveness lies in their ability to entrap between the sand grains and results in the formation of a cardhouse structure [Shroff and Shah, 1993]. Clay grouts are generally used where large volumes of low cost and low strength grout are required, such as in the impermeation and compaction of soil.

3.1.1.3 Chemical grouts

The ACI committee on the repair of cracks in concrete structures defines a chemical grout as "a solution of two or more chemicals that combine to form a gel, a solid precipitate, or a foam, as opposed to cement grouts that consists of a suspension of solid particles in a fluid," [ACI 224.1R, 1989]. Chemical grouts are used essentially to fill small cracks in rock foundations, to consolidate, and/or to reduce the permeability of the soil.

The greatest advantage of chemical grouts lies in their ability to penetrate very fine cracks (cracks as narrow as 0.05 mm have been successfully injected) [ACI 224.1R, 1989]. Chemical grouts also possess the following characteristics [Houlsby, 1990], :

- the absence of solid particles, as opposed to cement grouts;
- fluid like consistency;
- controlled setting time,
- and applicability in moist environments.

The disadvantages of chemical grouts can be listed as follows [Houlsby, 1990], [Shroff and Shah, 1993]:

- they can only be injected in small inactive cracks;
- at high temperatures ($> 40^{\circ}\text{C}$), they set too rapidly;
- at low temperatures ($< 10^{\circ}\text{C}$), they set too slowly and behave in a brittle manner;
- they possess very low compressive strengths;
- shrinkage is usually observed as result of the loss of water;
- most chemical grouts are toxic;
- they are more expensive than cement grouts.

In 1866, the first patent for a sodium silicate grout was awarded to Jeriorsky, a European scientist [Cambefort, 1964]. However, the greatest strides in the field of chemical grouts have been accomplished during the last three decades. A multitude of chemical grouts exist today. The following section describes the rheological and physical characteristics of the most important types of chemical grouts [Shroff and Shah, 1993] and [Karol, 1990]:

- Sodium silicate gel: Silicate precipitates in the form of a gel as a result of the reaction of sodium silicate with acidic salts. The sodium silicate solution comes in the form of a thick syrup, which does not penetrate fine cracks. The main advantage with this grout is the strength of the gel (high strength for a chemical grout, but weak when compared to a cement-based grout). A grout with a fluid like consistency can be produced by combining a dilute sodium silicate solution with an acid solution. However, the disadvantages of the modified sodium silicate grout are its low strength, prolonged setting time, and shrinkage effects. Such grouts are recommended for waterproofing cracks, but not to consolidate them.
- Lignosulphonate: Lignosulphonate is a by-product of the wood processing industry. Soluble lignosulphonate reacts with bichromate to form a firm gelatinous mass. The consistency of the grout is a function of the lignosulphonate concentration, and the setting varies with the pH. In general, these grouts have fluid like consistencies and are relatively inexpensive, however they are quite weak and are not recommended for consolidation. Furthermore, the gel may leach toxic materials into the environment.
- Phenoplast: Phenol or resorcinol react with formaldehyde to form a non-toxic gel. The consistency of these grouts is thick, which makes them inappropriate for rock grouting, unless high injection pressures are used. Moreover, these grouts are weak, hence they are not recommended for consolidation purposes.
- Aminoplast: When urea and formaldehyde react with acid, polymerization of the materials forms a set mass. The strength of the set mass is low in comparison to normal cement-based grouts. The product is relatively inexpensive and its fluid consistency facilitates the injection of very fine cracks, however, the material is toxic.
- Acrylamide: The grout consists of polymerized acrylamide monomers. Although the grout exhibits a fluid consistency and adequate strength, it is very toxic and handling precautions are necessary.

- Resins:

Epoxy, polyurethane, and polymer resins are all two component chemical systems consisting of a base resin and a hardener. The advantages of using resins are:

- good bond strength, specially to dry surfaces;
- good resistance in compression.

The disadvantages of using resins are:

- thick consistency;
- prolonged setting time at low temperatures ($< 10^{\circ}\text{C}$);
- rapid setting time at high temperatures ($> 40^{\circ}\text{C}$);
- shrinkage with time.

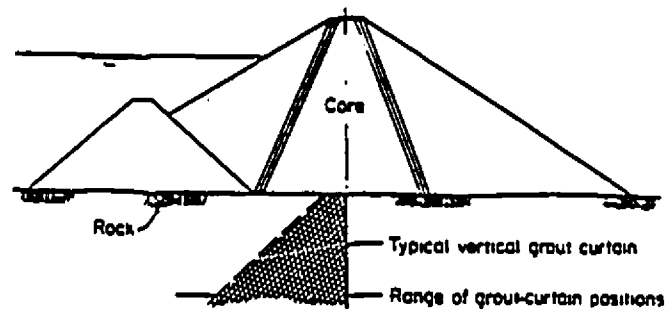
3.1.2 Grout applications

The application field of grouting is very wide ranging from structural to environmental engineering. The type of the grout required and its rheological and physical characteristics are dictated by the application. The following section presents some of the commonly encountered applications of grouts. Emphasis is placed on applications where large quantities of grout are required, that is, where the use of high-volume fly ash grouts are most beneficial economically and environmentally.

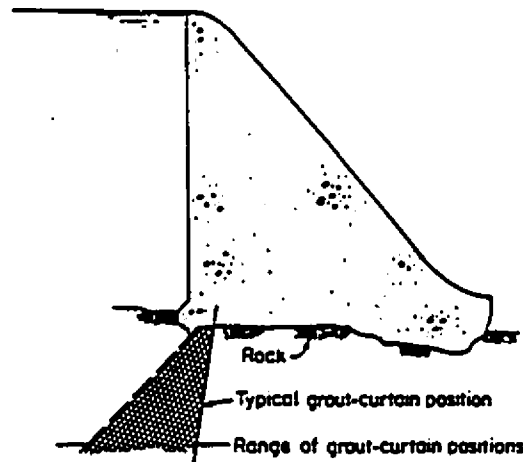
3.1.2.1 Rock grouting

Cement and chemical grouts when applied under pressure can seal and consolidate underground fissures in rock formations. The injection of cementitious product may be used to strengthen or make watertight the foundations under dams, buildings, and other structures.

Rock grouting is a very important part of the construction of a earth-fill dams and concrete gravity dams. The technique is used to increase the bearing capacity of heavily fractured or unstable rock and to decrease the water seepage under the dam (i.e. grout curtain, Fig. 3-1).



(a) Earth-core dam



(b) Concrete gravity dam

Fig. 3-1 Dam grout curtain [Kosmatka, 1990]

The depth to which the grout curtain should extend depends on the hydrostatic pressure in the reservoir. Therefore, for larger dams the depth of grouting is greater than that for smaller dams. As a rule of thumb, the grout curtain should extend to a depth which is about two thirds of the hydrostatic height of the dam.

The grout curtain is constructed by a line of grout holes which are perpendicular to the ground water flow vectors. Grouting should extend to all seamy rock that extends to the surface which are in the line of the grout holes. This prevents the water from going around the dam.

Conventional grouting techniques consist of boring a hole in the substrate to a desired depth and then injecting grout in the hole with the help of a pump. The grout starts by filling the hole and then proceeds to fill the cracks or voids which intersect the grout hole (Fig. 3-2 and 3-3). Pressure is used to push the grout into the network of cracks and voids. The grout will stop moving when one of the following conditions is attained:

- cement particles are too large for the crack or void;
- the pressure gradient is smaller than that required to initiate the flow of the grout;
- the grout has set and become too stiff to flow under the applied pressure;
- the crack has been completely filled.

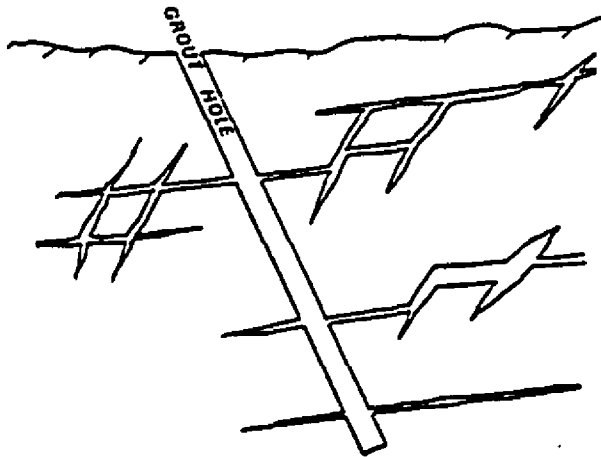


Fig. 3-2 Grout hole intercepting cracks
[Houlsby, 1990]

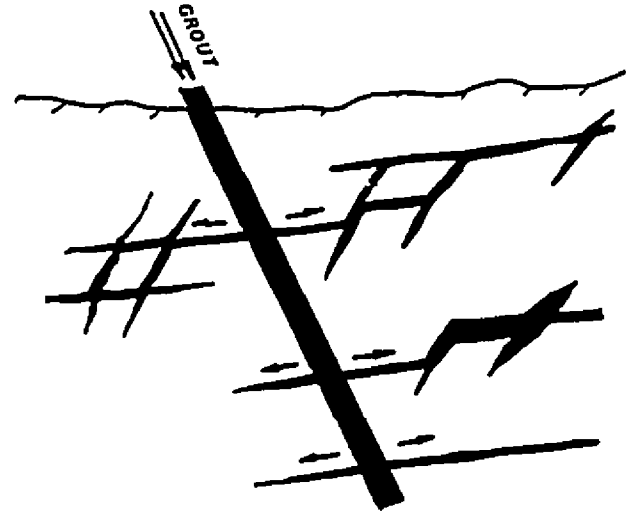


Fig. 3-3 Grout traveling down hole
and filling cracks[Houlsby,
1990]

Most grouts used for the consolidating rock and to reduce the water flow seepage are cement-based. Ideally, the network of fissures should be consolidated with the thickest grout possible. This ensures adequate strength, good durability, and low permeability. Too thin a grout will normally be washed away by underground water seepage and will fill the crack incompletely as a result of excessive bleeding. Furthermore, the cement particles of unstable grouts may separate from the water during injection, the free water may then create large upheaving forces and as a result displace the rock [Lombardi, 1985].

Grouting is usually initiated with a thin grout (water/cement ratio of 1.3 by weight) injected at a low pressure. Too thick a grout might prematurely clog the fissure(s). If the fissure(s) intercepting the grout hole takes a long time to fill, this indicates that the grout is flowing outside of the designated grouting area. Therefore, suggesting that the system is quite pervious. Hence, the grout should be thickened in steps until refusal has been reached. Refusal refers to the point where no more grout or very little grout for a defined

period of time under specified applied pressure can be pumped into the hole. Care must be taken to prevent displacing the rock due to excessive pressures.

Stable grouts made with normal Portland cement and water are recommended for the injection of rock foundations where the crack width is larger than 0.5 mm [Kosmatka, 1990]. The minimum crack width of the medium dictates the consistency of the grout (i.e. water/cement ratio) required to completely fill the network of cracks. If the width of the cracks in the medium are between 0.1 to 0.5 mm, grouts with very small particles in suspension or no particles at all, such as ultrafine cements (microfine) and chemical grouts, are recommended [Clark, 1987].

Most of the dams in North America are 50 to 100 years old. The original curtains were grouted with normal Portland cement, and now need to be rehabilitated. Rehabilitation of these dams has recently been initiated by the U.S. Army Corps of Engineers. For each dam, approximately 100 metric tons of grout is required [Clark, 1987]. Considering the cost of cement, grout curtain rehabilitation and construction is rather expensive, therefore, there is a need to develop low cost grouts which should possess the following characteristics:

- fluid consistency;
- very little or no bleeding;
- anti-washout characteristics in the fresh and hardened state;
- low permeability;
- good durability.

3.1.2.2 Soil grouting

Unlike rock grouting, four different methods are used to grout soils. They include penetration grouting, deep-soil mixing, compaction grouting, and jet grouting. These methods can be used:

- to increase soil bearing capacity;
- to reduce water flow (i.e. grout curtains);
- to reduce expected settlement;
- to stabilize and contain soil contaminated with hazardous chemicals.

Penetration grouting consists of using a thin cement grout to permeate the soil. Once the grout hardens, a rigid mass of soil and grout is formed which increases the bearing resistance and reduces the permeability of the soil (Fig. 3-4). Cement grouts made with conventional Portland cement with or without fill material such as fly ash are used to grout coarse gravel free of finer material. Sand with a grain size larger than 0.8 mm may also be grouted with normal cement. In the finer-sized materials, such as sand and silt, the voids are too small to allow normal cement to circulate. The water/cement ratio of the cement grouts may vary from 0.5:1 to 20:1 by volume. Microfine cements are recommended when grouting sand and silt with a grain size greater than 0.05 mm. Chemical grouts may also be used to consolidate sand and silt. However, many chemical grouts are toxic to the environment, hence they should be evaluated before they are used. Figure 3-5 summarizes the suitability of various grouts in different foundation materials.

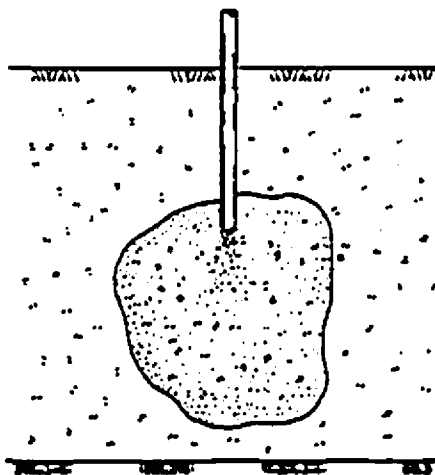


Fig. 3-4 Penetration grouting of soil [Shroff and Shah, 1993]

Grout type	Gravel		Sand			Silt		
	Coarse	Fine	Coarse	Medium	Fine	Coarse	Medium	Fine
Portland cement							
Microfine cement							
Chemical								
- high viscosity							
- low viscosity							

Fig. 3-5 Application range of various grouts in gravel, sand and silt [Houlsby, 1990]

Deep-soil mixing consists in agitating the soil with an auger and simultaneously injecting the soil with a cement grout. Grouting up to depths of 36 m (118 feet) can be achieved with this method [Kosmatka, 1990]. The advantage of deep-soil mixing is that a much broader range of grouts can be used, since there is no consideration for the nature of the foundation material.

Jet grouting is an alternative to conventional grouting when consolidating sand and clay substrates. Jet grouting can be performed in one of two ways. Conventionally, a hole is drilled into the ground in which a pipe, with a jet nozzle at the bottom, is inserted. A grout with a water/cement ratio of 1:1 (by volume) is jetted horizontally. The pipe is slowly rotated and raised, which results in a column of blended soil and grout (Fig 3-6 (a)). The second method consists in eroding the soft material by means of a very high pressure water jet and then filling the resulting space with a cement grout (Fig 3-6 (b)). Jet grouting may be used to construct seepage control barriers, to underpin structures, to increase bearing strength, and to strengthen the sides of excavations in substrates such as clays, silts, and fine sands where conventional grouting is impossible. A study by Allan and Kukacka [1994] demonstrated that jet grouting a cement grout containing superplasticizer and silica fume produced a grout-treated soil with a permeability coefficient less than 10^{-7} cm/sec, which is suitable for the containment barriers around hazardous waste landfills (permeability of concrete used in dam construction varies from 8 to 35×10^{-10} cm/sec).

Compaction grouting consists in injecting a thick cement and sand grout in a grout hole to consolidate the soil by compaction. The injected grout does not permeate into the surrounding soil but instead exerts a pressure on the surrounding soil. This method may be used:

- to increase pile friction by compacting soil around the pile;
- to stop settlement of structures;
- to stabilize culverts by compacting soil on both sides of the culvert.

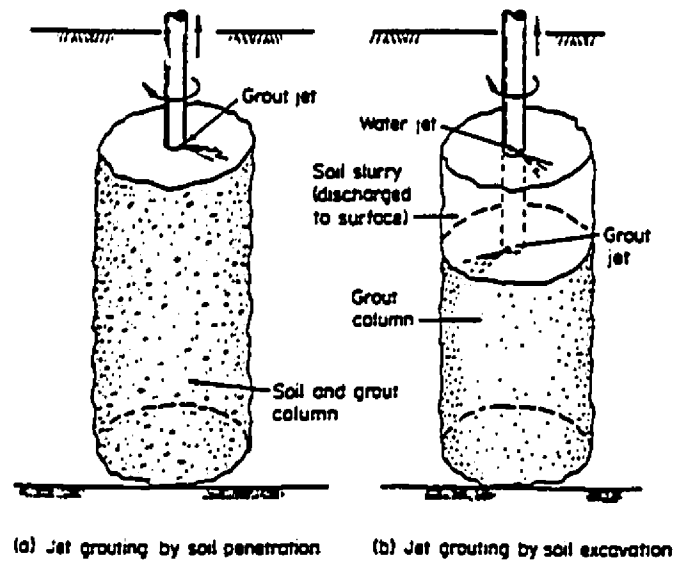


Fig. 3-6 Jet grouting [Kosmatka, 1990]

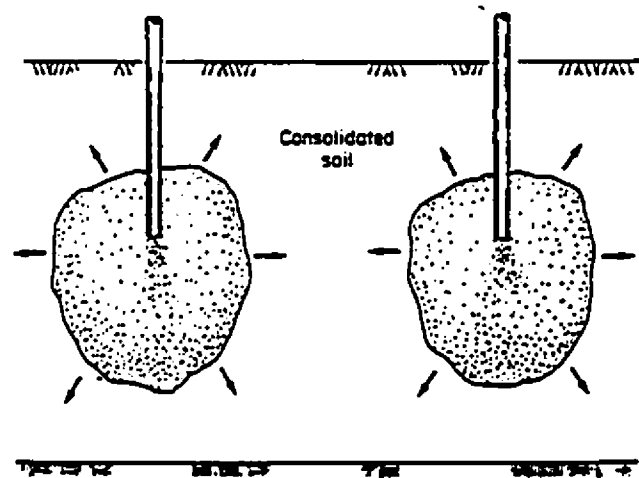


Fig. 3-7 Compaction grouting [Kosmatka, 1990]

3.1.2.1 Flowable fill

Flowable fill or controlled low strength materials (CLSM) are defined by the ACI Committee 229 as "a cementitious material that is in a flowable state at placement and has a specified compressive strength of 8.3 MPa (1200 psi) or less at 28 days. It is used primarily for non-structural applications below grade where low strength is required," [Larsen, 1990]. Flowable fills are essentially composed of fly ash, a small percentage of cement (4 to 5 percent cement is added to the dry weight of fly ash), and enough water to provide the desired consistency for the specified application. Sand or small aggregates are sometimes added to the mixture.

The strength of CLSMs is similar to that of compacted soils, which makes them an ideal backfill material for trenches containing ducts, pipes, and electrical cables, as well as backfill around concrete walls (Fig. 3-8 and 3-9). Flowable fill can also be used to fill abandoned tunnels, sewers, and other underground voids. The following is a brief description of the applications and advantages of flowable fills.

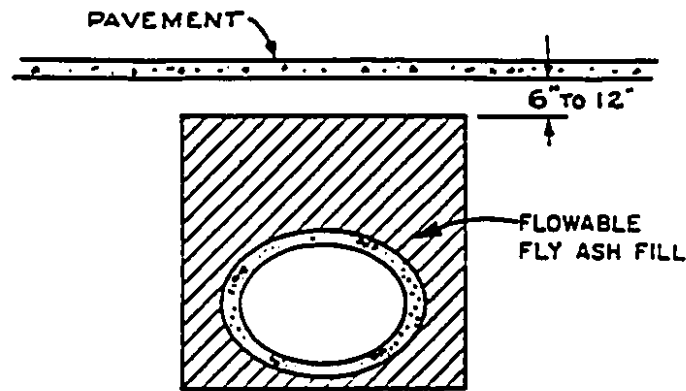


Fig. 3-8 Flowable fill encasing pipe [Krell, 1989]

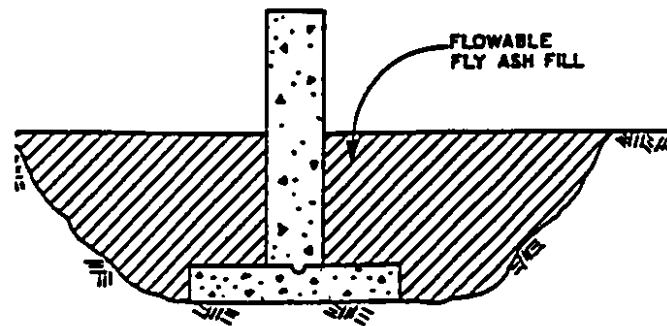


Fig. 3-9 Flowable fill around concrete wall [Krell, 1989]

- *Filling abandoned underground fuel tanks*

Filling abandoned underground fuel tanks with CLSMs is an alternative to the costly removal. In 1987, the Iowa Department of Transportation (IDOT) determined that removing the abandoned underground fuel tanks, near Ames, might disturb the foundation of the nearby garage. Instead, the fuel tanks were filled with a flowable fly ash mixture. The project was completed at a cost of \$ 1,140 (US), as opposed to removing the tanks which was estimated at \$ 8,000 (U.S.) [Larsen, 1990].

- *Filling voids under bridge piers*

Voids under bridge pier footings in the Robert Street Bridge, in St. Paul, Minnesota, were filled with a flowable fill. The erosion had been caused by the flow of water, and an erosion resistant material was needed. Approximately 125 m³ of flowable fill was pumped from the bridge deck to the voids under the two bridge piers, thus avoiding the need to build cofferdams [Larsen, 1990].

- *Backfill for utility trenches*

Controlled low strength materials may be used as backfill for utility trenches containing pipes, electric cable, etc. The advantages of using flowable fill as a backfill are [Larsen, 1990], [Krell, 1989], [Naik et al., 1990], [Ramme et al., 1994]:

- prevents the settlement of pipes;
- easy to excavate with a back hoe or an air spade;
- offers a different resistance than natural soils, alerting the operator of the presence of pipes in the event of future excavations;
- obtains density without compaction;
- assures uniform placement of materials;
- does not need to be vibrated or compacted;
- the corrosion potential of flowable fly ash grout is significantly less than that of typical soils.

3.1.2.4 Post-tensioned duct grouting

Post-tensioned construction uses either bonded or unbonded tendons to prestress concrete to counteract the tensile stresses caused by the applied service loads. Unbonded tendons are used mainly in slab construction, and bonded tendons are used for bridges and other structures where the risk of corrosion is present.

In post tensioned concrete construction, grouting the tendon duct protects the tendons from corrosion and develops a bond between the tendon and the surrounding concrete. This is achieved by completely filling the duct and making sure that all tendons are coated with grout. It is very important that grouting be successfully accomplished, since the service life of the structure depends on it. Moreover, the risk of corrosion is more

important and more dangerous in prestressed members than in conventionally reinforced concrete due to the high tension induced in the tendon [Venuat, 1989].

Grout used for bonding tendon ducts normally consists of Portland cement, water, and admixtures. Admixtures such as fly ash are used to increase flowability and to reduce bleeding. These grouts should exhibit the following characteristics [Venuat, 1989] and [Kosmatka, 1990]:

- sufficient flowability to fill duct without leaving any empty voids;
- very little bleeding (ASTM C940-89, bleeding water should not exceed 2 % of the total grout after three hours);
- adequate strength (30 MPa in compression and 4 MPa in tension determined using the flexural test after 28 days);
- very little shrinkage (shrinkage must be less than 2,800 microns/meter after 28 days at 50 % relative humidity);
- excellent freeze-thaw resistance;
- very little capillary adsorption.

A grout that is too fluid might bleed and form voids that can expose the tendon to corrosion problems (Fig 3-10). On the other hand, too thick a grout may leave unfilled voids and will require the use of high injection pressures which may damage the structure. The Portland Cement Association (PCA) recommends a maximum water/cement ratio of 0.45 when using CSA Type 10 and CSA Type 20 Portland cements [Kosmatka, 1990].

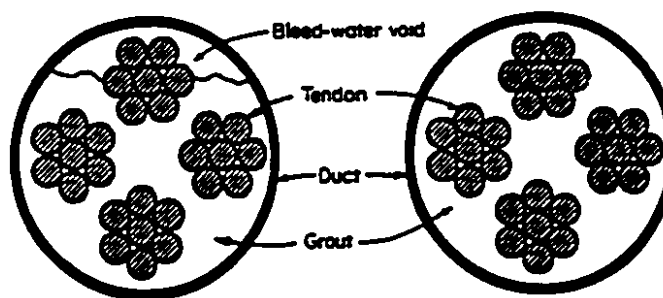


Fig. 3-10 Grouted tendon ducts with and without bleed water [Kosmatka, 1990]

3.1.2.5 Preplaced aggregate concrete

Preplaced aggregate (PA) concrete is defined by the ACI 304.1R Committee as "concrete produced by placing coarse aggregate in a form and later injecting a Portland cement and sand grout, usually with admixtures, to fill the voids." The use of PA concrete dates back to the late 1930's. Since then the technique has been successfully used for numerous applications. The following section briefly describes the various applications and advantages of PA concrete:

- In structural members where excessive heat of hydration must be taken into consideration, the PA method enables the cooling of the aggregate before the injection of the grout. Furthermore, the grout itself may be cooled prior to injection.
- In structural members where reinforcement is too closely spaced to permit the use of vibrators. The aggregate is placed around the reinforcement as the forms are erected. Finally, the member is grouted into a monolithic unit of PA concrete.
- When heavyweight (high density) concrete is needed, such as dams (mass concrete), the risk of segregation is eliminated by preplacing the heavyweight aggregate.
- When embedded items require high slump concrete which may result in segregation, the PA method should be considered.
- In remote regions where ready-mixed concrete is not available, PA concrete may be more economical than constructing an on-site plant.

The grout consists of normal Portland cement, water, and may include fine sand. Fly ash and natural pozzolans are often included, since they improve the workability and lower the permeability of the grout. In the majority of applications, class F fly ash has been used. The proportions of pozzolana to cement is usually 20 to 30 percent by weight, however as much as 40 percent fly ash is sometimes used [ACI 304, 1991].

3.1.2.6 Oil well grouting

Oil well grouting is used to restrict the movement of water, oil, or gas between the rock and the steel casing and to bond the casing to the rock. The casing is used to extract the oil from the ground and to prevent the dispersion of oil into the surrounding rock.

Grouts used for oil well grouting are made with Portland cement or oil well cements depending on the depth, temperature, and pressure. Oil well grouting, rock grouting, and soil grouting are typical examples of grouting operations that require enormous amounts of cement. The use of supplementary cementing materials is recommended to reduce cost without sacrificing grout quality.

3.1.2.7 Grouting cracked concrete

Cracks occur in concrete structures during construction and/or while in service. Cracks formed during construction are principally due to temperature differences between the fresh concrete and the cool surrounding air, and cracks which occur during the service life of the structure may be due to:

- seasonal temperature variations such as freezing/thawing and wetting/drying cycles;
- stresses from the applied load on the structure such as retained water, vehicular traffic, or earthquakes;
- alkali-aggregate reactivity, etc.

Taking into consideration the high cost of constructing concrete structures, there is little choice but to maintain and rehabilitate our existing infrastructure. The injection of cracks with a cement or chemical grout is the most popular approach to deal with this kind of degradation. Grouts suitable for injection should possess the following characteristics:

- fluid consistency for a complete penetration of the crack;
- very little bleeding;
- good bonding with concrete in dry, moist, and wet conditions;
- good mechanical resistance (compression, tension, and shear);
- good freeze/thaw resistance.

The procedure for grouting a concrete structure, such as a dam, is much the same as rock grouting. Grout holes are drilled to intercept the fissure along the longest possible length. Grout is then injected into the grout hole to fill all intercepting cracks. A water/cement ratio of 1.3 by weight is normally used to start. This will prevent premature blocking of the crack. The grout is thickened successively until refusal. For cracks widths > 0.5 mm, normal Portland cements are used, however for smaller cracks (< 0.5 mm) microfine cements and chemical grouts are recommended [Langevin, 1993].

3.1.2.8 Grouting old stone masonry structures

Many buildings, bridges, and canals were constructed of stone and mortar during the 17th century. In Canada, most of these structures were built with limestone. Old stone masonry structures can be divided in two categories:

- Those with earth fill on one side such as retaining walls, abutments, and canal walls;
- Those on which no earth rests such as piers, buildings, and open spandrel arches.

With time, the mortar in these structures has gradually deteriorated as a result of freezing/thawing, wetting/drying cycles, and other environmental effects. These structures need to be repaired, given their historical importance. In order to preserve their originality, no exterior repairs can be undertaken. Thus, the solution is to grout the structure.

One such example of stone masonry repair was carried out in November 1994, on one of the fifty locks between Ottawa and Kingston on the Rideau Canal (Fig. 3-11). Over the years the mortar had deteriorated as a result of the constant pressure of water, which increased permeability and caused leaching of the inside wall.

The Rock Mechanics Laboratory of the University of Sherbrooke was mandated to formulate a high performance grout for the job. CSA Type 30 Portland cement with silica fume as well as a microfine cement (Spinor A-12) were used. Superplasticizers were used to reduce the water/cement ratio of the grout and at the same time increase its fluidity and stability. Over 3000 L of grout were successfully injected into the west wall of lock no. 47 [Concrete Canada, vol. II No. 1 1994].



Fig. 3-11 Restoration of Lock 47 on the Rideau Canal [Concrete Canada, vol. II No. 1 1994]

3.2 Rheology of fluids

Before studying the rheological properties of grouts, it would be useful to define the term 'rheology'.

"Rheology is the branch of physics concerned with the flow and change of shape of matter, especially the viscosity of fluids." [Collins Dictionary, 1988]

Most materials may be designated as being either a solid, a liquid or a gas, although some materials have properties of dual designation. The difference between a solid and a fluid relies on the deformation characteristics of the material. A solid is a material that experiences a finite deformation in response to an applied set of forces. A fluid is defined as a material that deforms continuously when subjected to a set of forces. There are some materials that exhibit characteristics of either a solid or a fluid depending on the level of the shear stress applied [Olson and Wright, 1990].

A cement grout is a material composed essentially of cement, water and in some cases admixtures. Once the components of the grout come in contact with one another, a variety of chemical and physical reactions occur which give the grout its particular rheological properties. Factors which effect the rheological properties of grouts are:

- fineness and particle size distribution of cement and supplementary cementing materials,
- water to solids ratio,
- chemical composition of cement and supplementary cementing materials (SCM),
- admixtures,
- mixing time and
- temperature.

The water/cement ratio and the fineness of the cement are the two principal factors which dictate the behavior of the suspension system. Moreover, small clusters of cement particles called floccules tend to form in the water cement systems. This effect combined with inter-particle forces and attraction of the water to the cement surfaces also has an effect on the rheological behavior [Shaughnessy and Clark, 1988].

The purpose of studying the rheological properties of grouts is to estimate the deformations caused by the application of a specific set of forces or the force required for a given deformation to occur. For example, when grouting a crack, a good understanding of the cement grout is essential to evaluate the displacement and flow rate for maximum crack filling. Knowledge of the flow properties is useful in a number of other applications. For some applications, dense, impermeable composites are required. To achieve this, an understanding of the cement mixture in the plastic and fresh state is essential.

For liquids and soft solids, such as suspensions, four types of fluid behavior exists (Fig. 3-12) [Banfill, 1994]:

- Newtonian,
- dilatant,
- pseudoplastic and
- Bingham.

The behavior of these four types of fluids will be reviewed with after a brief discussion of fluid viscosity.

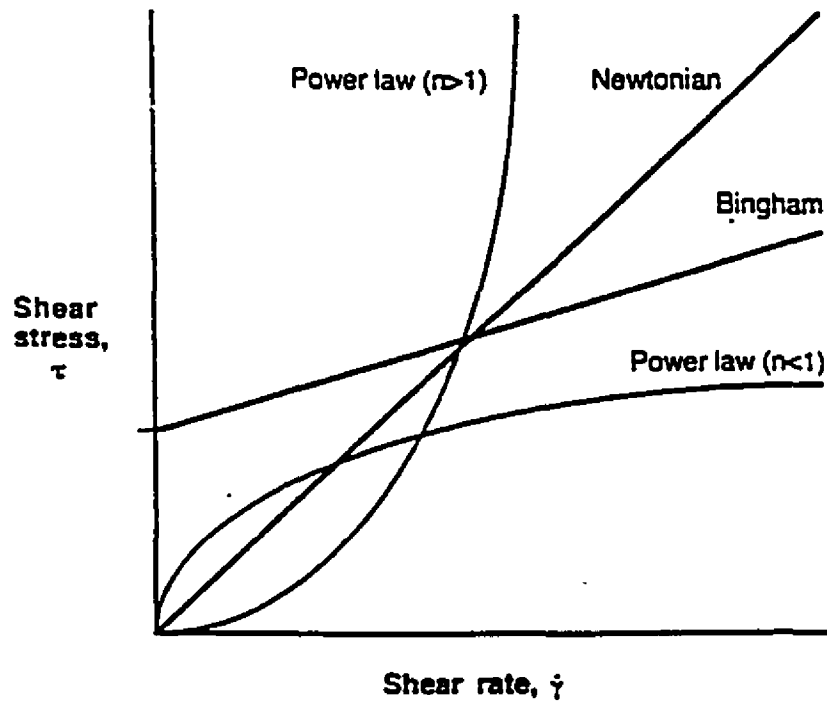


Fig. 3-12 Rheological behavior of the different fluids [Banfill, 1994]

3.2.1 Viscosity

The property of fluid viscosity is a measure of the internal friction of a fluid [Brookfield, 1993]. Viscosity (μ) can be represented by considering two parallel planes of fluid of equal area separated by a distance dx . Both planes are moving in the same direction at different velocities (Fig. 3-13). Newton postulated that the shear stress within a fluid is proportional to the velocity gradient between the two planes [Olson and Wright, 1990].

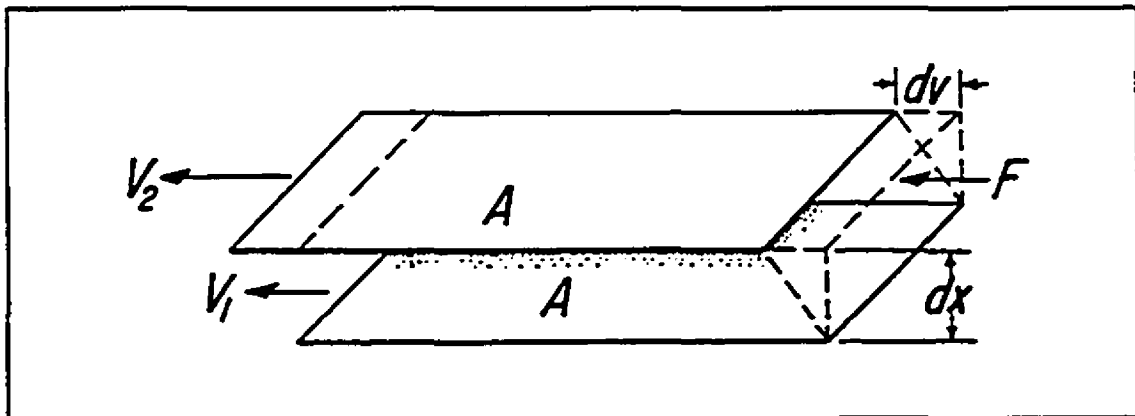


Fig. 3-13 Newtonian flow model [Brookfield, 1993]

The relationship for viscosity is as follows:

$$\tau = \mu \frac{dv}{dx} \quad (3.1)$$

The term τ indicates the force per unit area required to produce the shearing action, and μ is the factor of proportionality known as the coefficient of dynamic viscosity. The velocity gradient in the above equation (dv/dx) is also known as the shear rate (γ). Consequently, viscosity is the factor of proportionality which describes the resistance of a fluid to motion, in other words it is the shear stress required to overcome the internal friction forces within a fluid for a defined velocity gradient. Among the different flow behaviors of fluids, two families can be identified: Newtonian and non-Newtonian fluids.

3.2.1.1 Newtonian fluids

For a Newtonian fluid, such as water or oil, the relationship between the shear rate and the shear stress produces a straight line passing through the origin (Fig. 3-14 (a)). Hence, the viscosity is constant and is independent of the shear rate (Fig. 3-14 (b)). Other fluids which display more complex behavior are referred to as non-Newtonian fluids.

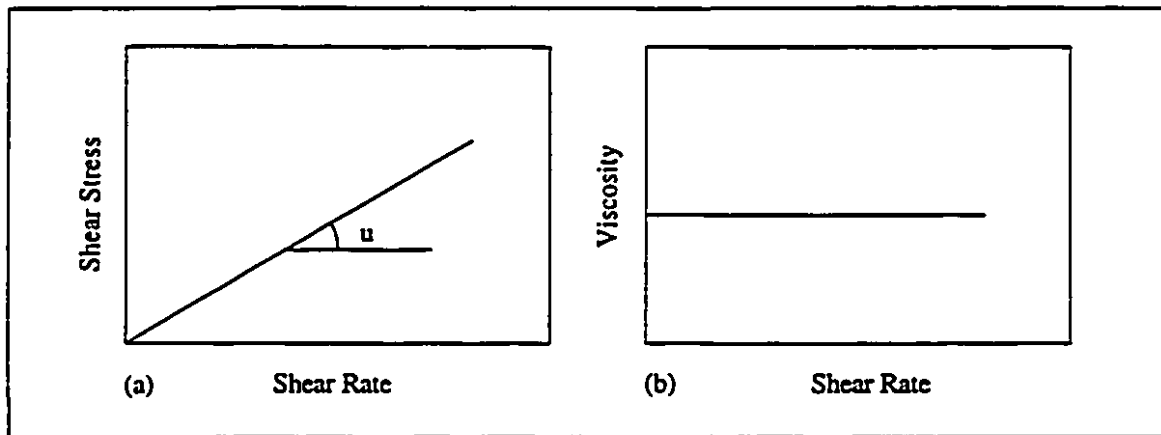


Fig. 3-14 Newtonian flow behavior

3.2.1.2 Non-Newtonian fluids

For a non-Newtonian fluid, the shear stress to shear rate ratio is not constant. The viscosity of the fluid is dependent on the shear rate. There is a variety of non-Newtonian fluids that are characterized by a change in viscosity with a change in shear rate. A brief discussion of the more important fluids follows:

(a) Pseudoplastic fluids

This type of fluid displays an increase in shear stress with an increase in shear rate (Fig. 3-15 (a)), however, a decrease in fluid viscosity is observed with an increase in the shear rate (Fig. 3-15 (b)). Pseudoplastic behavior is the most common among non-Newtonian fluids, they include paints, emulsions and dispersions [Brookfield, 1993]. This type of flow behavior is often termed 'shear thinning'. Such a behavior can be described by several models, the most common being the power law relationship [Banfill, 1994]:

$$\tau = k \dot{\gamma}^n \quad (3.2)$$

where τ is the shear stress, k is the fluid consistency and n is the flow behavior index ($n < 1$).

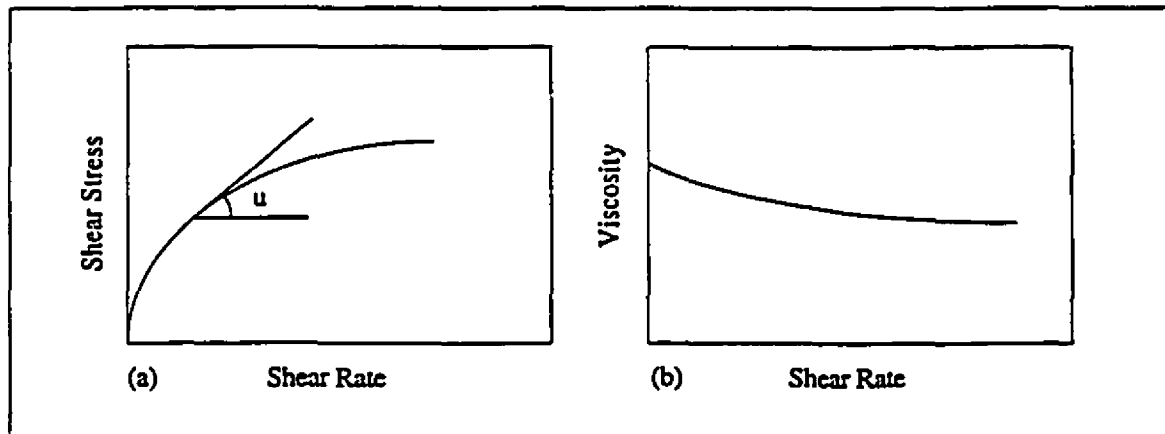


Fig. 3-15 Pseudoplastic flow behavior

(b) Dilatant fluids

This type of fluid also displays an increase in shear stress with an increase in shear rate (Fig. 3-16 (a)), however, an increase in fluid viscosity is observed with an increase in the shear rate (Fig. 3-16 (b)). This behavior is termed 'shear thickening'. Very few materials exhibit this type of behavior, they include clay slurries, candy compounds, corn starch in water, and sand water mixtures. The power law model (3.2) is also applicable here, but in this case $n > 1$.

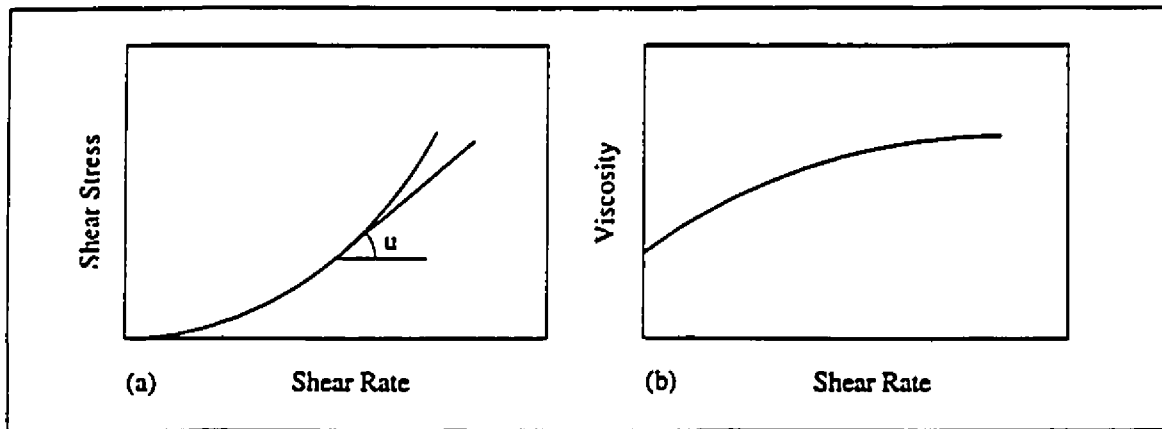


Fig. 3-16 Dilatant flow behavior

(c) Bingham fluids

The force required to initiate flow in a Newtonian fluid is initially small. On the other hand, Bingham or plastic fluids behave as solids when the stress applied is under a certain threshold value [Olson and Wright, 1990]. When the stress applied to the fluid exceeds this limit, the material starts to flow and behaves as a Newtonian fluid (Fig. 3-17 (a)). The shear stress limit is called the initial yield stress or the cohesion value (Fig. 3-17 (a)), and the viscosity above this point is called the plastic viscosity (Fig. 3-17 (b)).

The stress-strain relationship for Bingham bodies can be described by the following equation:

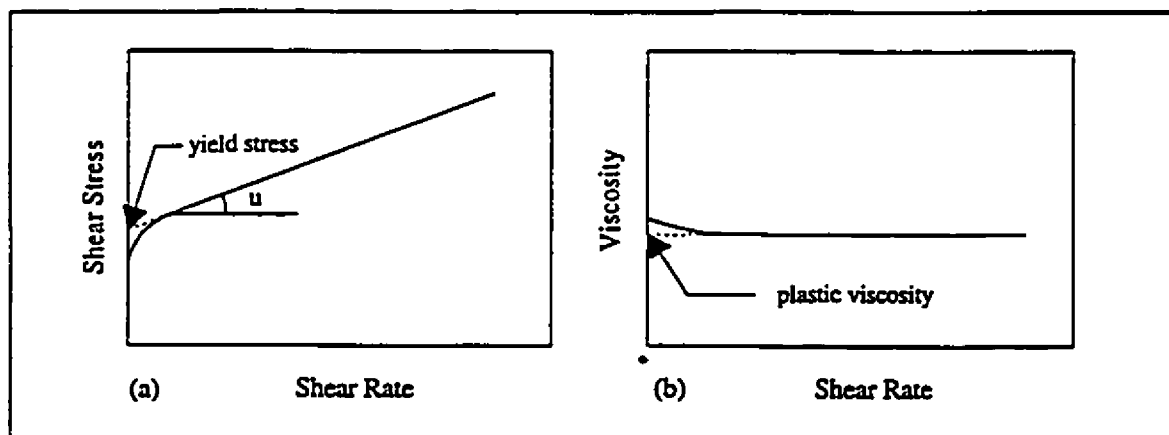


Fig. 3-17 Bingham flow behavior

$$\tau = \tau_0 + \eta_p \dot{\gamma} \quad (3.3)$$

η_p = plastic viscosity (Pa • sec)

τ = shear stress (Pa)

τ_o = initial yield stress (Pa)

γ = rate of shear (sec⁻¹)

Depending on the cement composition and experimental conditions, cement pastes exhibit flow behavior of any of the four main types: Newtonian, Bingham plastic, pseudoplastic, and dilatant [Shaughnessy and Clark, 1988]. A majority of cement pastes and grouts exhibit non-Newtonian flow behavior. Depending on the concentration of solid particles, the flow behavior varies progressively from Bingham plastic to dilatant. At high water/solid ratios, grouts display Bingham plastic flow behavior. With the addition of a superplasticizer, some cement pastes can behave as non-Newtonian fluids, but this is rarely observed. Superplasticizers will disperse the agglomeration of cement particles, i.e. floc structure, thus lowering the viscosity and cohesion. Domone and Thuraiatnam [1988] conclude that grouts can be satisfactorily described by the Bingham model. Thus, grouts exist in two contrasting states: (1) under static conditions, the grout resembles a weak solid, with a finite yield value, (2) with application of stress greater than the yield value, the grout behaves as a fluid. This factor is of great importance in the theory of rock grouting for the following reasons:

- a minimum pressure gradient is required to initiate the flow of the grout ;
- cohesion produces a velocity profile which is different from that produced by a Newtonian fluid (Fig. 3-18). Bingham bodies produce a stiff kernel in the center of the velocity profile for a flow through a circular pipe, as opposed to Newtonian fluids which produce a parabolic velocity profile [Lombardi, 1985]. The kernel is formed in the zone where the shear stress is lower than the yield stress (Fig. 3-19), and when the pressure gradient decreases, it grows until it reaches the wall (of a crack) and stops the flow.

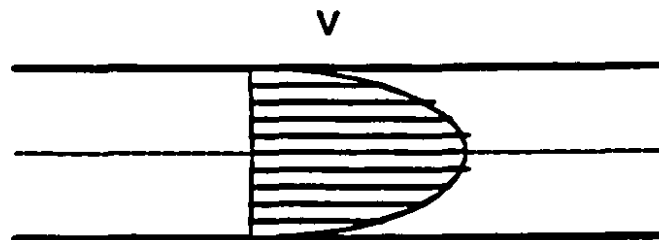


Fig. 3-18 Velocity profile for Newtonian fluids (pipe flow) [Olson and Wright, 1990]

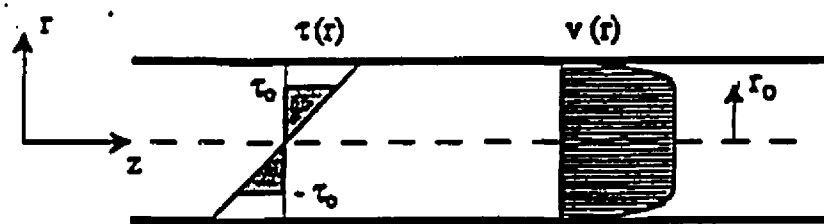


Fig. 3-19 Velocity profiles for Bingham fluids (pipe flow) [Olson and Wright, 1990]

3.2.2 Thixotropy

Thixotropy can be defined as the decrease in shear stress with time when a fluid is subjected to a constant shear rate. This behavior can be recognized when periodic tests at identical ascending and descending shear rates do not coincide. The descending branch of the flow curve is to the left of the ascending curve (Fig. 3-20 (a)). This phenomenon is attributed to the destruction of inter-particle bonds as a result of a constantly applied shear rate. Therefore, hysteresis loops are observed when the fluid in question is subjected to cycles of loading. The yield stress and plastic viscosity depend on the previous shear history. A hysteresis loop gives only an indication that structural breakdown has occurred, however, it does not provide a quantitative measure [Banfill, 1994]. Such a fluid recovers its original rigidity with time [Ritchie, 1965]. Hence, the thixotropic phenomenon is reversible. In the case of grouts, if left undisturbed for some time, there is an increase in the shear stress required to initiate the flow again. This property could cause a problem if grouting is interrupted for some time.

Rheopectic behavior is the reverse of thixotropic flow, inter-particle bonds are created as a result of a constantly applied shear rate. Therefore, the descending curve (curve representing measurements taken during successive decrease in the shear rate after initial loading) is to the right of the ascending curve (curve representing measurement taken during increments in the shear rate) (Fig. 3-20 (b)). In other words, the observed shear stress increases with the application of a constant shear rate. Ritchie [1965] indicates that various degrees of thixotropy and rheopecty can be observed with cement grouts, but there is no apparent trend.

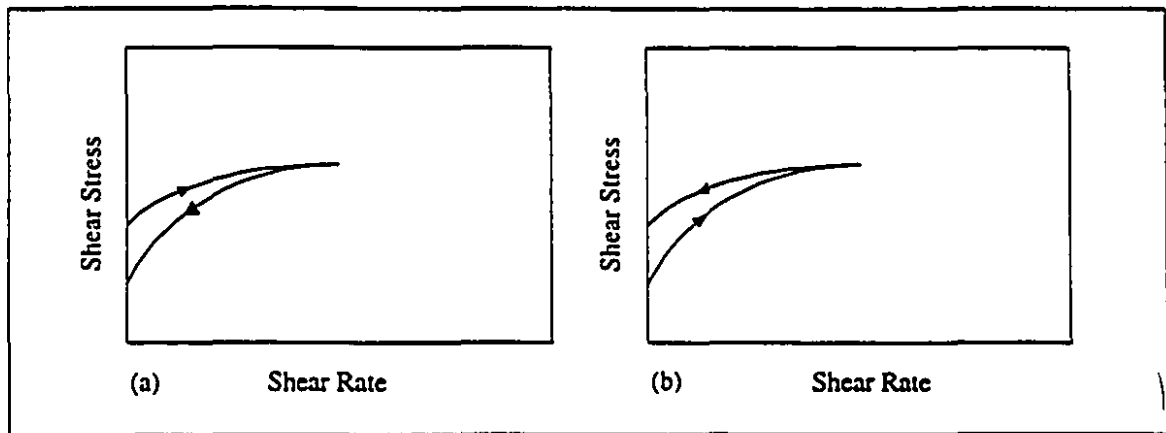


Fig. 3-20 Bingham thixotropic and rheopectic behavior [Ritchie, 1965]

CHAPTER 4

GROUTING MATERIALS

This chapter presents a detailed review of cementitious materials, supplementary cementing materials, and chemical admixtures used for grouting. More details can be found in standard textbooks on the subject [Neville, 1981], [Mehta, 1986].

4.1 Cements

Cement may be described as a material with adhesive and cohesive properties capable of bonding fragments or masses of solid matter to form a compact whole [Neville, 1981]. The ancient Egyptians were the first to use cement pastes by combining calcined impure gypsum with water. Later, the Greeks and Romans used calcined limestone and water in combination with sand and crushed stone. Hence, this was the first concrete. It was then discovered by the Romans that if finely ground volcanic ash (pozzolana) is combined with lime, a cement paste is produced which can be used under water.

Cements can be divided into two categories: hydraulic and non-hydraulic cements. Hydraulic cements are defined as cements that not only harden by reacting with water but also form a water-resistant product. On the other hand, the products of hydration of non-hydraulic cements are not water-resistant. These cements are produced by the calcination of gypsum or carbonates such as limestone. Non-hydraulic cements can be rendered hydraulic by addition of pozzalanic materials, the active silica and alumina components of these materials react with the lime to produce water-resistant cementitious products.

The majority of cements used today are of the hydraulic nature. This can be attributed to its water-resistant characteristics. A variety of hydraulic cements are available on the market today, they include:

- Portland cements,
- microfine cements, and
- special cements.

4.1.1 Portland cements

The invention of Portland cement is attributed to Joseph Aspdin. In 1824, Aspdin obtained a patent for his discovery, which he named Portland since the cement once hydrated resembled the color and quality of Portland stone quarried off the Isle of Wright. The name has remained to this day to describe a cement which is produced by calcareous and argillaceous, or other silica-, alumina, and iron oxide-bearing materials, burning them at a clinkering temperature, and finally grinding the clinker with gypsum or calcium carbonate to make cement [Neville, 1981].

Portland cement is the most widely used of all hydraulic cements. These cements may be used to produce concrete, mortar, grout, etc. Portland cement is used world wide, since the raw materials required to make cement are found in nearly all countries.

Calcium silicates are the major constituents of Portland cement, hence the raw materials must provide calcium and silica in suitable proportions. Limestone, chalk, marl, and seashells are naturally occurring materials which contain calcium. Clays and shales are preferred sources of silica to produce calcium silicates. Clay frequently contains alumina (Al_2O_3), iron oxide (Fe_2O_3) and alkalis. The presence of these compounds helps the formation of calcium silicates. Hence, when insufficient amounts of Al_2O_3 and Fe_2O_3 are present in the raw materials, additions of these compounds can be accomplished by incorporating secondary materials such as bauxite and iron ore [Mehta, 1986].

4.1.1.1 Manufacturing process

The production of Portland cement can be described in the following steps:

(1) Homogenization and grinding of raw materials

Rock is extracted from the quarry, then it is ground and crushed into smaller fragments (<20 mm in size). The crushed stone is finally blended together and stockpiled. From the chemical analysis of the stock piled material, the proportions of the raw materials are determined based upon the desired composition of the cement.

(2) Pulverization of raw material

Before the blend of raw materials is heated, it must be pulverized into fractions smaller than 75 μm . This is achieved by using a ball or roller mill.

(3) Baking the fine powder

The fine powder is heated to a temperature of 1450 °C to 1650 °C in a rotating kiln, where the calcium and silicate compounds react to form clinker. The clinker is in the form of small pellets of 5 to 25 mm in diameter.

(4) Pulverization of clinker

The final stage of the cement production consists in grinding the clinker in particle sizes mostly smaller than 75 μm in diameter. Approximately 5 % by weight of gypsum or calcium carbonate is added to the pulverized clinker to control early setting of the cement.

4.1.1.2 Chemical composition

Portland cement is essentially composed of four major constituents (C_3S , C_2S , C_3A and C_4AF). It is customary to express these compounds in terms of oxides of the elements, which are presented using abbreviation as shown in Table 4-1. Each of these compounds have different properties with respect to the rate of hydration, heat of hydration, and strength.

Table 4-1 Symbols for oxides and chemical compounds

Oxide	Abbreviation	Compound	Abbreviation
CaO	C	$3\text{CaO}.\text{SiO}_2$	C_3S
SiO_2	S	$2\text{CaO}.\text{SiO}_2$	C_2S
Al_2O_3	A	$3\text{CaO}.\text{Al}_2\text{O}_3$	C_3A
Fe_2O_3	F	$4\text{CaO}.\text{Al}_2\text{O}_3.\text{Fe}_2\text{O}_3$	C_4AF

Minor compounds such as MgO, TiO_2 , Mn_2O_3 , SO_3 , K_2O and Na_2O are also present. These compounds amount only to a few percent of the total mass of cement. Two of the minor compounds are of great interest: the oxides of sodium and potassium. These alkalis have been known to react with some aggregates (e.g. calcareous aggregates),

resulting in expansion and deterioration of the concrete. Although they are found in small amounts, compared to the calcium compounds, their presence is of great importance [Neville, 1981]. The quantities of the various compounds vary from one cement type to another. Typical oxide composition limits for Portland cements are listed in Table 4-2 [Neville, 1981].

Table 4-2 Approximate composition limits of Portland cement [Neville, 1981]

Oxide	Content, %
CaO	60-67
SiO ₂	17-25
Al ₂ O ₃	3-8
Fe ₂ O ₃	0.5-6
MgO	0.1-4
Alkalis	0.2-1.3
SO ₃	1-3

4.1.1.3 Crystal structure and reactivity of compounds

In order to understand the cement hydration process and the physical differences between the different Portland cements, one must understand the reactivity of the chemical compounds of cement. In this section, the reactivity of the calcium silicates, aluminates and the ferroaluminates is examined briefly. As discussed earlier, the four major compounds in cement are C₃S (tricalcium silicate), C₂S (dicalcium silicate), C₃A (tricalcium aluminate) and C₄AF (tetracalcium aluminoferrite), however, the exact chemical composition of the four compounds is actually much more complex. In reality, small amounts of impurities exist, however these do not alter the crystal structure. It should be noted that large amounts of impurities may alter the crystal structure. The reactivity of a cement depends on its crystal structure, fineness and temperature of hydration. The rate of hydration of the pure compounds of cement is demonstrated in Fig. 4-1.

• *Calcium silicates*

Placement of the oxygen ions around calcium leaves large voids, which accounts for the reactivity of C₃S. The C₂S compound has a more organized structure (less voids) and is, therefore, less reactive.

- *Calcium aluminate and ferroaluminate*

Both the structure of C₄AF and C₃A are very complex, but are characterized by voids in the molecular structure which account for their high reactivity [Mehta, 1986].

The reactivity of a cement is affected by its fineness. For a given compound the rate of reactivity, hence the strength development, can be increased by grinding the cement finer (Fig. 4-2) [Mehta, 1986]. As a result, more heat is generated during hydration. The fineness of a cement can be characterized by its specific surface, m²/kg. Measurement of cement fineness can be achieved by using the ASTM Standard C204 "Standard Test Method for Fineness of Portland Cement by Air Permeability Apparatus".

4.1.1.4 Hydration of cement

Anhydrous Portland cement acquires its adhesive property only when mixed with water. The process of the chemical reaction between water and cement is known as the hydration of cement. This process yields products that possess setting and hardening characteristics [Mehta, 1986]. The main products of hydration can be classified as calcium silicate hydrates (CSH) and tricalcium aluminate hydrates.

- *Calcium silicate hydrates*

The product of hydration of the silicates, C₃S and C₂S, is calcium-silicate-hydrate, C₃S₂H₃ (CSH). The reaction of hydration for C₃S and C₂S can be written as follows [Neville, 1981]:



One can see that the quantity of Ca(OH)₂ produced by C₃S is more than twice that produced by C₂S for approximately the same quantity of water. In fact C₃S, based on the above stoichiometric equations, produces 61 % CSH and 39 % Ca(OH)₂. On the other hand, C₂S produces 82 % CSH and 18 % Ca(OH)₂. The adhesive properties of hydrated cement paste are due to the formation of CSH. Hence, the strength would be greater for high C₂S cements than for high C₃S cements. Calcium hydroxide produced by the hydration of the silicates

reduces the durability of the cement with respect to acid and sulfate attack. Therefore, a cement with a high C_2S content will increase the durability of the hydrated cement paste [Mehta, 1986].

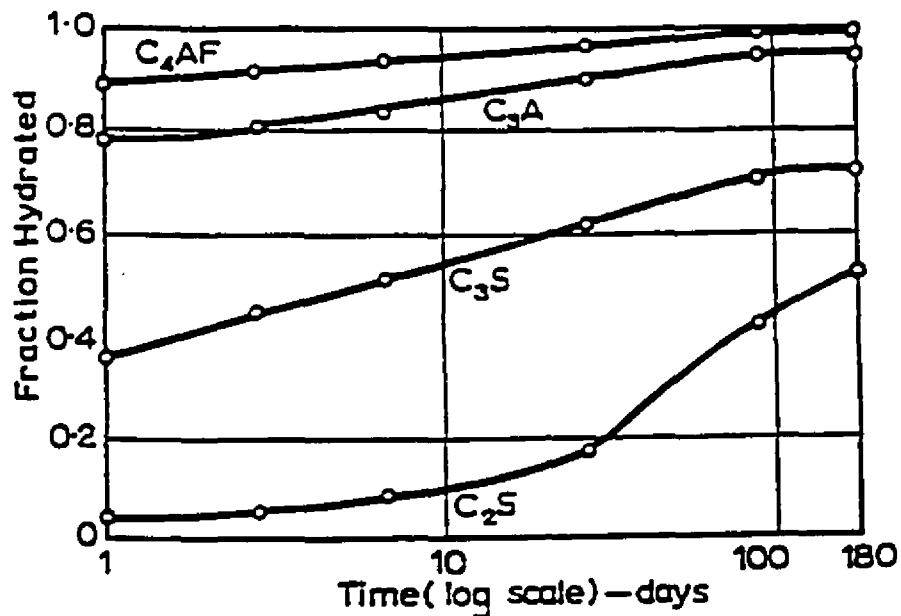


Fig. 4-1 Rate of hydration of pure compounds [Neville, 1981]

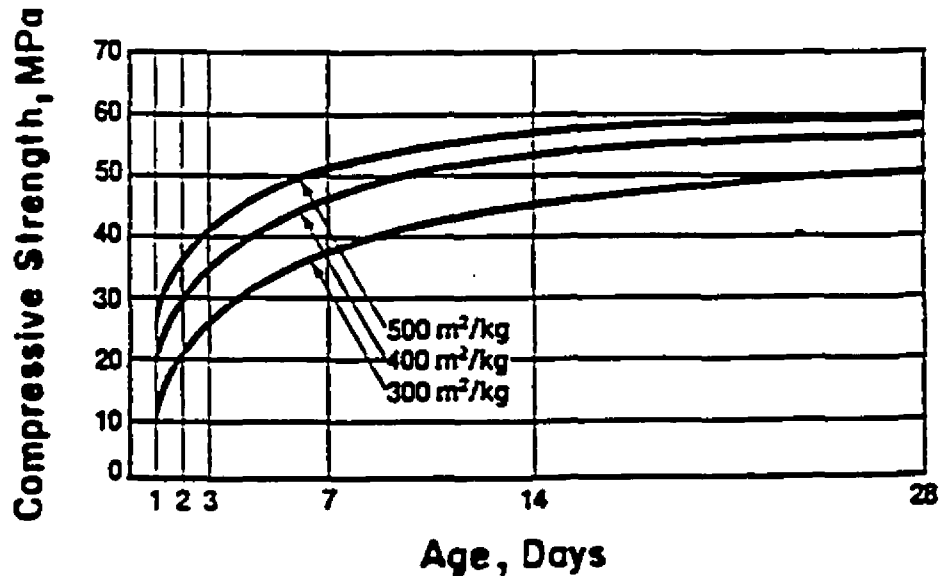


Fig. 4-2 Influence of cement fineness on strength [Mehta, 1986]

As discussed in the previous section, C_3S is more reactive than C_2S , hence C_3S will hydrate at a faster rate and will be responsible for the early strength of the cement paste. The quick rate of C_3S hydration is an important factor in the design of high early strength cements. On the other hand, cements containing high C_2S will produce less heat during hydration.

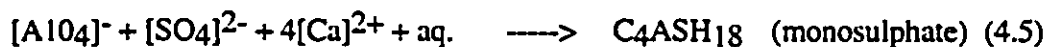
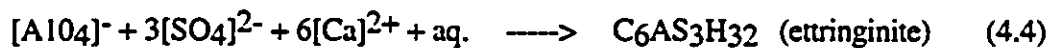
• *Tricalcium aluminate hydrate and the action of gypsum*

As discussed earlier, C₃A is very reactive, hence reaction of C₃A with water is quick and leads to immediate stiffening of the cement paste, known as flash set. In order to prevent rapid hardening of the cement paste, gypsum is usually added to the cement.

The stable form of the calcium aluminate hydrate existing in the hydrated cement paste is probably the cubic crystal C₃AH₆. The reaction can be written as follows.



To understand the hydration of C₃A with water, the implication of gypsum must be considered. The solubility of C₃A is depressed in the presence of hydroxyl, alkali and sulfate ions (alkalis and gypsum go into solution quickly). Depending on the concentration of the sulfate and aluminate ions in solution, a precipitating crystalline product is formed as either calcium aluminate trisulfate (ettringite) or calcium aluminate monosulfate.



Ettringite is the first hydrate to crystallize in normal Portland (which contains about 5% gypsum) cement in the first hour. The crystallization of ettringite contributes to the stiffening, setting and early strength of the paste. Then, after a certain time (after depletion of sulfates), ettringite becomes unstable and is converted to monosulfate.



The ferroaluminates produce compounds that have a structure similar to ettringite and monosulfate, but with variable compositions. It hydrates slower than C₃A.

4.1.1.5 Types of Portland cement

In order to satisfy specific requirements, several types of Portland cements are produced which possess different chemical compositions (Table 4-3). The Canadian Standards Association (CSA) defines five types of cement in the A5 Standard [Kosmatka et al., 1991]:

Table 4-3 Approximate chemical composition of different Portland cements [Kosmatka et al., 1991]

Type of Portland cement	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
10 Normal	50	24	11	8
20 Moderate	42	33	5	13
30 High Early Strength	60	13	9	8
40 Low Heat of Hydration	26	50	5	12
50 Sulfate Resistant	40	40	3.5	9

• CSA Type 10 - Normal Portland cement

CSA Type 10 cement is a general purpose cement. It is used when the cement or concrete is not exposed to particular conditions, such as sulfate attack, high heat of hydration, water, etc. It is used for the construction of sidewalks, floors, reinforced concrete members, etc.

• CSA Type 20 - Moderate Portland cement

CSA Type 20 cement is used when concrete or cement is exposed to moderate sulfate attack and for mass concrete structures such as dams. This cement produces less heat during hydration and will have a slower strength development than CSA Type 10 cement. Its use is particularly useful when concrete is poured at elevated temperatures. Table 4-4 illustrates that the quantity of C₂S in CSA Type 20 cement is higher than that in CSA Type 10 cement, which is responsible for the low heat of hydration, slower strength development, and low production of Ca(OH)₂ (responsible for the deterioration of the cement paste as a result of sulfate attack).

• **CSA Type 30 - High-early strength Portland cement**

CSA Type 30 cement acquires strength very rapidly, in one week or less. The same results can be obtained with rich mixtures of CSA Type 10 cement, however CSA Type 30 cement may provide it more satisfactorily and economically. It is used when the form work is to be removed as soon as possible or it may be used to control the curing period in cold weather concreting. Early strength development may be attributed to its fineness and the high quantity of C₃S, which is greater than that found in normal CSA Type 10 cement.

• **CSA Type 40 - Low-heat of hydration Portland cement**

CSA Type 40 cement is used when the amount of heat generated during hydration must be minimized. It is used in massive concrete structures, where the temperature rise during hydration is critical in order to avoid thermal cracking. The strength development is slower than that for CSA Type 10 cement. The characteristics of this cement are attributed to the low quantity of C₃S.

• **CSA Type 50 - Sulfate-resistant Portland cement**

CSA Type 50 cement is used when concrete or cement is exposed to high sulfate attack. Strength development is slower than CSA Type 10 cement. Low concentrations of C₃S are responsible for increased resistance to sulfates.

4.1.2 Microfine cements

Microfine cements were first introduced on the North American markets in 1984. These cements were first developed in Japan as a result of a ban on organic grouts following a toxicity incident in 1970 [Clarke, 1987]. A variety of microfine cements are now available; all vary greatly in chemical composition. Of all the microfine cements, two categories are identified, those made from Portland cement and those made with blast-furnace slag and activated by cement. But whatever their chemical composition, all microfine cements possess one similar characteristic, their fineness, which is defined by the surface area of the cement particle (specific surface). Specific surface has units of m²/g. High specific surface can be achieved by extensively grinding the cement particles.

Table 4-4 compares the specific surface of two Portland cements to those of four microfine cements.

Table 4-4 Specific surface of two Portland cements and four microfine cements [Langevin, 1993]

Type of cement	Blaine Fineness (m^2/kg)
CSA Type 10	225-320
CSA Type 30	325-420
Lanko 737	700
Microcems 650	650
Microcems 900	900
MC 500	800

The fineness of microfine cements permits the injection of very fine cracks, and it also increases their reactivity. From a rheological point of view, the viscosity of microfine cement grouts is considerably increased [Saleh et al., 1995].

4.1.3 Special cements

“Some cements exhibit special characteristics which are supposed to fill other engineering needs which are not covered by the previous cements. These special cements are as follows:

- **Alumina cements:** They have a high-alumina content because they are made by pulverizing calcium aluminate cement clinker. The raw materials used are limestone and bauxite. They have the following properties: high early strength (even at low temperatures), and superior resistance to sulfate attacks. The disadvantages are the loss of strength with time, especially at elevated temperatures.
- **Oil-well cements:** They are slow setting cements which are used in the petroleum industry when drilling wells.

- **Expansive cements:** These are hydraulic cements which expand during the process of hydration, setting and hardening (early hydration period). These cements are used when shrinkage cannot be tolerated in structural members (crack-free pavements, slabs, etc.).
- **White and colored cements:** White Portland cements are produced by the same process as the ordinary Portland cements except that they have a low content of iron and manganese (which gives the grey color). Colored cements are made by adding a chemically inert pigment to the Portland cement. These cements are used for aesthetic considerations.
- **Other cements:** Several other special cements exist such ultra high early strength cement, waterproofing cement, hydrophobic cement, antibacterial cement, barium cements, etc.” [Langevin, 1993]

4.2 Supplementary cementing materials (SCM)

Supplementary cementing materials are materials which, in combination with Portland cement, contribute to the various characteristics of hardened and freshly mixed water cement systems. Supplementary cementing materials contribute to the characteristics of the hardened system either by hydraulic cementitious action or by pozzalanic action; some materials possess both cementitious and pozzalanic properties. A material that possesses pozzalanic properties is one that, in finely divided form and in the presence of water, reacts chemically with calcium hydroxide released by the hydration of Portland cement to form compounds with cementitious properties [Kosmatka et al., 1991].

Supplementary cementing materials such as fly ash, condensed silica fume, slag, and rice-husk ash are all by-products of other industrial processes. Hence, their use in concrete, mortar, and grout is desirable from both an economical and energy conservation standpoint. Supplementary cementing materials can be used in a cement grout :

- as a replacement of cement to obtain reductions in the cement content,
- as an additive to improve grout properties, both in the fresh and in the hardened state, or
- to achieve both objectives, improved properties and reduced cement content.

In the past, it was common to have the SCMs blended with the cement at the cement plant. This had one major disadvantage, it did not permit the user to vary the dosage of the SCM to achieve the desired properties of the concrete or grout. The current trend, in concrete practice, is to mix the SCMs separately at the concrete ready mix plant.

4.2.1 Fly ash

Fly ash (FA) is a by-product of combustion of pulverized coal. During the combustion of pulverized coal, volatile matter and carbon is burned off, the remaining matter (clays, quartz, and feldspar) flies out with the flue gas stream, and is called fly ash. The ash can be removed from the gas by the help of electrostatic precipitators [Mehta, 1986]. Particle size distribution of the fly ash varies from 1 to 150 μm in diameter, and is essentially composed of solid spheres with a small number of hollow spheres [Berry and Malhotra, 1986]. In 1989, the world wide production of fly ash was approximately 400 million tons. The price of fly ash may vary from only a few dollars to \$90/ton [Malhotra, 1993].

Fly ashes may be divided into two categories:

- low calcium fly ash (class F) - fly ashes containing less than 10 percent CaO , product of the combustion of anthracite and bituminous coal.
- high calcium fly ash (class C) - fly ashes containing more than 10 percent CaO , product of the combustion of lignite and sub-bituminous coal.

The low-calcium fly ashes are essentially composed of aluminosilicate glasses. The aluminosilicates react with calcium hydroxide, released by the hydration of C_3S and C_2S , to form cementitious products (CSH). Generally, these ashes contain large proportions of crystalline minerals, such as quartz, mullite, and hematite or magnetite. These minerals are non-reactive, hence their large proportion reduces the reactivity of the ash [Mehta, 1986]. On the other hand, high calcium fly ashes contain calcium compounds in the form of C_3A , CS , and $\text{C}_4\text{A}_3\text{S}$ which are highly reactive. These compounds possess both cementitious and pozzolanic properties.

In 1948, the first practical application of fly ash was conducted by the US Bureau of Reclamation for the Hungry Horse Dam, U.S.A. Following the widespread use of fly ash

in concrete for massive hydroelectric structures in the early 1950's, an ASTM standard was issued [Berry and Malhotra, 1986].

4.2.2 Condensed silica fume

Condensed silica fume (CSF) is a by product of the reduction of pure quartz in the presence of coal in an electric arc furnace used to produce silicon metal and ferrosilicon alloys. In this process, quartz is transformed to silicon at very high temperatures (2000°C), and it also produces SiO vapors which oxidize and condense, at low temperature, to very fine particles of non-crystalline silica. The average particle diameter is 0.1 to $0.2\ \mu\text{m}$ [Regourd, 1986]. When compared to normal Portland cement, condensed silica fume particle size distribution are two orders of magnitude finer (Fig. 4-3). Its specific surface is around $20\ 000\ \text{m}^2/\text{kg}$ when determined by the nitrogen adsorption method [Mehta 1983], and its density is $2.2\ \text{g}/\text{cm}^3$. Although no distinction is made between the types and forms of CSF, the majority of CSF used contain levels of SiO_2 between 85 to 98 % [Sellevold and Nilsen, 1987].

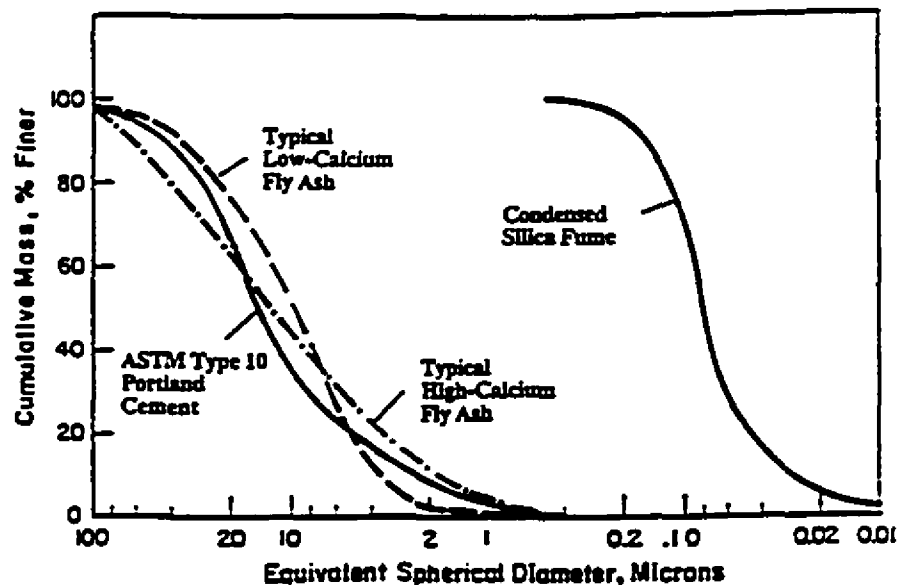


Fig. 4-3 Particle size distribution of Portland cement, fly ashes, and condensed silica fume [Mehta, 1986]

The first tests on CSF were conducted in Norway in the early 1950's. Since then, CSF has been used extensively in concrete by the Scandinavian countries. Estimated world wide production of CSF in 1982 was 780,000 tons [Malhotra, 1988]. Norway is the largest producer of CSF with a production of 140,000 tons in 1984, for a yearly consumption of 40,000 tons [Mehta, 1989]. Condensed silica fume was once considered to be industrial waste, the value of CSF is now estimated to be between \$300 and \$400 a ton.

Two Quebec cement producers actually buy and produce CSF cement. In the last ten years, Ciment Quebec has delivered over 100 000 m³ of concrete containing CSF [Durand, 1994].

Most studies show that CSF has an effect on the mechanical characteristics of concrete and a pronounced effect on its rheological properties. Condensed silica fume is sometimes used in concrete containing potentially reactive aggregates in order to decrease alkali-aggregate expansions [Durand, 1994].

Condensed silica fume reacts rapidly with the calcium hydroxide (CH) released during the hydration of C₃S and C₂S to form calcium silicate hydrate (CSH). Wu and Young [1984] studied the reaction products of CSF and C₃S systems. They discovered that three kinds of CSH are produced:

- that formed from the hydration of C₃S;
- that formed by the hydration between calcium hydroxide and CSF;
- that formed by the reaction of CSH and CSF;

The calcium to silica ratio (C/S) of the CSH produced by the reaction of CSF with calcium hydroxide is lower than the C/S ratio of CSH produced by the hydration of C₃S. The consequence of the low C/S ratio is the increased capacity to incorporate foreign ions, such as alkalis. Hence, CSF increases the resistance of concrete to chemicals and alkali-aggregate reactions [Sellevold and Nilsen, 1987].

4.2.3 Blast-furnace slag [Malhotra, 1993],[Mehta, 1986],[Kosmatka et al., 1991]

Blast-furnace slag is the by-product of the cast iron industry. The properties and structural form of slag are dependent on the rate of cooling. When the liquid slag is quenched in air, the chemical components is in the form of C₂AS-C₂MS₂ (crystalline melite). As a result of the slow cooling process, the slag particles generally resemble small pellets, and the slag is thus called pelletized slag. Pelletized slag is weakly cementitious when it is not pulverized. However, if the liquid slag is quenched rapidly, its chemical constituents assume a glassy state (non-crystalline form). As a result of the rapid cooling, the sand-sized particles are called granulated slag. Satisfactory cementitious properties can be achieved if the granulated slag is pulverized.

Strength contribution of granulated blast-furnace slag may be apparent in the early stages of strength development. However, reactivity of the slag depends greatly on the particle size, glass content, and structure of the glass. Some slags are cementitious to a

degree by themselves, whereas others need to be activated by the presence of cement or other activators.

Granulated slag may be divided into two types [Kosmatka et al., 1991]:

- Type G - ground granulated blast-furnace slag. This type in the absence of an activator, displays no cementitious properties.
- Type H - ground granulated blast-furnace slag displays some hydraulic activity with the absence of an activator.

Non-ferrous slag from copper, nickel, or lead are also potential cementing materials. The non-ferrous slag from the copper industry seems to be the most promising.

4.2.4 Rice-husk ash

Rice-husk ash (RHA) is a by-product of the rice paddy milling industry. The average chemical composition of rice-husk is 50 percent cellulose, 30 percent lignin, and 20 percent silica. The silica content in the husk is responsible for the pozzalanic activity of the ash. With properly controlled combustion, cellulose and lignin can be removed, leaving the silica behind. By incinerating the rice-husk at a temperature of 500° to 700 °C, for a very short time, an ash is produced with the following composition [Mehta, 1992]:

- silica content of 90 to 95 percent,
- alkalis ranging from 1 to 2 percent and
- unburnt carbon from 3 to 18 percent.

The result of incineration is an ash with a specific surface of 60 to 100 m²/g and a bulk density of 0.2 g/cm³ [Malhotra, 1993]. The ash of properly controlled combustion, is highly pozzalanic, and therefore an excellent supplementary cementing material. World production of rice paddy is approximately 500 million tons. Rice-husk represents a fifth of the rice paddy, and one ton of ash produces approximately 200 kg of ash. Hence, the potential annual production of RHA is 20 million tons. China is the biggest producer of rice paddy and the US is the smallest (Table 4-5) [Mehta, 1992].

Table 4-5 1990 World production of rice paddy and rice-husk ash (million metric tons) [Mehta, 1992]

Country	Rice Paddy	Rice Husk
China	180	36
India	110	22
Indonesia	45	9
Bangladesh	27	5.4
Thailand	20	4
Vietnam	18	3.6
Burma	13	2.6
Japan	13	2.6
Brazil	9	1.8
Korea	9	1.8
Philippines	9	1.8
USA	7	1.4
Others	40	8
Total	500	100

4.3 Chemical admixtures to modify the properties of grouts

An admixture can be defined as a material other than Portland cement, water, aggregates, and supplementary materials that is added to the concrete, mortar, or grout before or during mixing. Admixtures modify the rheological and physico-mechanical properties of cement systems. It is reported that chemical admixtures are added to 88 percent of the concrete placed in Canada, 85 percent in Australia, and 71 percent in the United States [Mehta, 1986]. A variety of chemical admixtures exist which are generally used in concrete to improve:

- workability,
- acceleration or retardation of setting time,
- enhancement of resistance to frost action,
- cohesion (reduced bleeding and segregation), and
- other properties.

Chemical admixtures can be divided into two categories; surface active chemicals (surfactants) and set-controlling chemicals. The surfactants begin to act immediately on the surface tension of water and by adsorbing on the surface of cement particles. On the other hand, set-controlling chemicals break up into their ionic constituents and influence the chemical reaction between cement and water.

4.3.1 Water-reducers

Surface-active chemicals are essentially composed of long-chain organic molecules, some of which are hydrophilic (water-attracting) and others that are hydrophobic (water repelling). The latter are used as water-reducers. By adding a water-reducer to a grout it is possible to increase the workability without increasing the water content, or to decrease the water content but maintain a given consistency. Water-reducers fall into five general classes [ACI 212.1R-81, 1982]:

- lignosulphonic acids and their salts (Fig. 4-4);
- modifications and derivatives of lignosulphonic acids and their salts;
- hydroxylated carboxylic acids and their salts (Fig. 4-5);
- modifications and derivatives of hydroxylated carboxylic acids and their salts;
- other compounds (melamine derivatives, naphthalene derivatives, etc.).

When a clinker is ground into fine particles, the links between molecules are broken, which results in cement surfaces which are electrically neutral. During hydration, the cement particles in suspension tend to flocculate in order to balance the electrical charges at the corners of the cement particle. These flocculates tend to unite into one large floc. The particles within the floc structure are non-uniformly distributed thus creating agglomerations [Shaughnessy and Clark, 1988]. Moreover, the surface tension in water is high, which contributes to the decreased dispersion of the cement-water system. Flocculation of cement particles may provoke the rapid sedimentation of the cement, and hence decreases the stability of the mixture. A diagram representing such a flocculated system is shown in Fig. 4-6.

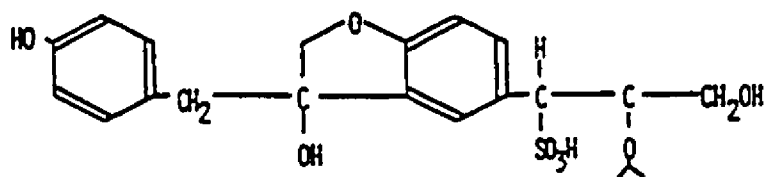


Fig. 4-4 Typical unit of a liginosulphonate molecule
[Ramachandran, 1984]

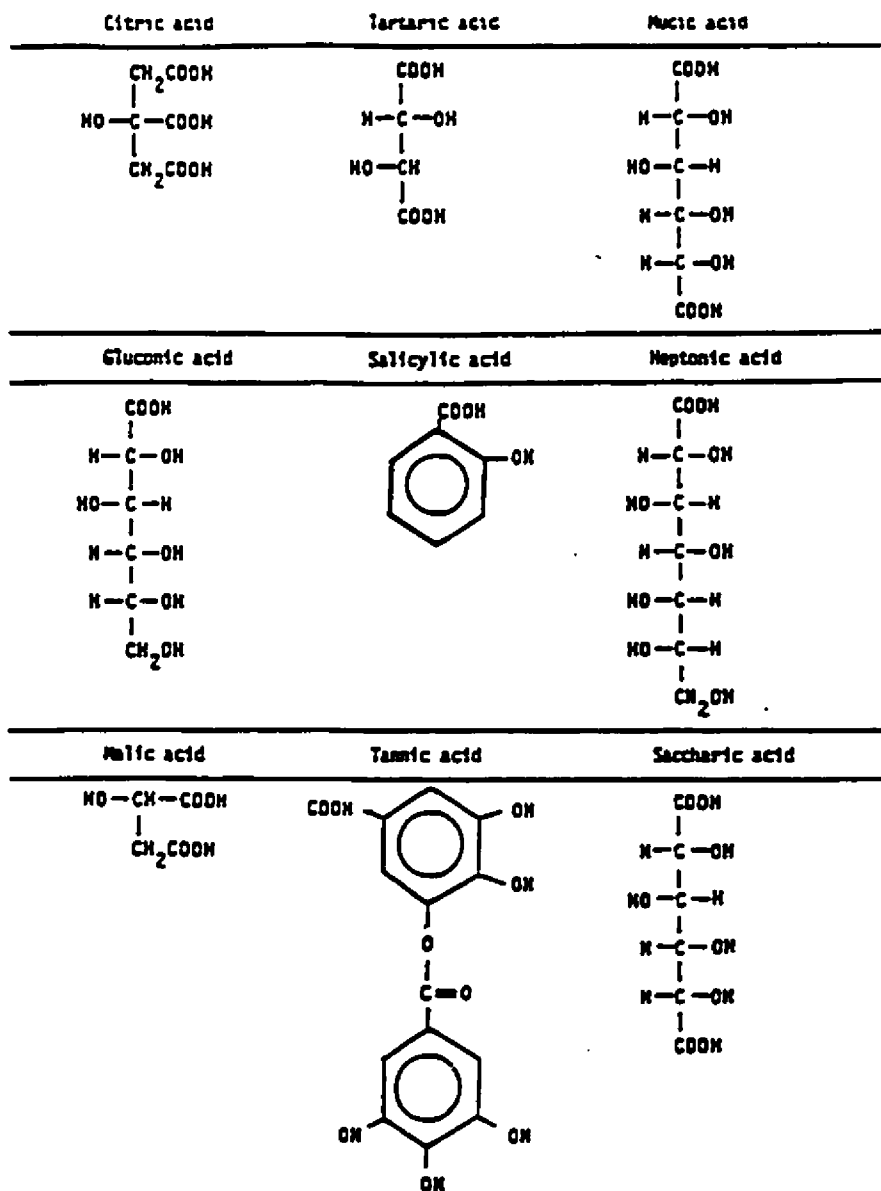


Fig. 4-5 Typical hydroxylated acids [Ramachandran, 1984]

Neville [1981] illustrates the mechanism of water reducers as follows: " These are substances which are concentrated at the interface between two immiscible phases and which alter the physico-chemical forces acting at this interface. The substances are adsorbed on the cement particles, giving them a negative charge which leads to the repulsion between the particles and results in stabilizing their dispersion (Fig. 4-6); air bubbles are also repelled and cannot attach to the cement particles. In addition, the charge causes the development around each particle of a sheath of oriented water molecules which prevent a close approach of the particles to one another. The particles have, therefore, a greater mobility, and water freed from the restraining influence of the flocculated system becomes available to lubricate the mixture so that the workability is increased."

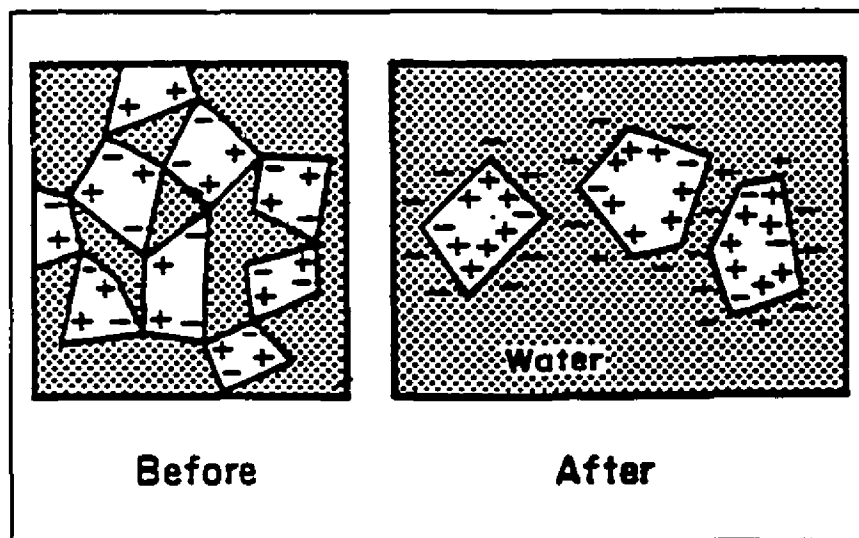


Fig. 4-6 Diagram representing floc formation of cement particles and dispersion of floc after the addition of a water-reducing agent [Mehta, 1986]

The addition of a water-reducer can effect the strength of the cement grout in two ways [Neville, 1981];

- dispersion of the cement particles expose a greater surface area of cement to hydration, which increases the rate of hydration at early ages.
- a more uniform distribution of the dispersed cement will contribute to the improved strength.

The reduction in the quantity of mixing water is anywhere between 5 to 15 percent. The period of effectiveness of a water reducer must be considered, since it does not last indefinitely. Once hydration of the cement has started, the products of hydration will engulf the small quantity of surfactants present in the system [Mehta, 1986]. Water-reducers may increase the air content depending on their type. A decrease in the compressive strength may be observed if too much air is present.

Increasing the quantity of water-reducer to increase fluidity for a given water/cement ratio is not recommended, since it may result in undesirable effects on the setting, air content, bleeding, and hardening characteristics [Ramachandran, 1984]. Finally, to avoid any undesirable effects, it is recommended to undertake a laboratory investigation before using an admixture, or a combination of two or more admixtures.

4.3.2 Superplasticizers

Superplasticizers (SP), or high range water-reducers, permit the reduction of water in concrete by 25 to 30 percent [ACI 212.1R-81, 1982]. Three types of superplasticizers are commercially available; they are sulphonated melamine formaldehyde condensates, sulphonated naphthalene formaldehyde condensates and calcium lignosulphonate or modified sodium, which consists of long-chain, high-molecular weight anionic molecules with a large number of polar groups of the hydrocarbon chain (Fig. 4-7). These admixtures adsorb themselves on the surface of cement particles, thus making the cement particles mutually repulsive as a result of the anionic nature of the superplasticizer, or, they decrease the surface tension of water, and hence increase the fluidity of the system [Neville, 1981].

In cement grouts, a superplasticizer may be used in one of two ways:

- to increase the fluidity of the grout for a given water/cement ratio and
- to maintain a given consistency for a reduction in the water content.

The prescribed dosage of superplasticizer is usually less than 1% of dry superplasticizer by mass of cement. Additional amounts of superplasticizer may effect the setting time and stability of the grout. When used to increase fluidity, the concrete usually loses its additional workability within 30 to 60 minutes, following preparation of the mixture [ACI 212.1R-81, 1982].

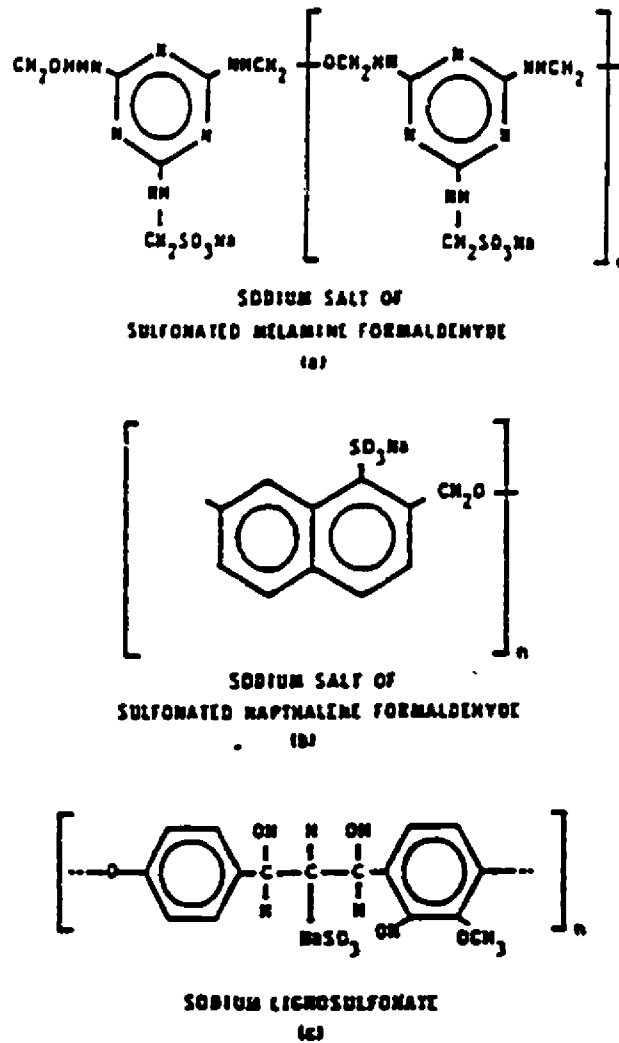


Fig. 4-7 Typical superplasticizer molecules [Ramachandran, 1984]

4.3.3 Anti-washout agents

Anti-washout agents (AWA) serve to increase the stability (reduce bleeding) and cohesion of the mixture. When injecting into rock where a hydraulic gradient is present, the increased cohesion of the AWA prevents the water from washing away the grout. Under static conditions, this type of admixture impedes the migration of water and thus reduces the bleeding. Under dynamic conditions, AWA prevents the separation of fine particles from the grout [Saleh et al., 1995]. In other words, AWA increases the cohesiveness of the mixture. Three types of AWA exist [Kosmatka et al., 1991]:

- cellulose derivatives;
- polysaccharides from micro-organisms, natural soluble gum;
- acrylic polymers.

Anti-washout agents alter the properties of the grout according to three mechanisms:

- adsorption: molecules of the AWA adsorb water while expanding;
- association: particles develop bonds with other particles as a result of Van der Waals forces and hydrogen bonds;
- overlapping: polymers overlap when they are in large concentrations.

Anti-washout agents do not only affect the stability and cohesion properties of the grout, but in doing so increase the viscosity and the yield stress in the process. To offset the effect of these admixtures on the flow properties, a superplasticizer can be added to the mixture. It is recommended to undertake some laboratory testing, since problems of compatibility between certain SPs and AWAs may exist.

CHAPTER 5

GROUTS

Many factors influence the rheological and mechanical properties of grouts, such as water/cement ratio, the use of admixtures and the addition of supplementary cementing materials. This chapter reviews the effects of water/cement ratio, fly ash, superplasticizer and anti washout agent on the fresh and hardened properties of cement grouts.

5.1 Effect of water/cement ratio

Based on the calculations by Hansen [taken from Mnif, 1992], 1.28 cm^3 of water is needed to hydrate 1 cm^3 of cement. The free water which is not used for the hydration of cement, is used to provide fluidity to the paste. A portion of this water migrates to the surface as bleed water. The amount of bleeding is a function of the cement fineness, particle size, type of admixture added, and the water/cement ratio. The amount of bleeding increases as the water content of the grout is increased. At high water/cement ratios, the amount of bleeding is excessive and it may have detrimental effects on the physico-mechanical properties of the grout. In fact, increasing the water content decreases its compressive strength.

As mentioned earlier, the free water increases the fluidity of the grout, therefore the fluidity is proportional to the water/cement ratio. However, at fairly high water/cement ratios, the fluidity becomes almost invariable [Saleh, et al., 1993]. Ritchie [1965] observed that as the water content of the mixture was reduced, the initial yield stress value increased as did the plastic viscosity (Fig. 5-1).

Water content has a pronounced effect on the setting time. Langevin [1993] observed that the setting time increases with an increase in the water content. As the water content increases the cement concentration decreases, the excess water slows the hydration reaction thus taking longer for the grout to set.

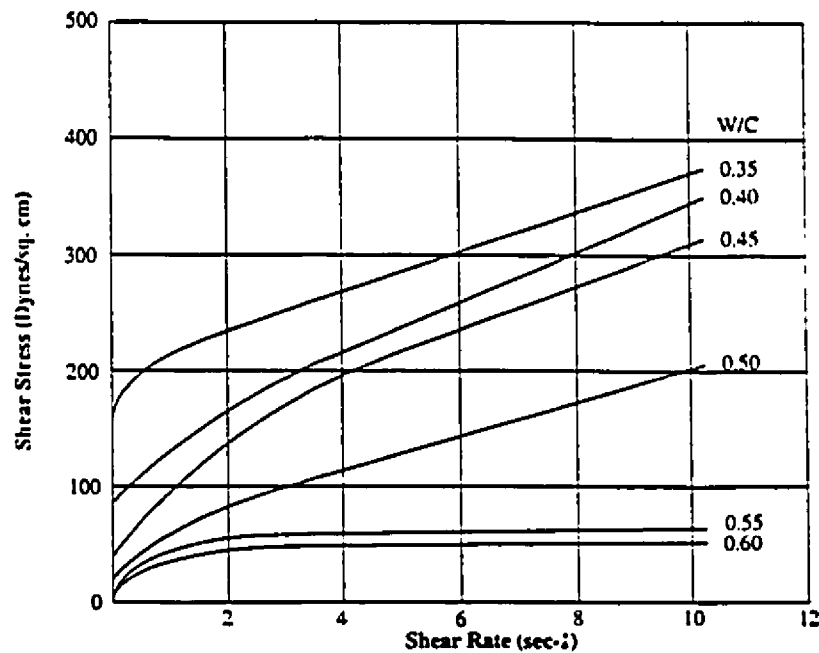


Fig. 5-1 Shear stress vs. shear rate curves of cement pastes of different water/cement ratios [Ritchie, 1965]

5.2 Effect of admixtures on the properties of grouts

Admixtures may be used to modify the characteristics of a grout or as in the case of supplementary cementing material (SCM), they may be used to reduce the amount of cement. The addition of an SCM may also be able to achieve both objectives, i.e. a reduction in the amount of cement and modification of the grout characteristics. The following sections describe the effect of SCM and chemical admixtures on fresh and hardened properties of grouts.

5.2.1 Fly ash

- *Effect on fresh properties*

“The small size and the essentially spherical form of the low-calcium fly ash particles have been credited with influencing the rheological properties of cement pastes; this causes a reduction in the amount of water required for a given degree of workability from that required for an equivalent paste without fly ash.” [Davis et al., 1987]

Particle size and shape greatly influence the rheological properties of grouts. As discussed above, the spherical particles of fly ash have a lubricating effect on the cement particles, hence they increase the fluidity of the grout.

Banfill [1994] studied the effects of fly ash on cement mortars; he observed that fly ash reduces both plastic viscosity (η_p) and the yield stress (τ_0). Reduction of the plastic viscosity and the yield stress increased with an increase in the fly ash content (Fig. 5-2). Banfill also observed that substitution by volume had a more pronounced effect than substitution by mass. This may be attributed to the difference in particle density of cement and fly ash.

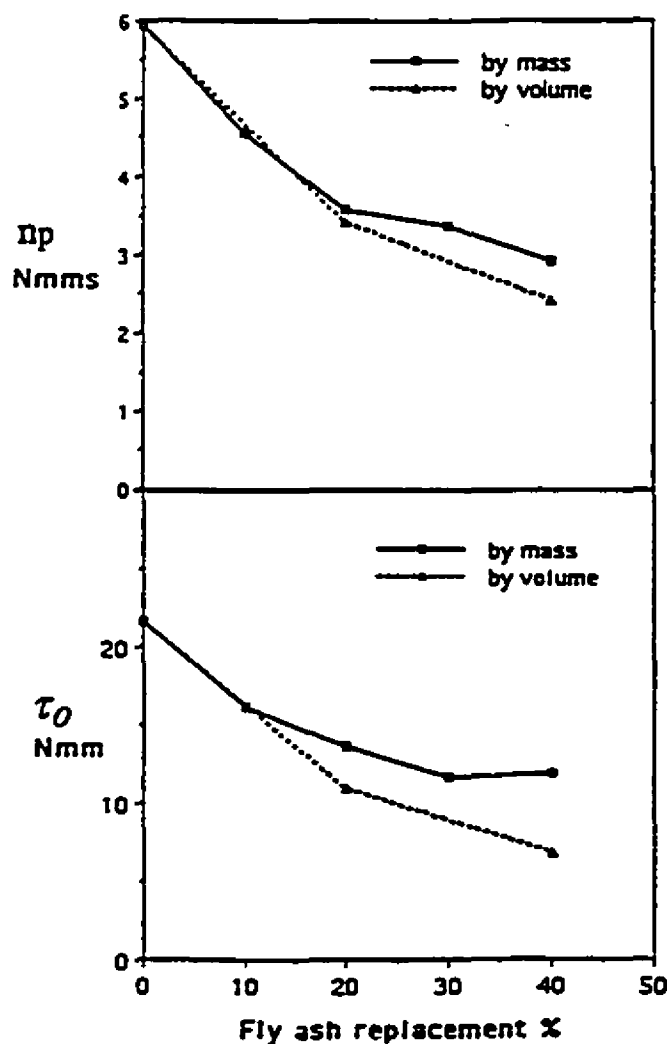


Fig. 5-2 Effect of fly ash on the yield stress (τ_0) and the plastic viscosity (η_p) of cement mortars [Banfill, 1994]

The carbon content of the fly ash may be responsible for influencing the rheological properties of the grout. Brink and Halsted [1956] observed that some fly ashes reduced the water content required for test mortars, while others (generally of high carbon content) increased the water requirement above that of the control mortar.

Generally, it has been seen that the particle size and shape, and chemical composition have an effect on the rheological properties. It is important to note that the physical and chemical characteristics of fly ash vary from one thermal plant to another. Hence, it is important to test these materials with cement before undertaking any injection work.

The setting time of cement pastes including fly ash as a partial replacement for cement, show a prolonged setting time from those of an equivalent paste with no fly ash. More pronounced effects have been observed with paste made with low calcium fly ash [Davis et al., 1987]. Dilution of the cement concentration and low reactivity of fly ash are responsible for the prolonged setting time.

Stability is controlled principally by the particle size distributions and the specific surfaces of the cement and the mineral admixture. The particle and chemical characteristics vary from one thermal plant to another, which suggests that the stability of fly ash grouts depends on the type of fly ash. A study of the bleeding characteristics of fly ash concrete revealed that out of 11 ashes, only 2 increased bleeding [Carette and Malhotra, 1984].

• *Effect on hardened properties*

Many variables influence the strength development of fly ash cement systems, the most important ones being the chemical composition, particle size, and temperature. Very little research has been done on fly ash grouts. However, the characteristics of fly ash concrete may be used to illustrate the behavior of cement fly ash systems.

The strength development of concrete seems to be only slightly affected by the presence of high calcium fly ash (class C) [Berry and Malhotra, 1986]. Class C fly ashes are cementitious, even in the absence of Portland cement, and pozzalanic. On the other hand, class F fly ashes are only pozzalanic, which means that they possess cementitious properties only in the presence of calcium hydroxide released by the hydration of cement. Concrete made with class F fly ash generally have a lower initial strength than that made with class C fly ash [Kosmatka et al., 1991].

5.2.2 Superplasticizers

- *Effect on fresh properties*

Superplasticizers and water-reducers significantly affect the properties of fresh cement grouts. They achieve this by deflocculating the agglomerations of cement particles, and in the process they liberate the water which is not used for hydration. The released water increases the fluidity and may also increase bleeding. Mirza et al. [1993] studied the effects of two types of superplasticizers on the viscosity and stability of cement grouts made with CSA Type 10 Portland cement. It was observed that both naphthalene and melamine based superplasticizers decrease the viscosity of the cement grouts with increasing percentage of superplasticizer. Superplasticizer was added as a percentage of the total mass of cement. As indicated in Fig. 5-3, the addition of 1 % SP (melamine based) considerably increases the fluidity of the grout. However, addition of more SP does not cause a further increase in the fluidity. For water/cement ratios greater than one, the addition of SP does not affect the viscosity. A study by Domone and Thurairatnam [1988] indicates that the effect of SP is more pronounced on the yield stress than on the plastic viscosity (Fig. 5-4).

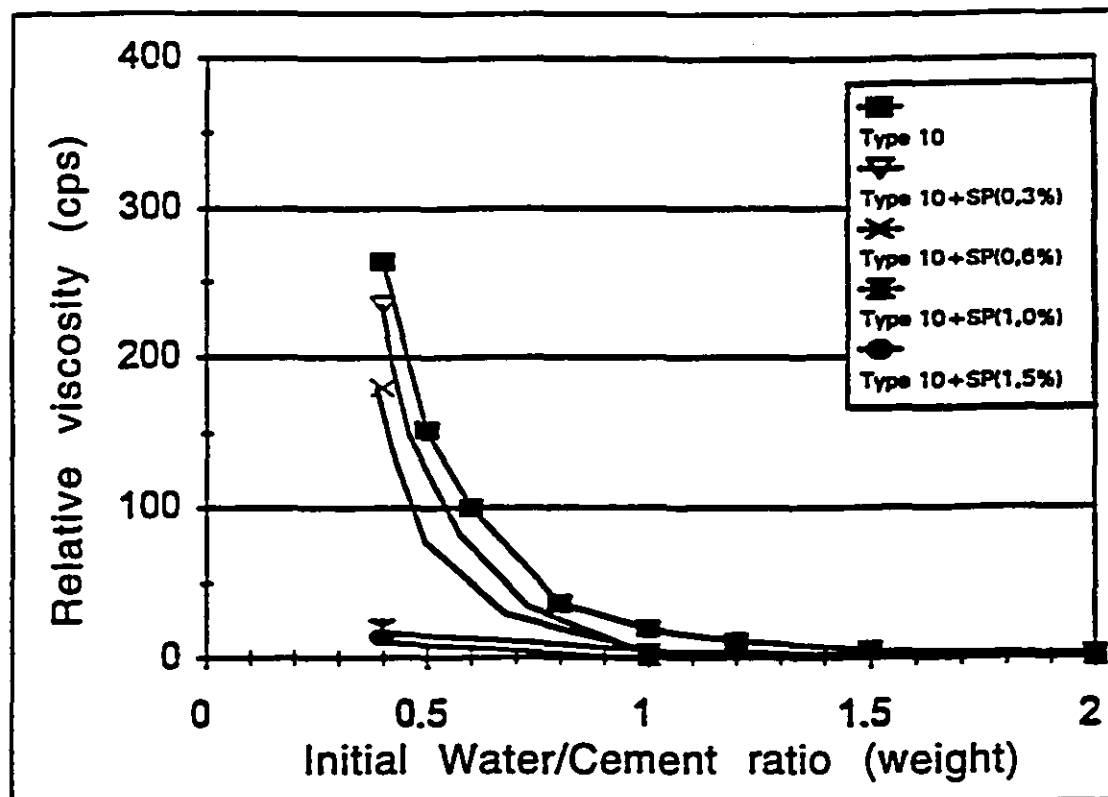


Fig. 5-3 Viscosity of CSA Type 10 cement grout with melamine based SP at different initial W/C ratios [Larjevin, 1993]

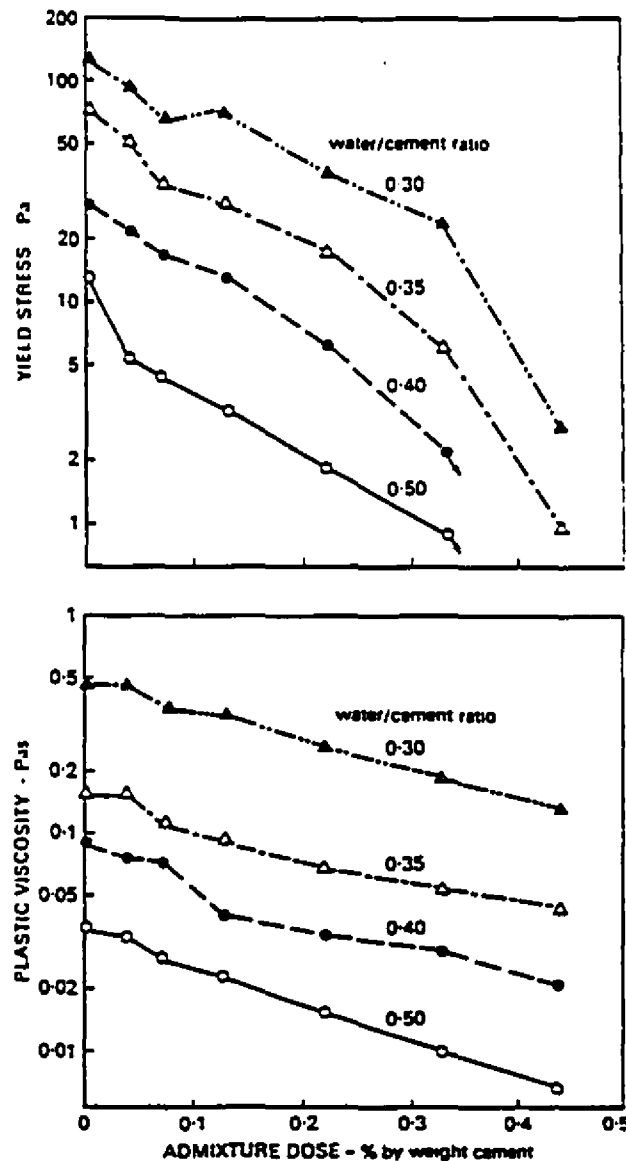


Fig. 5-4 Effect of melamine formaldehyde superplasticizer on yield stress and plastic viscosity of Portland cement pastes [Domone and Thurairatnam, 1988]

The influence of SP on grout stability is dependent on the type of SP and on the chemical and particle characteristics of the cement. At low water/cement ratios the effect of superplasticizer on the stability of grouts is insignificant. Mirza et al. [1993] observed that a very small percentage of SP ($\leq 0.2\%$ by mass of cement) improves the stability of CSA Type 10 cement grouts with a high water/cement ratio (between one and two). However, it can be seen in Fig. 5-5 that at water/cement ratios of one and two, the volume in suspension, for a dosage of SP > 0.2 percent, is significantly reduced. On the other hand, the stability of CSA Type 30 cement is increased with the addition of one percent SP [Mnif, 1992].

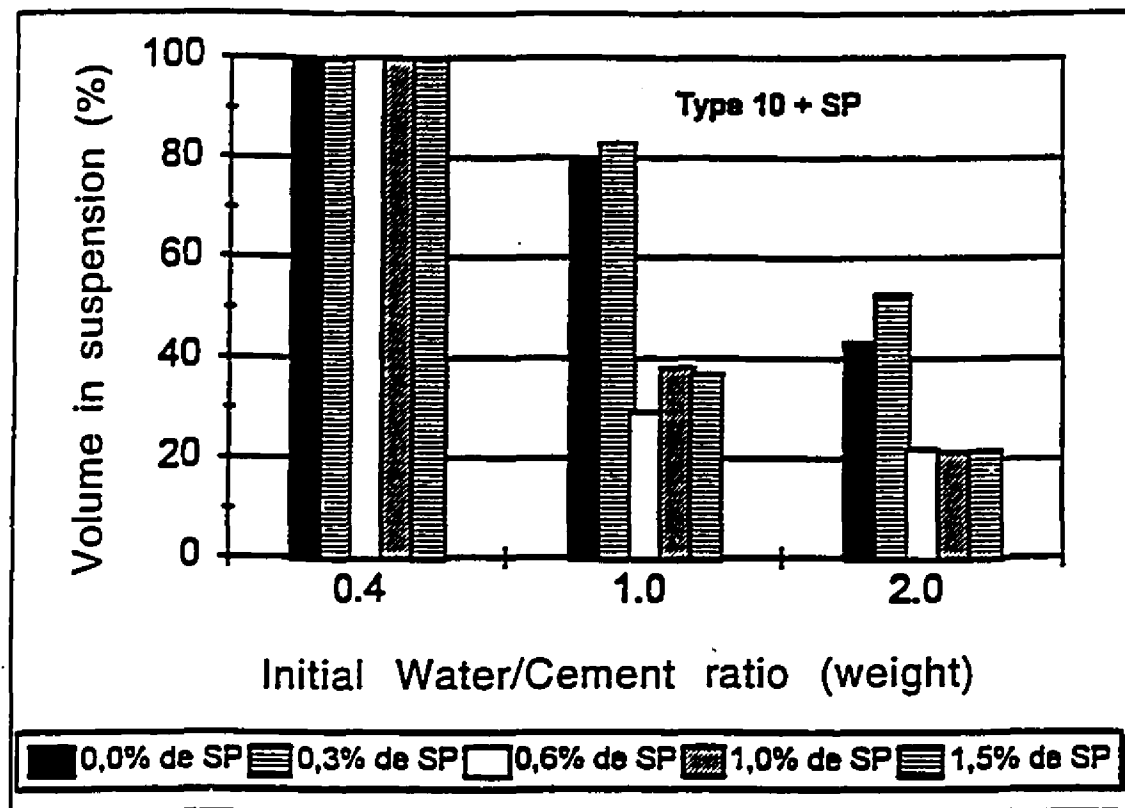


Fig. 5-5 Stability of CSA Type 10 cement grout with melamine based SP at different initial water/cement ratios [Langevin, 1993]

Addition of SP considerably increases the initial and final setting time of CSA Type 10 cement grout. Saleh et al. [1993] observed that the setting increased from 7h for CSA Type 10 cement grout without SP to 12h for a grout with a lower water/cement ratio and SP. Prolonged setting time may increase the risk of fresh grout freezing when these products are used in cold climates. Moreover, this phenomenon may result in significant grout leaching if water flow is present.

5.2.3 Anti-washout agents

The use of AWA has a dramatic effect on the stability and the viscosity of CSA Type 10 Portland cement grouts. Langevin [1993] examined the effects of an AWA (Welan Gum - polysaccharide) and one concrete pumping agent (Sika 100 SC - AWA combined with SP) on the stability and viscosity of CSA Type 10 cement grouts. Both of them increase the viscosity of the grout at all water/cement ratios. The AWA caused a greater increase in the viscosity than the pumping agent. Bleeding decreases as a result of the addition of AWA for grouts with any water/cement ratio. This admixture is particularly effective at high water/cement ratios, where the amount of bleeding in normal cement grouts is very high (Fig. 5-6).

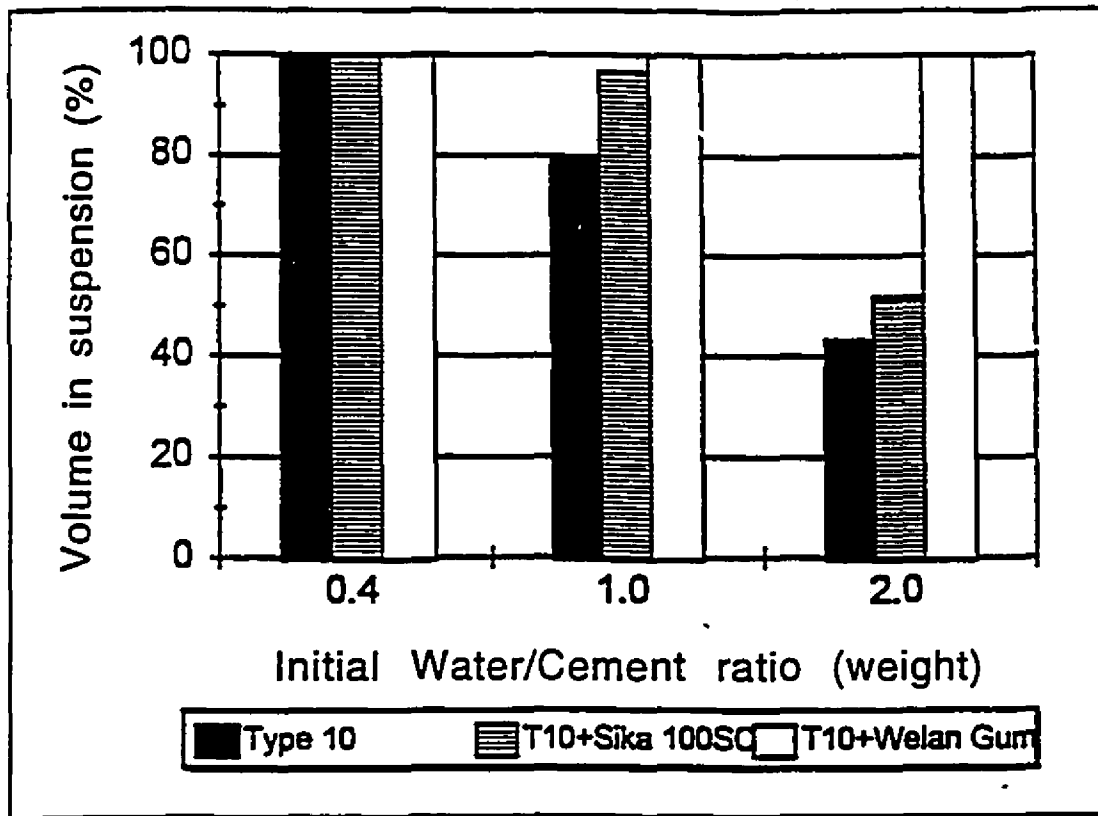


Fig. 5-6 Effect of AWA (Welan Gum) and pumping agent (Sika 100SC) on the stability of CSA Type 10 cement grouts [Langevin, 1993]

CHAPTER 6

EXPERIMENTAL PROGRAM

The rheological and mechanical properties of the cement grouts incorporating Canadian fly ashes were investigated in this study. Rheological studies were also conducted on cement-fly ash grouts with variable dosages of SP and AWA.

6.1 Materials used

All grouts were made with CSA Type 10 hydraulic Portland cement from Lafarge Canada. All cement bags and fly ashes were homogenized separately and then stored in 25 liter air-tight containers to ensure uniformity of the materials throughout the testing program. The fly ashes used in this study came from three sources:

- Sundance (Alberta) - class C fly ash (C1);
- Thunder Bay (Ontario) - class C fly ash (C2);
- Point Tupper (Nova Scotia) - class F fly ash (F).

A melamine formaldehyde-based SP (Melment) from Euclid was used to increase the fluidity. The percentage of the solids by weight of SP in solution is 42 percent. The AWA used to reduce bleeding was a polysaccharide based (Welan Gum) in the form of a powder.

6.2 Particle size distribution

The particle size distribution of all cementing materials (cement and SCM) is a very important factor that controls the rheological and mechanical behavior of grouts. This characteristic is very important from the point of view of injection. In fact, if the particles of the cement or SCM are too large, they may cause the agglomeration of the solid particles, and hence obstruct complete grouting of the crack.

Particle size distribution of the cement and fly ashes is determined by the sedigraph method. The procedure consists in putting a 100g of cement or SCM into a test tube filled with water. The tube is then shaken vigorously to enable the solid particles to remain in suspension. The particle size distribution is then determined with the aid of an X-ray

machine. Table 6-1 and Fig. 6-1 show the particle size distribution for CSA Type 10 Portland cement and the three fly ashes.

Table 6-1 Particle size distribution of CSA Type 10 cement and the three fly ashes

Particle Size (microns)	Percent Finer			
	PC	C1	C2	F
100	98	88	88	90
90	98	88	87	90
80	97	87	86	89
70	97	87	86	88
60	95	86	85	87
50	93	84	82	85
40	87	82	78	82
30	76	78	73	77
20	59	70	63	68
10	37	49	42	45
9	34	45	39	42
8	31	42	35	38
7	27	37	31	32
6	24	32	26	26
5	20	26	21	19
4	17	20	15	11
3	12	12	8	3
2	7	5	2	0
1	2	0	0	0

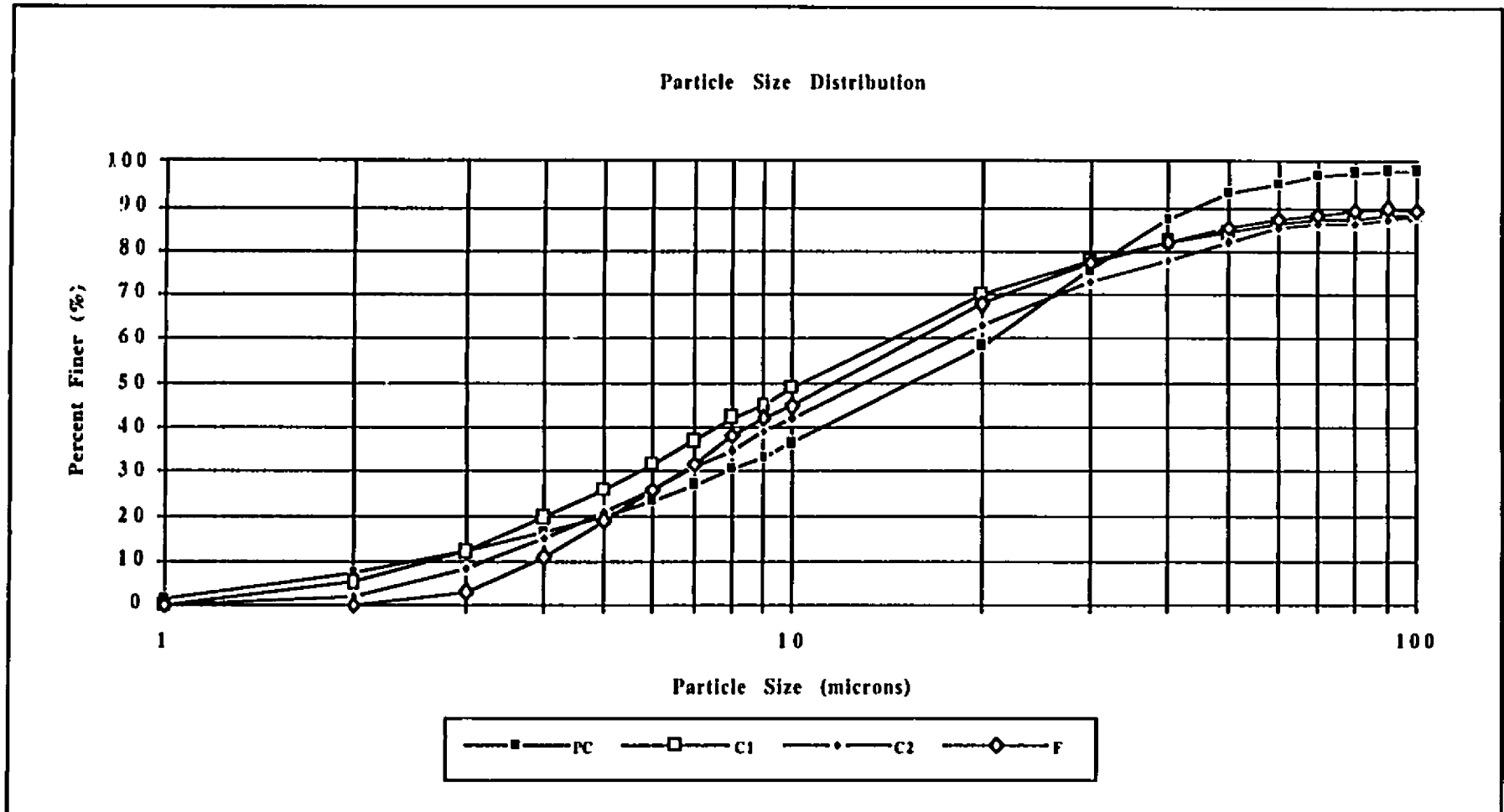


Fig. 6-1 Particle size distribution of CSA Type 10 cement and the three fly ashes

Analysis of the particle size distribution can be summarized as follows:

- The average particle size for the CSA Type 10 cement is 16 microns (value at D₅₀); less than three percent of the particles are larger than 90 microns.
- The average particle size for C1, F, and C2 fly ash is respectively 11, 12, and 13 microns.
- The maximum particle size for the C1, C2, and F fly is respectively 239, 220, and 205 microns.
- The fly ashes can be arranged in ascending order of fineness as follows: C1, F, and C2.
- Twelve percent of the particles, for each of the three fly ashes, are larger than 100 microns.
- Eighty three percent of the particles of C1 and F fly ash are smaller than 45 microns. As for C2 fly ash, 80 percent of the particles are smaller than 45 microns.
- The three fly ashes investigated have a slightly finer gradation than the CSA Type 10 cement, which holds for 60 percent of the particle size distribution curve. However, for particles sizes smaller than 3 microns and larger than 30 microns, CSA Type 10 cement has a larger percentage of finer material than any of the fly ashes analyzed.
- The particle size distribution of the fly ashes is generally similar to that of the CSA Type 10 cement analyzed.

Fly ash by itself possesses very little cementitious properties, however, it can react in the presence of moisture with calcium hydroxide released by cement during hydration to form cementitious products. The degree of reactivity is greater in fine ash than in coarse ash [Houlsby, 1990]. The Australian Standard AS3582 suggests that 75 percent of the particles of fly ash should be finer than 45 microns. Of the three different fly ashes used, all contain 80 percent or more particles smaller than 45 microns.

6.3 Chemical analysis

The chemical composition of a fly ash enables identifying its classification. As discussed in section 4.2, the ashes which have a CaO content lower than 10 % are considered to be class F, and are produced from the combustion of bituminous coal. On the other hand, class C ashes have a CaO content of 15 to 35 percent, and are the result of the combustion of subbituminous coal. The chemical analysis also gives an insight into the

rheological and mechanical behavior of cement-fly ash grouts. The chemical analysis of the three ashes and the CSA Type 10 cement was carried out in the laboratories of Lafarge Canada. Table 6-2 shows the breakdown of the chemical constituents expresses as a percentage of the total weight.

Table 6-2 Chemical composition (% weight) of the cement and fly ashes

CHEMICAL ANALYSIS (% weight)				
	Portland Cement	Fly Ashes		
		C1	C2	F
SiO ₂	21.36	53.3	41.99	40.71
Al ₂ O ₃	3.98	23.63	21.44	17.93
TiO ₂	0.19	0.71	1.05	0.85
P ₂ O ₅	0.21	0.12	0.58	0.17
Fe ₂ O ₃	3.15	4.4	4.45	29.86
CaO	62.41	12.45	15.81	2.8
SrO	0.27	0.22	0.6	0.15
MgO	2.57	1.15	3.18	1.09
Na ₂ O	0.2	3.03	7.03	0.73
K ₂ O	0.8	0.42	0.36	1.56
SO ₃	3.43	0.2	1.79	1.27
LOI	1.72	0.71	0.75	1.95
Total	100.29	100.34	99.03	99.07
Moisture	0.11	0.06	0.02	0.06
LOI at 750°C	0.55	0.37	0.16	0.25
Total Carbon	0.64	0.63	0.36	0.48
Available Na ₂ O	1.54	1.14	1.32	1.36
Available K ₂ O	0.23	0.29	0.28	0.26

The findings can be summarized as follows:

- The calcium oxide content (CaO) for the C1 and C2 ashes are 12.45 and 15.81 percent, respectively. Therefore, both of these ashes are considered as high calcium ashes and are designated class C. On the other hand, the CaO content of F fly ash is 2.8 percent, making this a low-calcium fly ash (class F).
- The iron content (Fe_2O_3) of the F ash (30 %) is considerably higher than that of the other two ashes (4.5 % for both C1 and C2 ashes).
- The CaO content of CSA Type 10 cement (62 %) is roughly five times more than that of the class C ashes and 22 times more than that of the class F ash. The iron content of CSA Type 10 cement is similar to that of the class C ashes.
- All fly ashes have a higher content of silica and alumina than that of CSA Type 10 cement. Cement contains 20 percent SiO_2 compared with the fly ashes which contain roughly 40 percent or more. The Al_2O_3 content is 4 percent in CSA Type 10 cement as opposed to roughly 20 percent in the ashes.

The chemical analysis concludes that fly ashes are essentially composed of calcium oxide, silica, and alumina, which is similar to cement. However, the percentage of these constituents are quite different from those of CSA Type 10 cement. CSA Type 10 cement has a very high calcium oxide content, while fly ash has a high silica content. To understand the effect of these chemical components on the cementitious and pozzalanic properties of the fly ash, one must also examine its crystalline structure. However, such a study is beyond the scope of this research program.

6.4 Specimen preparation

Grout samples were prepared for eight water/solids ratios (0.4, 0.45, 0.5, 0.55, 0.65, 0.8, 1.0 and 1.3). For every water/solid ratio, 4 different mixtures were prepared:

- 0 % fly ash (pure cement grout);
- 60 % fly ash (by weight of cementitious materials) for three different fly ashes (C1, C2 and F).

At low water/solids ratios (0.4, 0.45 and 0.5), 24 additional mixtures were prepared to verify the effects of SP (1.5 % SP by weight solids) and the combined effects of SP and AWA (1.5 % SP and 0.04 % AWA by weight of water) on the rheological properties (flow

time and bleeding) of fresh grouts. At high water/solids ratios (0.8, 1.0 and 1.3 where excessive bleeding is encountered), 12 additional mixtures were prepared to verify the combined effect of SP and AWA (1.0 % SP and 0.15 % AWA) on the rheological properties of fresh grouts. Hence, the total number of mixtures prepared was 68.

All ingredients were maintained at 20 °C prior to mixing. The grouts were mixed with a "Jiffy" type mixer for 5 minutes with an angular speed of 2300 rpm. Mixing and testing were performed at 20 °C. Immediately after the preparation of the mixture, rheological test were conducted on the fresh grouts (flow cone, bleeding and setting time). Then, specimens for compression, bond strength, modulus of elasticity and drying shrinkage were prepared.

6.5 Tests on fresh grouts

This section presents the tests performed to examine the properties of freshly mixed grouts. The rheological properties (flow time, viscosity, and cohesion) as well as grout stability (bleeding) and setting time are investigated. The results of the tests on freshly mixed grouts are expressed as a function of water/(cement + SCM) or water/solids (W/S) ratio.

6.5.1 Marsh flow cone

The flow cone test is a measure of the apparent viscosity of the grout. It measures the combined effects of viscosity and the cohesion of the grout. The cone test is easy and quick to perform, which makes it a practical field test. The test consists in measuring the time required for a certain volume of grout to flow through a funnel. The flow time is a function of the friction forces produced by the wall roughness and the dimensions of the cone.

A variety of flow cones exist [Houlsby, 1990]:

- ASTM cone (ASTM Standard C 939).
- Marsh cone.
- Prepakt cone and
- Mecasol cone.

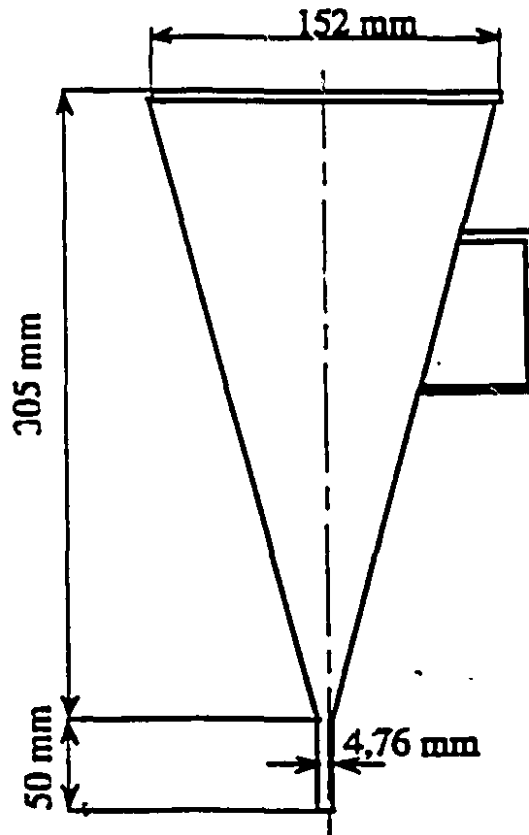


Fig. 6-2 Marsh flow cone [adapted from Houlsby, 1990]

The size varies from one cone to another, as does the flow time for an equivalent volume of water. Some cones work best with concrete and thick mortars, while other cones work best with thin grouts. Unfortunately, there is a lack of standardization, hence it is important to specify the type of cone used.

The cone used to measure the flow time is a Marsh cone made of extruded plastic. The interior wall of the cone is smooth and the outlet pipe diameter is 4.76 mm (Fig. 6-2). Time required for a liter of water to flow through the cone is 32 seconds. A sample volume of 1000 mL is used from every grout mixture produced, and the flow time is determined twice to ensure reliability of the results.

6.5.2 Bleeding (stability)

When grouting cracks in rock or concrete structures, adequate mobility is required to provide sufficient grout penetration. High water/cement ratios are often used to provide mobility and workability. In most cases, the water needed for mobility exceeds that required for the hydration of the cement paste. Cement particles settle to the bottom of the

crack and the excess water accumulates on the top. The sedimented particles cause the effective water/cement ratio to be smaller than the initial water/cement ratio at the bottom of the crack, and increases the effective water/cement ratio in the upper portion of the crack (Fig. 6-3), thus leaving the crack partially filled or improperly filled and hence prone to water permeability, which is unfavorable if the grout is used to form an impermeable curtain. Moreover, cracks that are partially filled provide a weak point in a foundation which is subjected to high stresses.

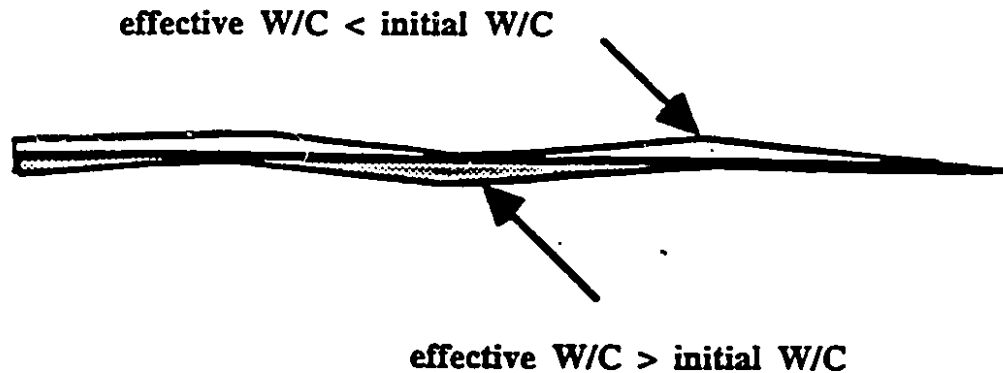


Fig. 6-3 Sedimentation of cement particles in an injected crack
[Saleh et al., 1995]

The lifting force of the rock mass is inversely proportional to the grout cohesion. Grouts which are unstable allow water to separate from the mixture. As a result, the separated water, which has very little cohesion, may cause large lifting forces. Therefore, the risk of upheaving is much smaller in stable mixtures [Lombardi, 1985]

The amount of bleed water and time required for all particles to sediment is a function of the particle size and shape, chemical composition of the cement, water and admixtures. Sedimentation of particles is a result of gravity and electrostatic forces.

This test was performed in accordance with ASTM Standard C 940-89 'Standard Test Method for Expansion and Bleeding of Freshly Mixed Grouts'. The test consists in placing an 800 mL sample of fresh grout into a 1000 mL graduated cylinder (Fig. 6-4). The volume of bleed water, on the surface of the settled grout, is recorded every 15 minutes for the first hour, and every 60 minutes thereafter. This is done until two equal readings are obtained. Final bleeding is calculated using the following equation:

$$\text{Final bleeding, \%} = \frac{V_w}{V_i} \times 100$$

V_i = volume of sample at beginning of test (mL)

V_w = volume of decanted bleed water (mL)

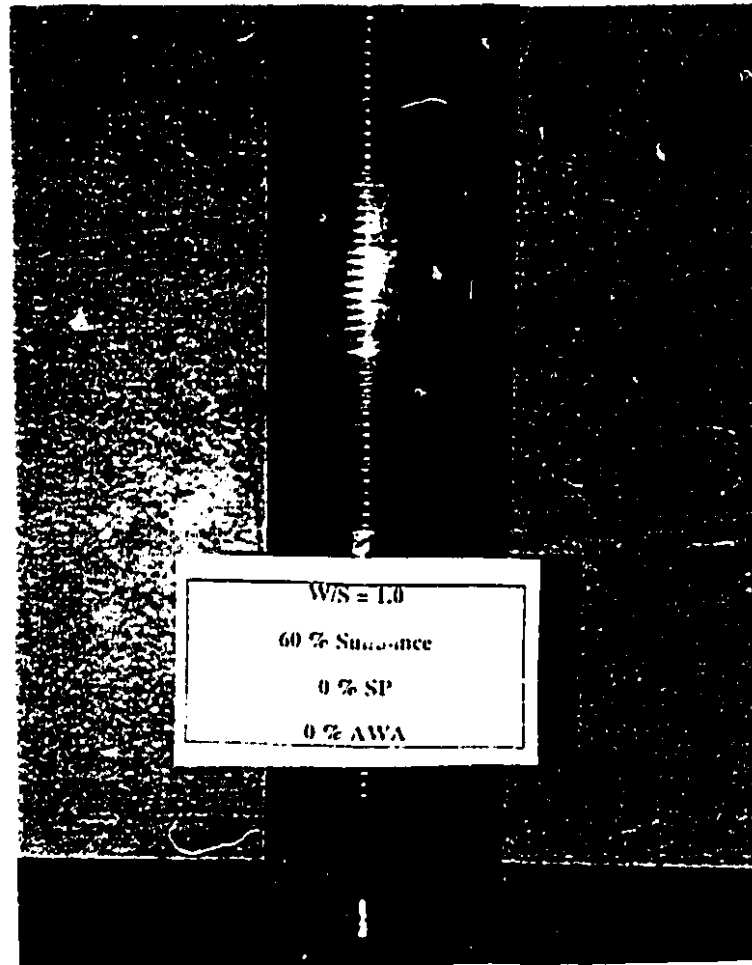


Fig. 6-4 Bleeding test

A grout is considered stable when bleeding is less than 5 % after 120 minutes [Lombardi, 1985]. In other words, a grout is unstable if the quantity of water on the surface exceeds 5 %.

As a result of bleeding, the effective water/cement ratio of the sedimented mass is smaller. In order to compare the mechanical properties of the hardened grouts, it is important to determine the effective water/cement ratio. The effective water/cement ratio may be calculated using the following equation :

$$\text{Effective W / C} = (100 - \% \text{bleed}) \times \text{initial W / C}$$

6.5.3 Setting Time

The initial setting time is defined as the time from mixing until the cement paste is no longer workable. Consequently, the final setting time is defined as the time when the paste is completely rigid. The initial set represents the time at which the paste or grout can no longer be handled, and the final set represents the time after which strength begins to develop. Knowledge of the initial and final setting time is essential for any grouting operation. For example, a grout with a fast initial setting time may cause the injection tubes to clog. On the other hand, a grout with a long setting time may increase the risk of freezing or leaching by water.

The hydration process is initiated as soon as the water comes in contact with the cement. The rate of this reaction is a function of:

- cement components: (C₃A and C₃S are respectively responsible for setting and strength development),
- cement fineness,
- water/cement ratio,
- presence and type of mineral admixture,
- presence of chemical admixture and
- temperature.

The test used to determine the initial and final setting time of the grout mixtures was performed according to the ASTM Standard C 191-82 "Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle." The apparatus consists of a needle attached to a 300g weight (Fig. 6-5). Setting time is determined by measuring the penetration of a needle in a grout sample. Initial setting time is obtained when the needle penetration is less than or equal to 25 mm. On the other hand, the final setting time is reached when the needle does not visibly sink into the paste.

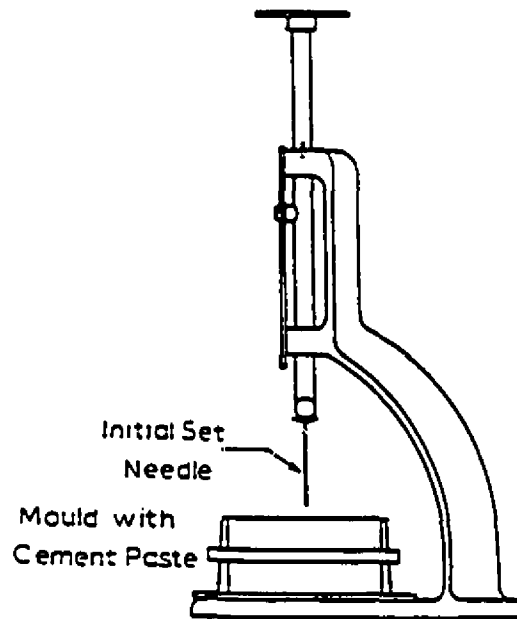


Fig. 6-5 Vicat apparatus [Neville, 1981]

6.6 Tests on hardened grouts

This section describes the tests performed on hardened grout. The specimens were demolded after 24 hours and then moist cured until the time of testing. The following mechanical tests were performed:

- compressive strength,
- bond strength,
- modulus of elasticity and Poisson's ratio and
- drying shrinkage.

6.6.1 Compressive strength

The compressive strength (f_c') determines the maximum axial compressive force that a specimen can withstand. Knowledge of the compressive strength of the grout is useful when the grout is to be used to solidify rock foundations. A grout with a low compressive strength, injected in a rock foundation which is subjected to high stresses, will be the weak point in the foundation. When the grout is used to form a grout curtain to reduce flow seepage through a foundation, high compressive strength characteristics might not be as important as grout permeability.

The compressive strength of the prepared grouts was determined using 51 mm diameter by 102 mm high cylinders (Nx size cylinders). Cylinders were moist-cured after removal from their molds until the time of testing. The test was performed following the ASTM Standard C 39-86 'Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens'. The compressive strength of grouts was measured after 7, 14, 28 and 91 days of moist curing.

6.6.2 Bond strength

Irrespective of where the grout is injected, whether it is in a concrete structure or in rock, once the grout has set it must bond completely with the surrounding material to effectively strengthen the structure. For example, if the grout is injected to solidify a rock mass, there is a difference in the thermal coefficients of expansion between the two materials, therefore it is important to determine the bonding properties of the grout.

The test was performed according to the ASTM Standard C 882, "Bond Strength of Epoxy Resin Systems Used With Concrete". For each grout mixture, three 75 mm diameter by 150 mm high concrete cylinders (35 MPa) were cast. These cylinders were sawn diagonally into two equal sections at an angle of 60 ° to the horizontal. The cut surfaces were then sandblasted, cleaned and kept wet. One half of the cylinder was placed in a 75 * 150 mm cylindrical mold. The procedure of the test differs somewhat from the ASTM Standard, and instead of applying a thickness of grout between the diagonal surfaces of the concrete cylinder, the remaining portion of the cylinder is filled with grout (Fig. 6-6). Once the remaining portion of the cylinder is filled, the specimen is wet cured until the time of testing.

The specimens are tested in uniaxial compression after 14 and 28 days of curing. Close examination of the specimen indicates whether a shear failure occurs at the concrete grout interface (bond strength), or whether the specimen fails in compression (bond strength > compression failure).

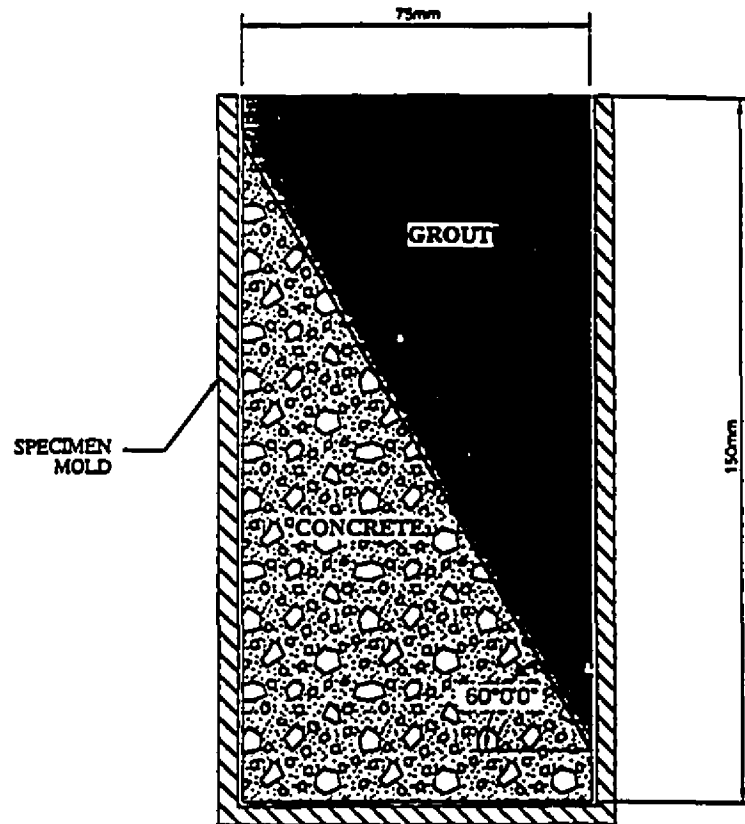


Fig. 6-6 Bond strength specimen

6.6.3 Modulus of elasticity and Poisson's ratio

The modulus of elasticity (E) is defined as the coefficient of proportionality between the applied stress and strain. Poisson's ratio (ν) is the coefficient of proportionality between the lateral and axial strains.

This test was performed according to the ASTM Standard D 3148-86, "Elastic Moduli of Intact Rock Core Specimens in Uniaxial Compression". For this test, three 51 mm diameter by 102 mm high cylinders were fabricated from every mixture. Axial and lateral strains were determined from the data obtained using LVDT's (Linear Variable Differential Transformer). Three LVDT's were used to measure the axial deformations, sensors were spaced equally around the circumference of the cylinder. Finally, three more LVDT's, located at mid-height of the cylinder, were used to measure the lateral deformations (Fig. 6-7). Specimens were loaded in uniaxial compression up to 40 percent of the specimen ultimate capacity. Measurements of axial and lateral deformations were taken for each load increment.

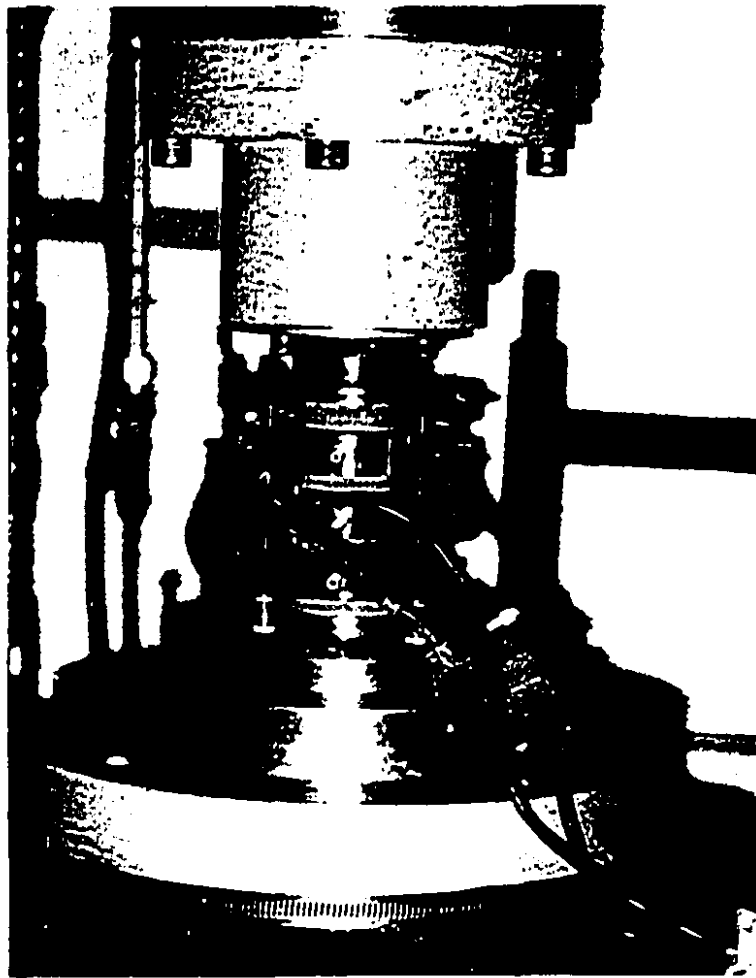


Fig. 6-7 Apparatus used to measure axial and lateral deformations

The values of E were obtained by calculating the slope of the average axial strains against the applied stress. Similarly, the Poisson's ratio was calculated from the slope of the plot of lateral strains against axial strains.

6.6.4 Drying-shrinkage

“ After a cement-based grout has been injected into a crack to strengthen the structure, bleeding may appear and proceed at different rates until the grout sets. Even after the grout has hardened over a period of several days or possibly years, the cement particles continue to hydrate if moisture is present in the crack. Throughout that time, the hardened grout may either shrink or expand, depending on the characteristics of the cement (chemical composition) and the ambient humidity.

Shrinkage or expansion of hardened grout can cause serious problems. If shrinkage occurs because of lack of moisture, the crack may reopen, reducing the water tightness of the structure" [Langevin, 1993].

The standard test method used was the ASTM C 596-82, "Standard Test Method for Drying Shrinkage of Mortar Containing Portland Cement ". The linear length change of 25.4 mm by 25.4 mm by 254 mm bars is determined at 3 days, and every seven days for the first 28 days, and monthly thereafter for a period totaling 91 days. The bars were left to dry at 22 °C and a relative humidity of 30 %.

CHAPTER 7

RESULTS AND DISCUSSION

7.1 Introduction

Three Canadian fly ashes from three different sources and normal CSA Type 10 Portland cement grouts were used for the study, which involved determining three rheological properties (flow time, bleeding and setting time) and five mechanical properties (compressive strength, bond strength, modulus of elasticity, Poisson's ratio, and drying-shrinkage). The chemical analysis and particle size distribution for the cement, two class C fly ashes (C1 and C2) and one class F fly ash (F) were presented in Chapter 6. In an attempt to reduce the flow time of low water/solids ratio grouts and increase the stability of high water/solids ratio grouts with or without fly ash, the effects of a melamine formaldehyde-based SP (dosage by weight of cement) as well as the combined effects of this SP and a polysaccharide based AWA (dosage by weight of water) were investigated.

It should be mentioned that all of the results presented here are for 60% cement replacement by fly ash. This value was chosen after conducting the flow test (essential for grouting jobs) with cement grouts containing 50, 60, 70 and 75% of fly ash which showed that grouts containing more than 60% fly ash have no profound effect on the flow characteristics.

7.2 Fresh grouts

The following properties of fresh grouts are reported based on the initial water/solids ratio.

7.2.1 Marsh flow cone

Flow time was determined for eight water/solids ratios ranging from 0.4 to 1.3 (0.4, 0.45, 0.5, 0.55, 0.65, 0.8, 1.0 and 1.3) by weight, for grouts with and without fly ash. Measurements were conducted at an ambient temperature of 20 °C. The effect of SP was investigated with the objective of increasing the relatively low fluidity encountered with low water content grouts ($W/S = 0.4$ to 0.55). One and a half percent (1.5 %) SP by mass

of dry cement was added to the mixtures with and without fly ash for water/solids ratios ranging from 0.4 to 0.5. Preliminary testing demonstrated that the addition of SP increased the quantity of bleed water. Furthermore, the effect of SP on grout instability was accentuated when the grout contained fly ash. This phenomenon was observed with the three fly ashes. In an effort to stabilize the cement fly ash and SP mixture, 0.04 percent AWA by mass of water was added. Anti-washout agent dosage was determined from preliminary testing, as the minimum amount of AWA required to increase the grout stability.

As discussed in section 5.1, extensive bleeding is usually observed for CSA Type 10 cement grouts with a high water content. Anti washout agents may be added to increase grout stability, however, these agents generally increase the flow time. For example, the flow time of a CSA Type 10 cement and C1 fly ash grout containing 0.15 % AWA with a water/solids ratio of 1.0 is 71 seconds. Similarly, the flow time of a CSA Type 10 cement and C1 fly ash grout with a water/solids ratio of 0.5 is 67 seconds. Hence, SPs should be used in combination with an AWA to offset the effects on fluidity. Preliminary tests were conducted on grouts with and without C1 fly ash at a water/solids ratio of 1.0 to determine the appropriate dosage of SP and AWA. These tests demonstrated that 0.15 percent of AWA agent was necessary to increase the stability (bleeding less than 5 % after two hours), and the critical dosage of SP was 1.0 percent, i.e. higher dosage of SP showed no decrease in the flow time. Hence, flow characteristics were examined for grouts containing 1.0 percent SP and 0.15 percent AWA for water/solids ratios of 0.8, 1.0, and 1.3.

The results shown in Fig. 7-1 to 7-5 lead to the following conclusions (a tabulated form of the results is presented in Appendix A):

- The flow time of a 1000 mL of water at 20 °C is 32 seconds.
- The flow time is inversely proportional to an increase in the water content, i.e. higher the water/solids ratio, lower is the flow time (Fig. 7-1). For a CSA Type 10 cement grout, the flow time varies from as high as 126 seconds, for a water/cement ratio of 0.45, to as low as 36 seconds for a water/cement ratio of 1.3.
- For water/solids ratios larger than 0.65, the flow time is invariable to an increase in the water content. Flow time is approximately 36 seconds, which is slightly higher than that of water at 20°C (Fig. 7-1).

- No flow was observed through the cone with a CSA Type 10 cement grout for a water/cement ratio of 0.4, since the grout was too thick to pass through the opening of the Marsh flow cone.
- The addition of fly ash reduces the flow time for grouts with water/solids ratios below 0.65 with respect to the reference grouts of equivalent water/solids ratio (Fig. 7-1). For example, at a water solid ratio of 0.45, the reduction in flow time for grouts made with C1, F, and C2 fly ashes is respectively, 27, 40, and 56 percent, and at a water/solids ratio greater than 0.65, the effect of fly ash on the flow characteristics is negligible.
- Of the three fly ashes investigated, C2 ash shows the greatest increase in fluidity followed by F and C1 ashes (Fig. 7-1). This phenomenon holds true for low water/solids ratios, i.e. between 0.4 and 0.65, after which there are no significant differences in the flow characteristics of grouts with or without fly ash.
- A significant reduction in the flow time was observed with the addition of 1.5 percent SP for the CSA Type 10 cement-based grouts with and without fly ash. Superplasticizer was most effective in grouts that contained a high cement content. As the water content was increased, the effect of SP on the fluidity decreased and for water/solids ratios greater than or equal to 0.55, the effect of SP on flow time was negligible (Fig. 7-2 to 7-5). This is in agreement with the observations made by Mirza et al. [1993], which states that for a water/cement ratios above 1.0, no further decrease in the relative viscosity is observed for grouts containing SP.
- The addition of SP enabled the CSA Type 10 cement grout with a water/solids ratio of 0.4 to flow through the cone. Flow time was 71 seconds (Table A-1 Appendix A).
- For water/solids ratios ranging from 0.4 to 0.55, the cement-based grouts containing SP which demonstrated the largest increase in fluidity were those containing C2 fly ash followed by F and C1 ashes.

- Anti-washout agent, used to increase the stability of low water/solids ratio grouts containing fly ash and SP, caused an increase in the flow time. Flow time values, at water/solids ratios of 0.45 and 0.5, for C1 and F grouts containing SP and AWA were similar to the flow time obtained for cement fly ash grouts containing no SP and no AWA (Fig. 7-3 and 7-5). Hence, no benefit is gained in using SP for grout ratios of 0.45 and 0.5, since SP destabilises cement fly ash suspensions and the use of AWA causes an increase in the flow time. On the other hand, a reduction of 33 percent and 24 percent in the flow time is observed with grouts containing 1.5 percent SP and 0.04 percent AWA at a water/solids ratio of 0.4 (C1 and F ash mixtures only). Similar grouts, at low water/solids ratios (0.4 to 0.5), made with C2 fly ash resulted in flow times which were superior to those containing no SP and no AWA (Fig. 7-4). As Table A-1 (Appendix A) shows, increasing the percentage of SP did not result in any further reduction in the flow time.
- Very stable grouts can be produced at high water/solids ratios by the addition of AWAs. However, the use of AWAs dramatically decreases the fluidity. For example, the flow time of a mixture containing CSA Type 10 + C2 fly ash + 1.0 % SP + 0.15 % AWA, with a water/solids ratio of 1.0, is 50 seconds. Similarly the flow time of CSA Type 10 + C2 fly ash + 1.5 % SP, with a water/solids ratio of 0.45, is also 50 seconds.
- Close examination of Table A-1 (Appendix A) reveals that cement grouts with C2 fly ash produces the best flow characteristics at all water/solids ratios with or without SP and AWA.

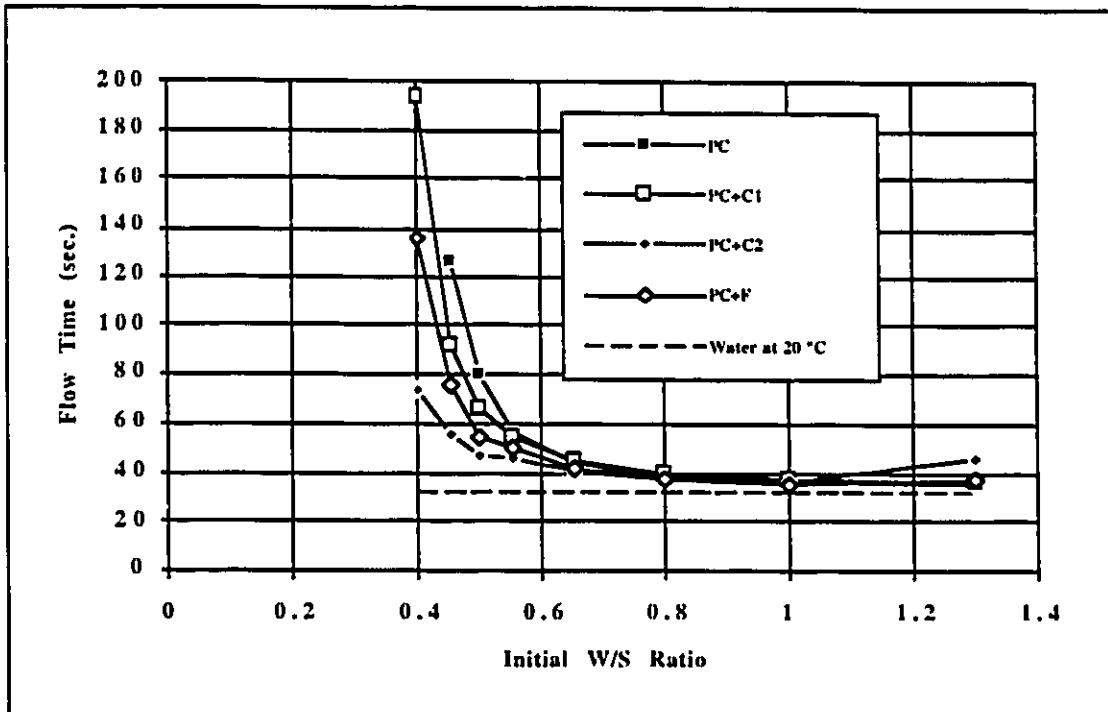


Fig. 7-1 Flow time of CSA Type 10 cement-based grouts with and without fly ash

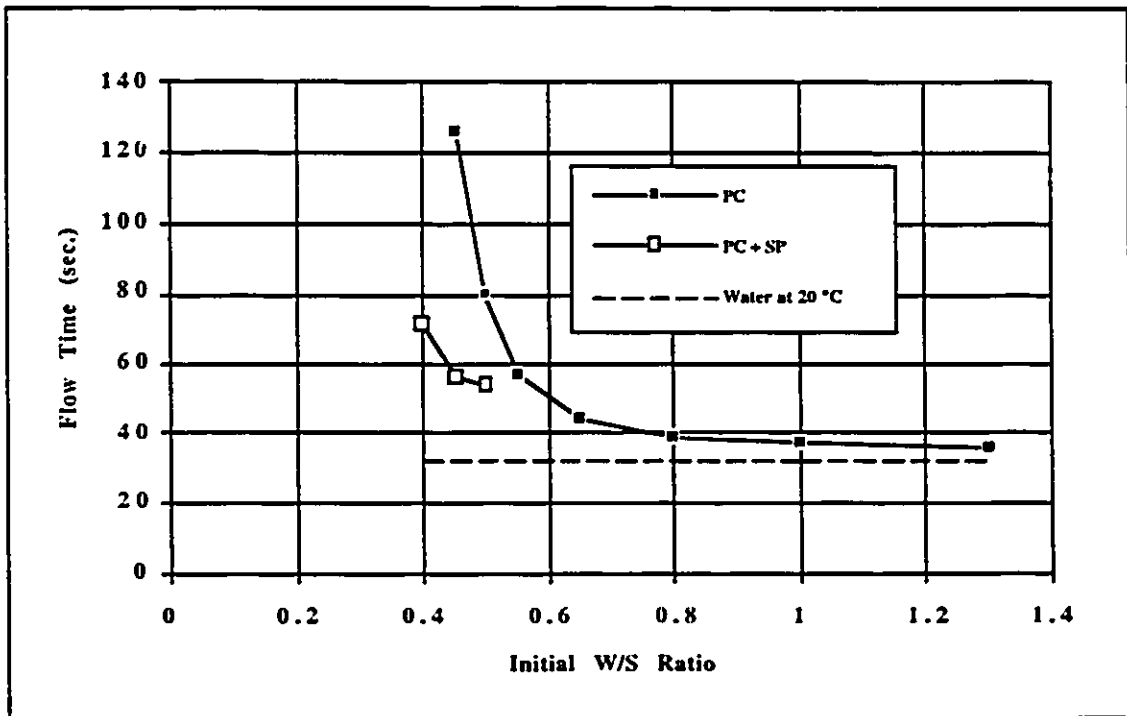


Fig. 7-2 Effect of SP on flow time of CSA Type 10 cement grouts

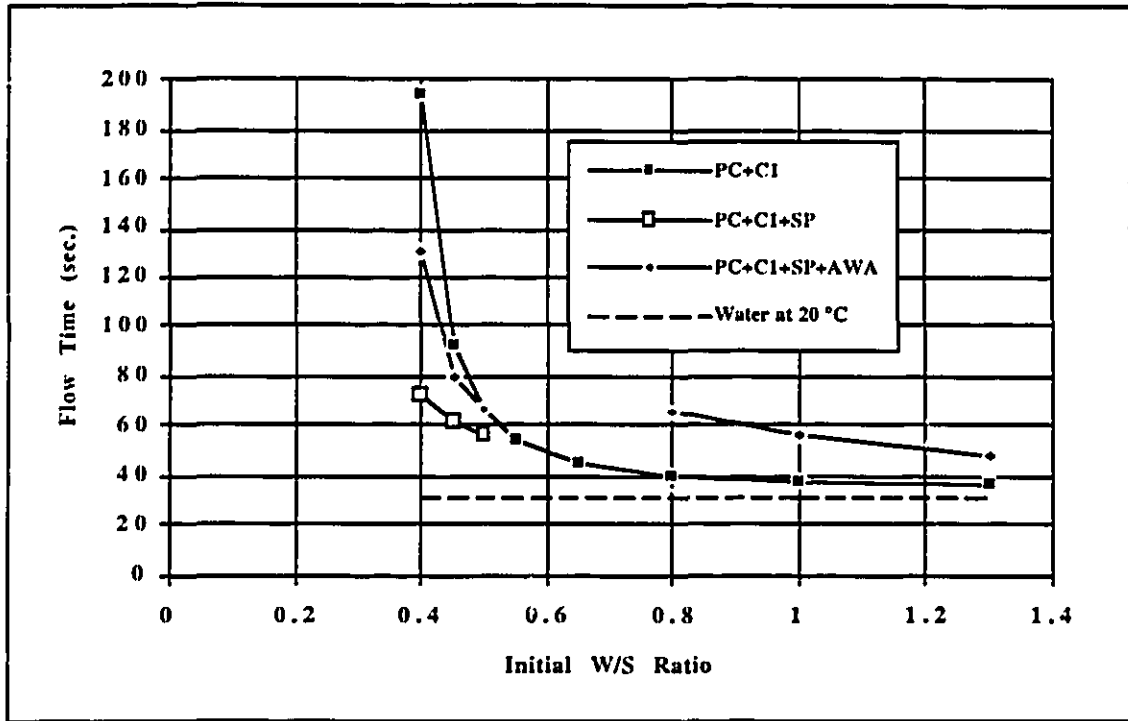


Fig. 7-3 Effect of SP and AWA on flow time of CSA Type 10 cement and C1 fly ash grouts

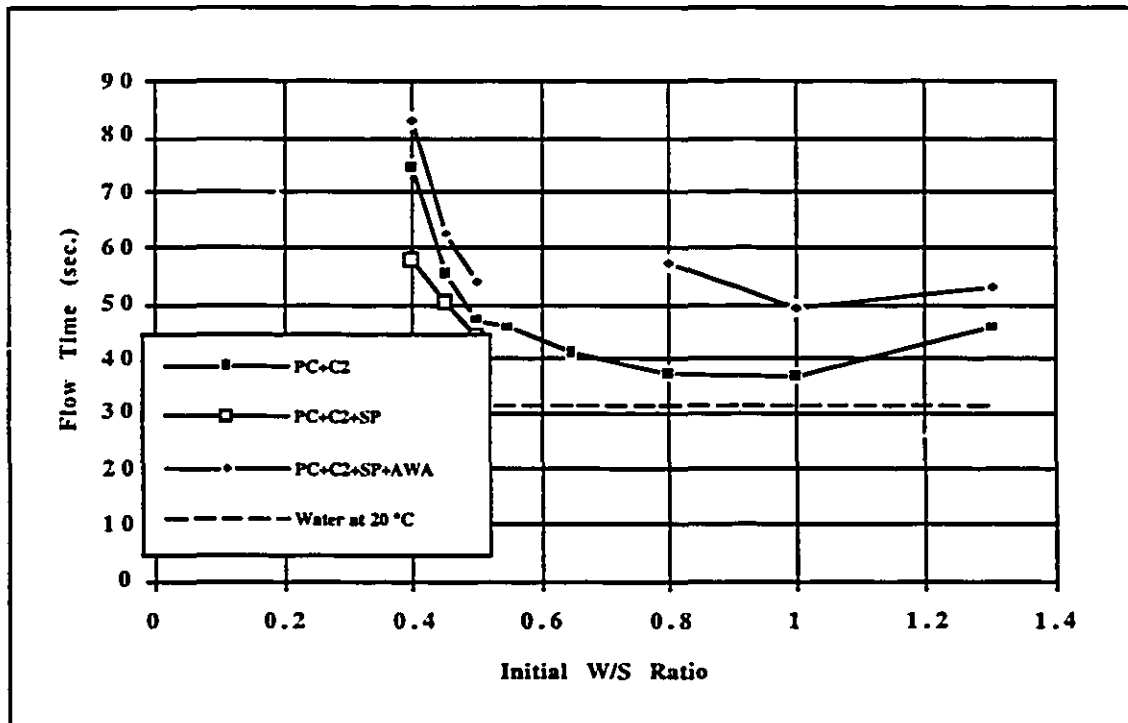


Fig. 7-4 Effect of SP and AWA on flow time of CSA Type 10 cement and C2 fly ash grouts

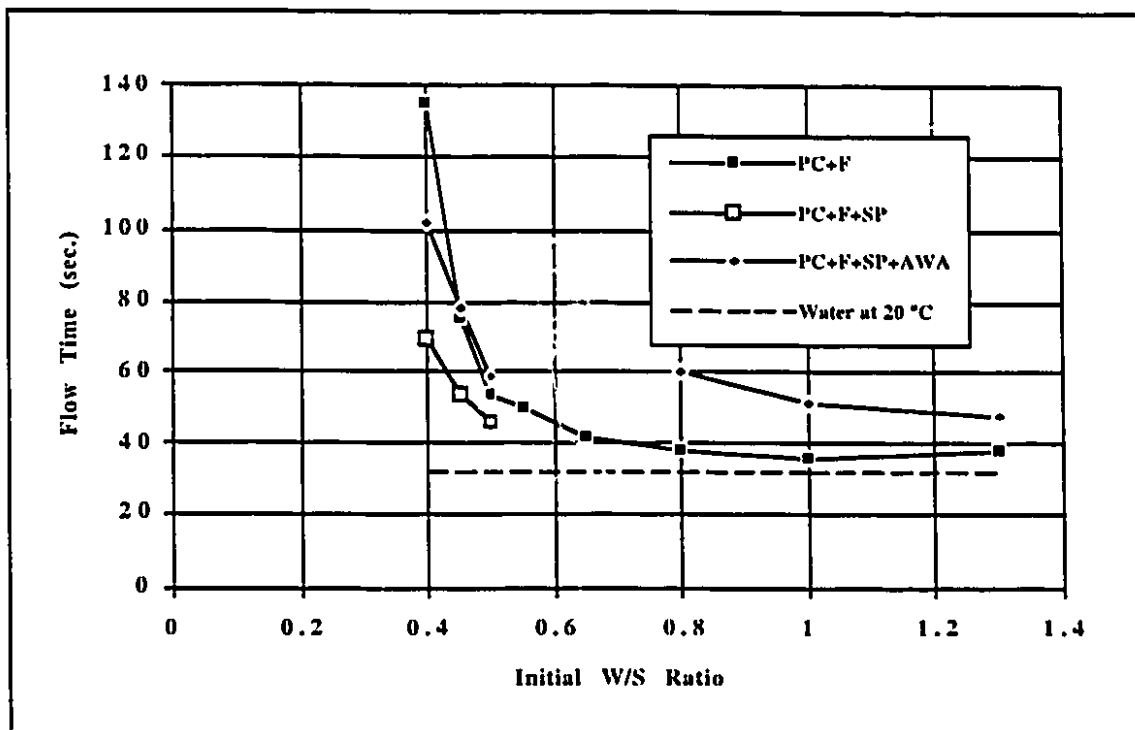


Fig. 7-5 Effect of SP and AWA on flow time of CSA Type 10 cement and F fly ash grouts

7.2.2 Bleeding (stability)

Bleeding tests were performed for grouts with water/solids ratios ranging from 0.4 to 1.3 (0.4, 0.45, 0.5, 0.55, 0.65, 0.8, 1.0 and 1.3). The effect of SP on grout stability was verified at low water/solids ratios as well as the combined effect of SP and AWA. As discussed in section 7.2.1, SP and AWA dosage was based on flow and bleeding characteristics observed during the preliminary testing phase of the program. In an effort to reduce the excessive quantities of bleed water encountered with high water content grouts, a combination of 1.0 percent SP and 0.15 percent AWA was employed. The purpose of the SP was to offset the effects of the AWA on the flow characteristics.

The results of the bleeding tests are presented in Fig. 7-8 to 7-11 (the remaining results are presented in Appendices D and E). These figures indicate the percentage of volume in suspension (VIS) as a function of the initial water/solids ratio. The following observations can be made from the test results:

- Very little bleeding was observed with CSA Type 10 cement grouts, with or without fly ash, for water/solids ratios of 0.4 to 0.55 (both the two hour VIS and the final VIS were greater than 95 percent). A grout is considered unstable if the VIS is under 95 percent after 2 hours (see section 6.5.2 for further details).
- The VIS is inversely proportional to the increase in the grout water content, i.e. higher the water/solids ratio, lower is the VIS (Fig. 7-8 to 7-11).

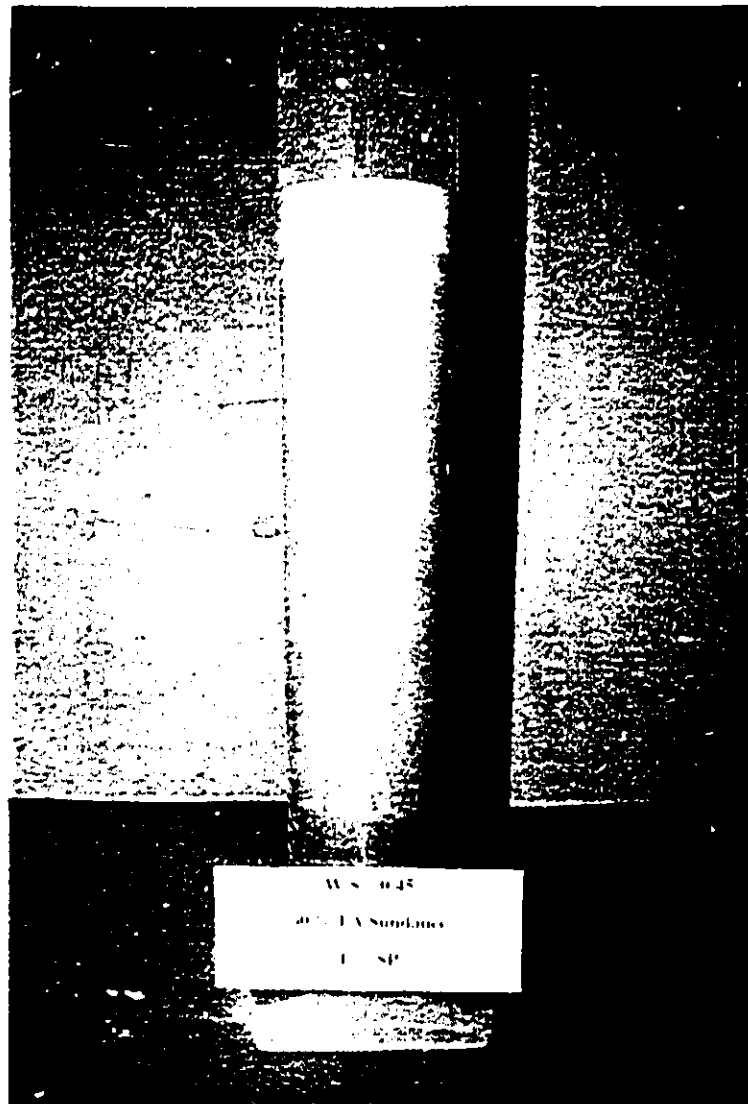


Fig. 7-6 Destabilization of CSA Type 10 cement-based grouts containing C1 fly ash and 2.0 percent SP

- For water/solids ratios greater than 0.55, all grouts, excepting those with AWA, were found to be unstable (Fig. 7-8).
- At low water/solids ratios, the VIS after two hours is similar or very close to the final VIS. However, for ratios greater than or equal to 0.65, the final VIS is slightly smaller (Table C-1 Appendix C). Hence, at low water/solids ratios, all of the bleed water is expelled during the first two hours of the test procedure.

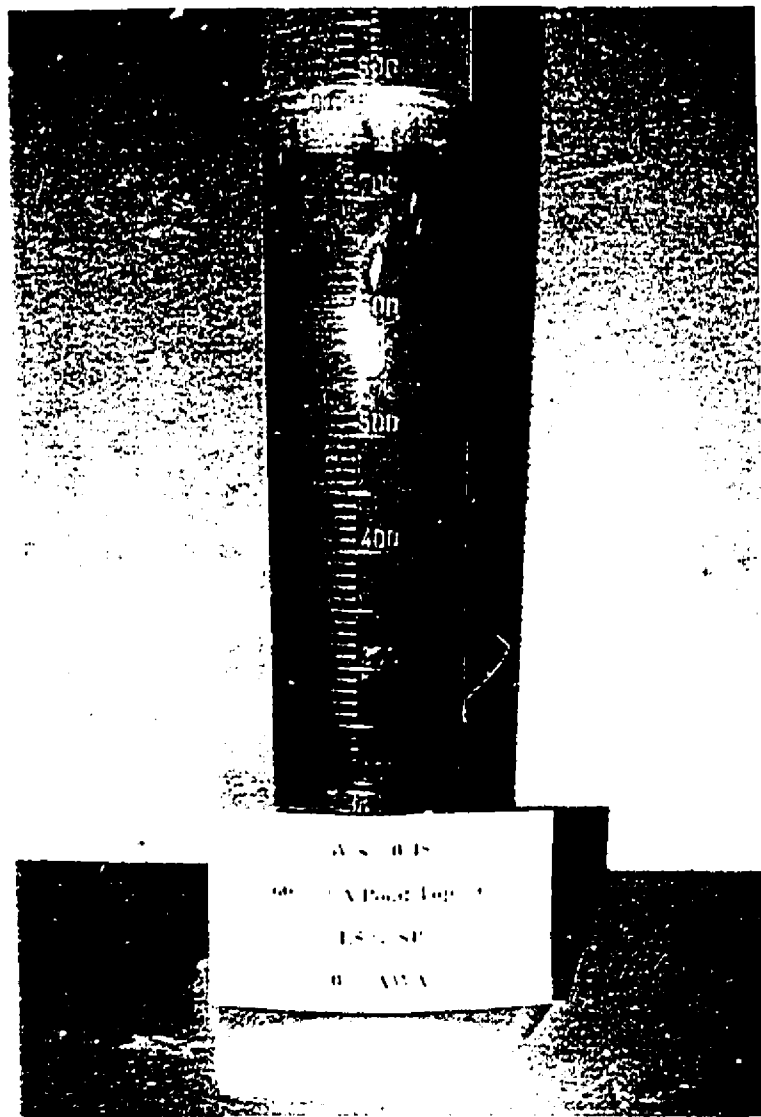


Fig. 7-7 Destabilization of CSA Type 10 cement-based grouts containing F fly ash and 2.0 percent SP

- The addition of fly ash has practically no influence on the stability of CSA Type 10 cement grouts with low water content (Fig. 7-6 and D-1 Appendix D).
- Grout stability is improved by the presence of fly ash at water/solids ratios greater or equal to 0.65 (Fig. 7-8). A reduction in bleeding as high as 12 percent, with respect to the reference grout, was observed for grouts containing C1 fly ash. The C2 and F fly ashes increased the VIS of the reference grout by an average of 5.2 and 1.3 percent, respectively.
- C1 fly ash increased the two hour VIS of a grout with a water/solids ratio of 0.65, from 91 percent (unstable mixture) to 96 percent (stable mixture) (Fig. D-1 Appendix D).
- The addition of 1.5 percent SP did not influence the stability of CSA Type 10 cement grouts with a water/solids ratio of 0.4 and 0.45 (Table C-1 Appendix C). However, a considerable increase in bleeding was observed for grouts with a higher water content.
- The stability of low water/(cement + fly ash) ratio grouts was affected by the addition of 1.5 percent SP. In all instances, the use of SP increased the amount and rate of bleeding (Fig. 7-9 to 7-11 and Fig. E-2 to E-4). As illustrated in Fig. 7-6 and 7-7, the melamine based SP caused the formation of three distinct layers. The layers contain particles originating from the fly ash and the cement. However, without a proper chemical analysis, it is impossible to determine their exact composition. With class C ashes (C1 and C2) a small, rigid, and beige colored layer was formed at the top followed by a gray and a black layer (Fig. 7-6). Some difficulties were encountered when attempting to measure the amount of bleed water, since it did not always manifest itself at the top of the grout. Bleed water was sometimes entrapped between the upper and middle layer due to density differences between the bleed water and top layers water and fine particulate suspension. In the case of class F ash, a beige layer was formed at the top followed by a brown and dark layer (Fig. 7-7). Layer thickness is dependent on the type of the ash and the grout water content.

- The addition of 0.04 percent AWA eliminates the bleeding and the particle separation phenomenon described above, since it increases the cohesion between all solid particles in suspension (Fig. 7-9 to 7-11). However, as described in section 7.2.1, AWAs increase the flow time (i.e. viscosity).
- The quantity of AWA agent required to eliminate bleeding is proportional to the grout water/solids ratio.
- The addition of 0.15 percent AWA and 1.0 percent SP eliminated bleeding of cement fly ash grouts with higher water/solids ratios, i.e. between 0.8 and 1.3 (Fig. 7-9 to 7-11).

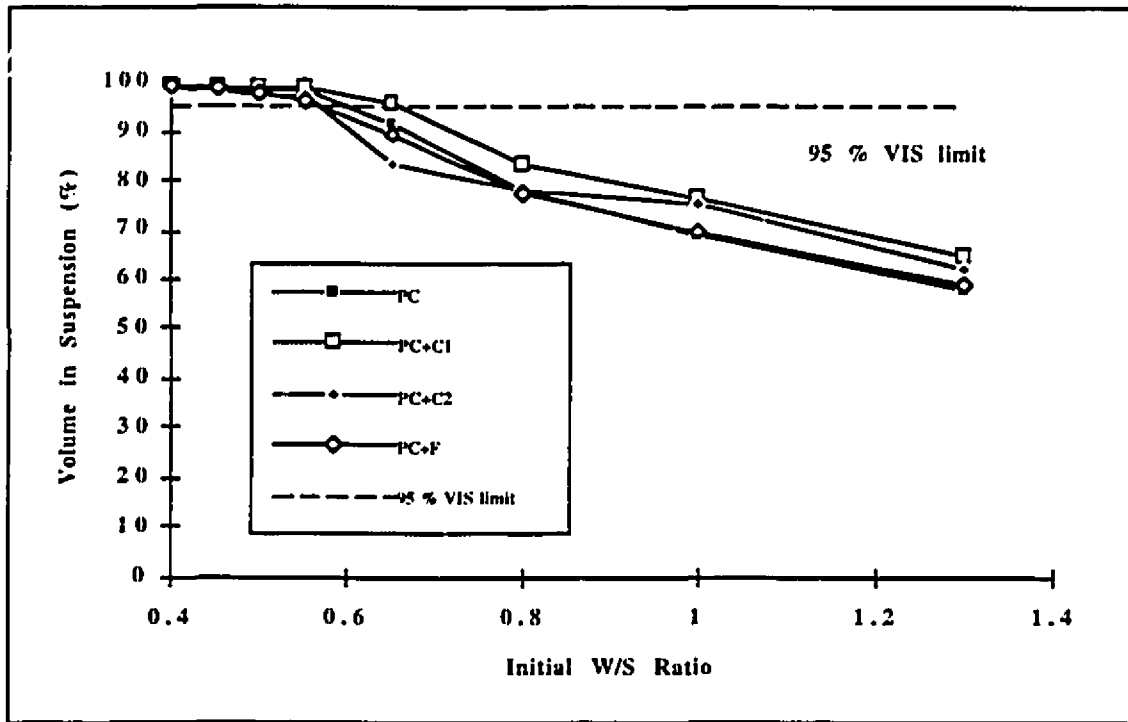


Fig. 7-8 Effect of fly ash on final bleeding of CSA Type 10 cement-based grouts

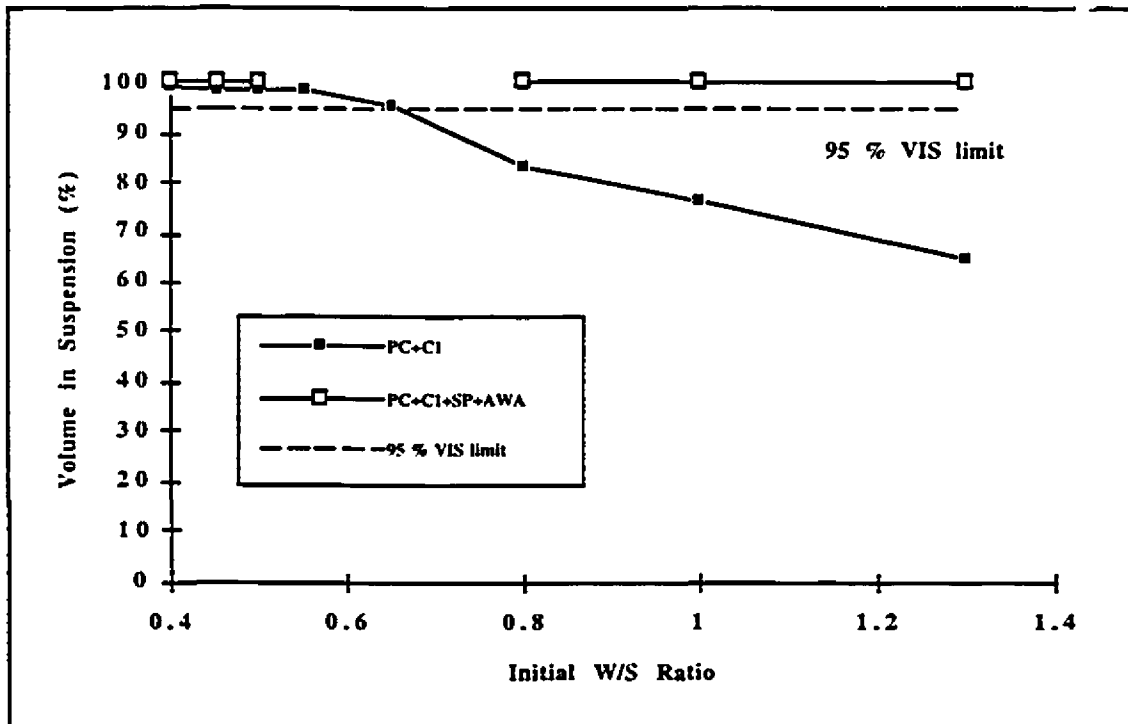


Fig. 7-9 Effect of SP and AWA on final bleeding of CSA Type 10 cement and C1 fly ash grouts

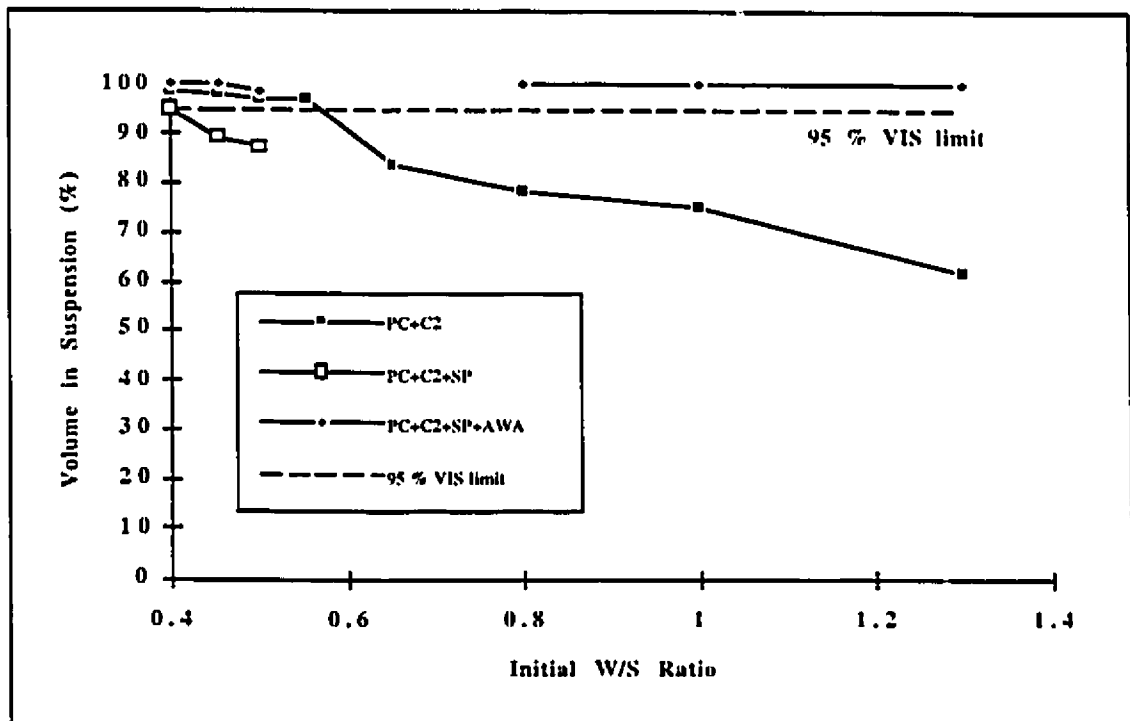


Fig. 7-10 Effect of SP and AWA on final bleeding of CSA Type 10 cement and C2 fly ash grouts

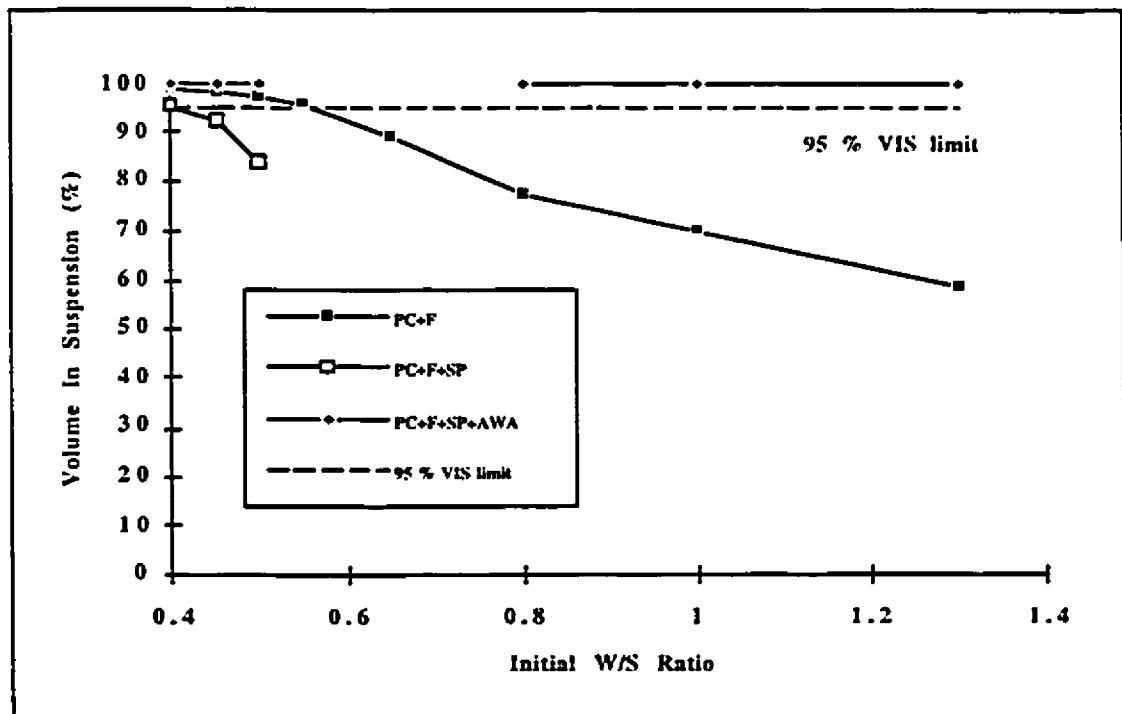


Fig. 7-11 Effect of SP and AWA on final bleeding of CSA Type 10 cement and F fly ash grouts

7.2.3 Setting time

The initial and final setting time of CSA Type 10 cement grouts with and without fly ash, was measured for the following water/solids ratios: 0.5, 0.55 and 0.65. The results are presented in Fig. 7-12 and 7-13 (a tabulated form of the results can be seen in the Appendix E). The following observations can be drawn from the results presented:

- The initial and final setting times of cement pastes and those containing fly ash vary proportionally with the water/solids ratio (Fig. 7-12 and 7-13). As a result of the increased water content, the cement concentration is decreased, thus the hydration reaction slows down and the grout takes a longer time to set.
- The setting time of cement grouts including fly ash show a prolonged setting time from those of an equivalent paste with no fly ash (Fig. 7-12 and 7-13). This phenomenon is attributed to the slow reactive nature of the fly ash and the decreased concentration of cement (60 percent reduction in the cement content as a result of fly ash replacement).
- The initial and final setting time of the cement grouts containing class C fly ashes (C1 and C2) is similar, since their chemical and physical characteristics are almost identical.
- A longer setting time is observed with a grout containing class F fly ash (F) than that of an equivalent grout containing class C fly ash (C1 or C2). The low reactivity of class F fly ash, i.e. low CaO content, is responsible for this phenomenon. As discussed in section 5.2.1, similar results were observed by Davis et al. [1987].

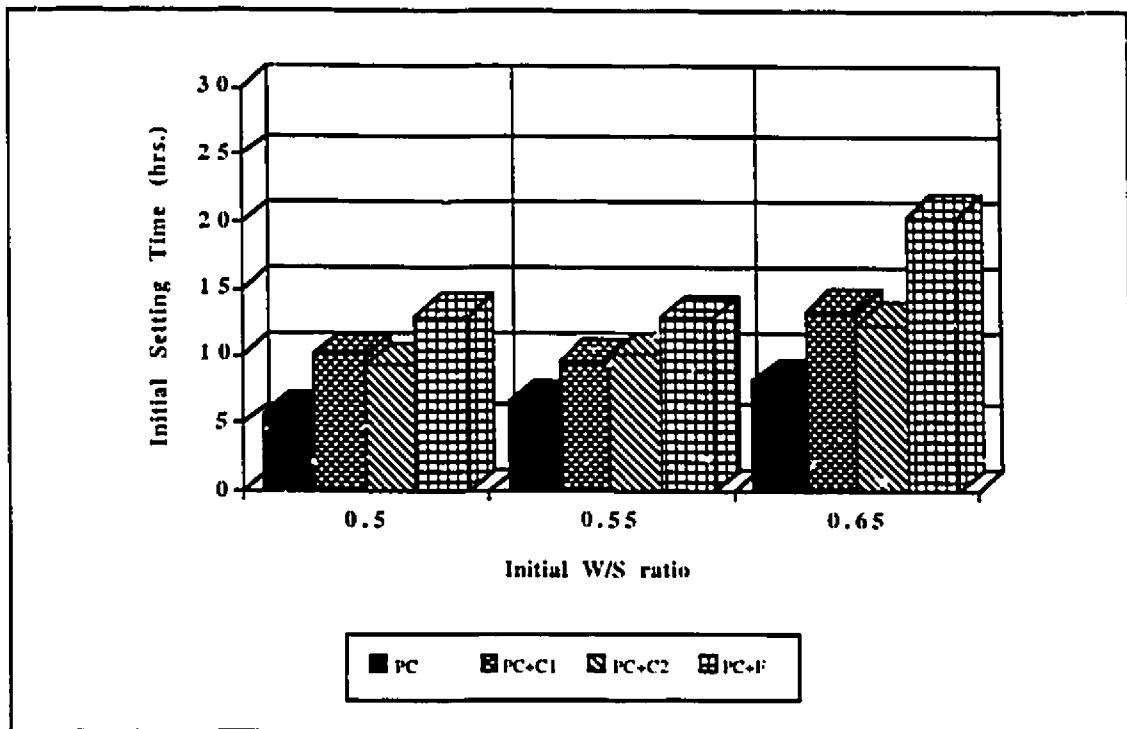


Fig. 7-12 Effect of fly ash on initial setting time of CSA Type 10 cement-based grouts

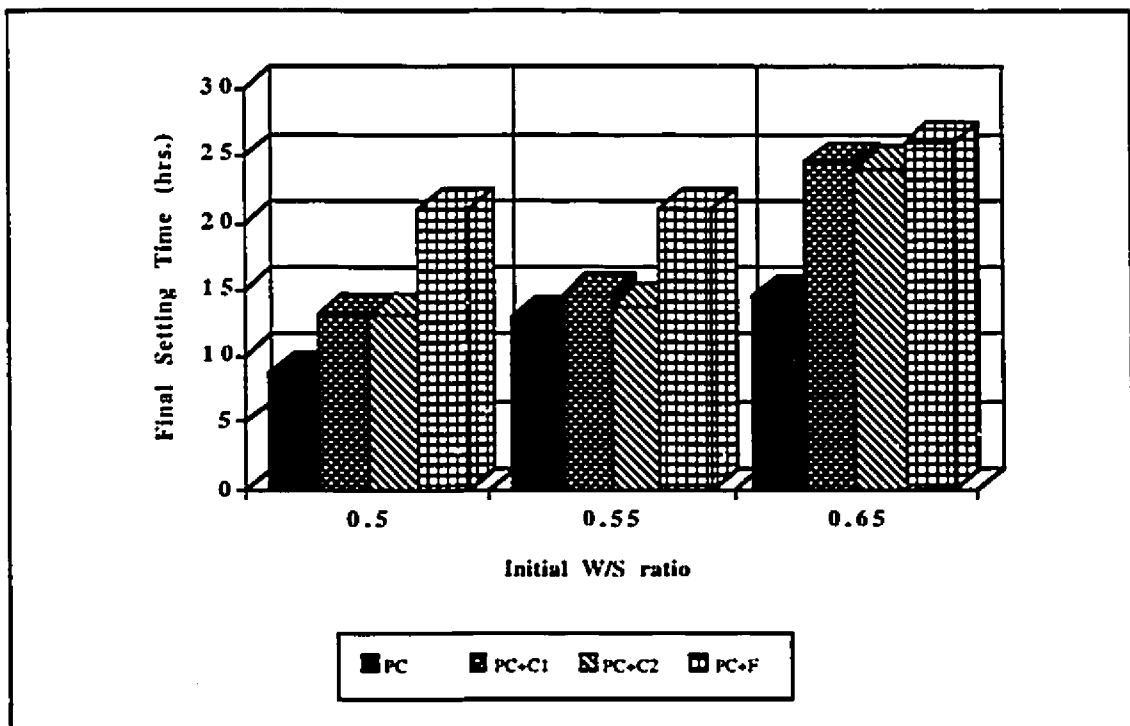


Fig. 7-13 Effect of fly ash on final setting time of CSA Type 10 cement-based grouts

7.3 Hardened grouts

7.3.1 Compressive strength

Compressive strength (f'_c) was determined at 7, 14, 28, and 91 days, for water/solids ratios of 0.5, 0.55, and 0.65 only. The values were adjusted to compensate for specimen size reduction effects as a result of bleeding (the height of high water/solids ratio grouts cylinders dramatically decreases as a result of extensive bleeding). For specimens with a length to diameter ratio (L/D) less than 1.8, the compressive strength results were multiplied by the following correction factors (ASTM Standard C39-86):

L/D^*	Factor
1.75	0.98
1.50	0.96
1.25	0.93
1.00	0.87

* L/D = Length/Diameter ratio

Results for compressive strength at 28 and 91 days as a function of the initial water/solids ratio are presented in Fig. 7-14 and 7-15 (complete results are presented in Appendix F). Table 7-4 presents the compressive strength of grouts containing fly ash with respect to the CSA Type 10 reference grout of an equivalent water/solids ratio. Strength development of the different cement and fly ash mixtures are presented in Fig. 7-16 to 7-18. The results reveal the following:

- The compressive strength (f'_c) of cement grout with and without fly ash at 28 and 91 days is inversely proportional to an increase in the water/solids ratio (Fig. 7-14 and 7-15), i.e. the higher is the water/solids ratio, the lower is the compressive strength.
- The use of fly ash caused a reduction in the 28 and 91 day compressive strength, when compared to the reference grout of an equivalent water/solids ratio (Table 7-4). The difference between the reference grout and the fly ash grout reduced with an increase in age. For example, at a water/solids ratio of 0.55 the apparent compressive strength of cement and C1 fly ash grout at 28 and 91 days is respectively 43 and 82 percent of the reference grout with the same water/solids ratio ($=0.55$).

Table 7-1 Apparent compressive strength of cement fly ash grouts

Initial Water/Solid Ratio	Time (days)	Apparent Compressive Strength * (%)			
		PC	PC + C1	PC + C2	PC + F
0.50					
	28	100	46.1	53.2	40.8
	91	100	67.8	61.0	57.7
0.55					
	28	100	42.7	55.2	36.4
	91	100	82.3	59.9	50.6
0.65					
	28	100	48.7	55.4	34.5
	91	100	64.4	55.1	45.5

* expressed as a percentage of the Portland cement grout with the same water/solids ratio

- As the cement content is decreased, the relative compressive strength also decreases, since fly ash reacts with calcium hydroxide, released by the hydration of cement, to produce CSH (see section 4.2.1) (Fig. 7-14 and 7-15). For a water/solids ratio of 0.5, the compressive strengths at 91 days of CSA Type 10 cement grouts with C1, C2, or F fly ash, as a percentage of the reference grout (CSA Type 10 cement) of an equivalent water/solids ratio, are respectively, 68, 61, and 58 percent, and 64, 55, and 46 percent for a water/solids ratio of 0.65.
- Higher compressive strengths were obtained with cement grouts containing class C fly ashes than with class F fly ashes. Grouts made with F fly ash (class F) produced the lowest compressive strength (Fig. 7-14 and 7-15) because of its low calcium content. Furthermore, as discussed in section 5.2.1, the pozzolanic and cementitious properties of class C ashes generally produce higher compressive strengths than class F ashes, which are only pozzolanic.
- The compressive strength of grouts containing either of the two class C fly ashes (C1 or C2), at an equivalent water/solids ratio, is similar. Slightly higher 28 day compressive strengths were measured with C2 fly ash than with C1 fly ash grouts (Fig. 7-14). Conversely, at 91 days, slightly higher compressive strengths were measured with C1 fly ash grouts (Fig. 7-15). This similarity between the compressive strength of C1 fly ash grouts and C2

fly ash grouts can be attributed to their close resemblance in the chemical and physical composition. Hence, the strength development at early ages (before 28 days) is slightly higher with grouts containing the C2 fly ash. As the grouts mature beyond 28 days, the rate of strength development is slightly higher with the C1 fly ash (Fig. 7-16 to 7-18).

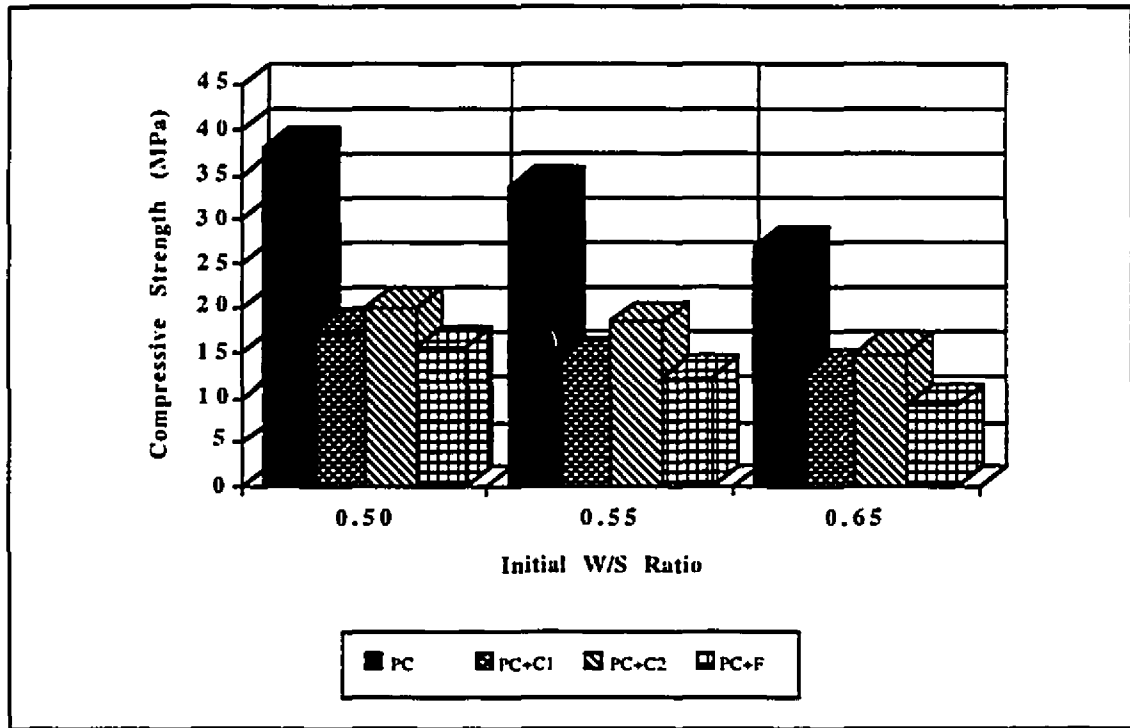


Fig. 7-14 Compressive strength of CSA Type 10 cement grouts with and without fly ash at 28 days

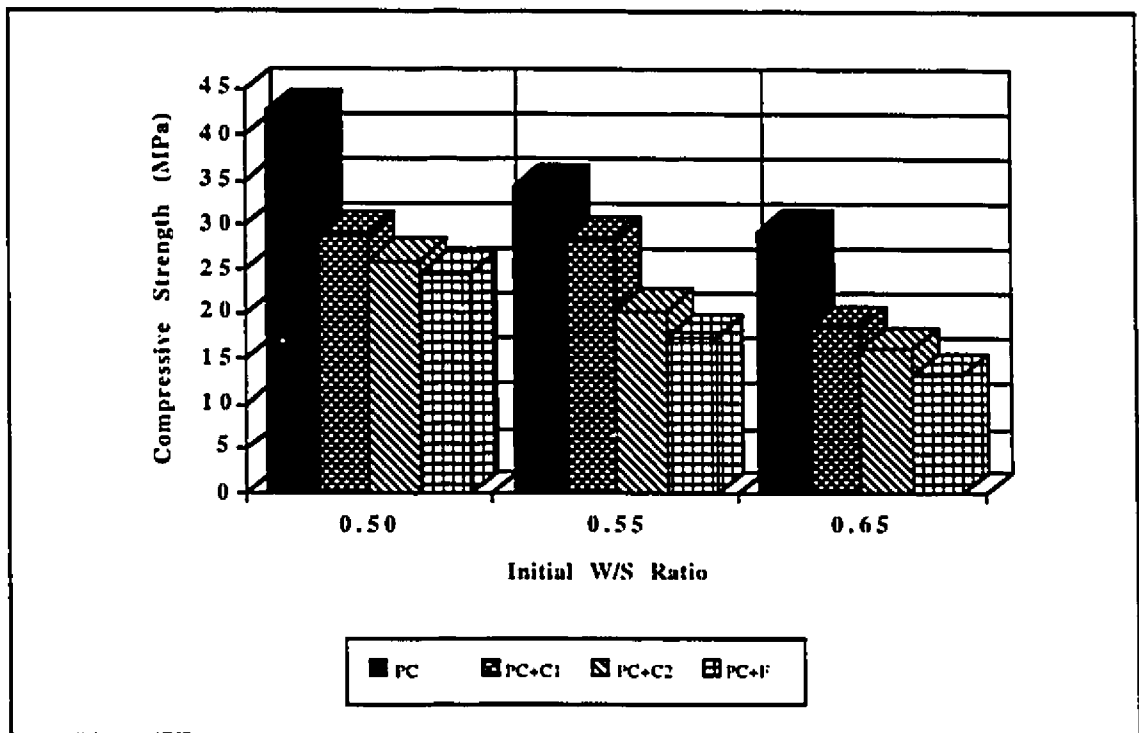


Fig. 7-15 Compressive strength of CSA Type 10 cement grouts with and without fly ash at 91 days

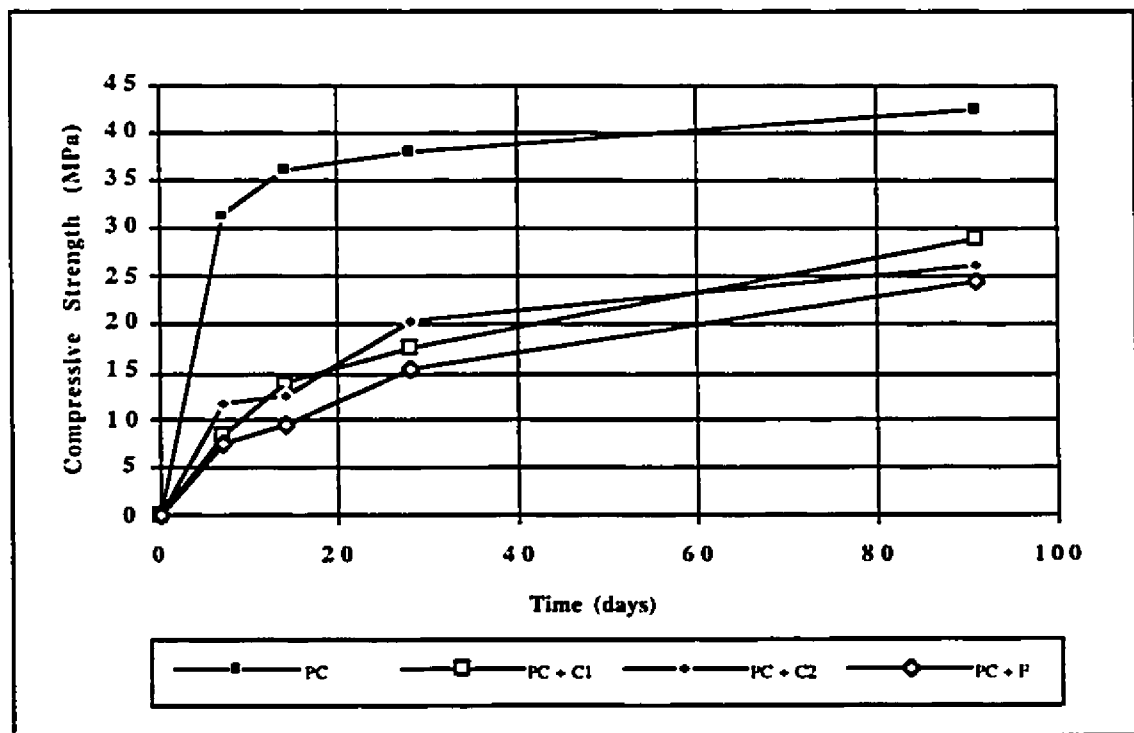


Fig. 7-16 Compressive strength of CSA Type 10 cement grouts with and without fly ash for a water/solids ratio of 0.5

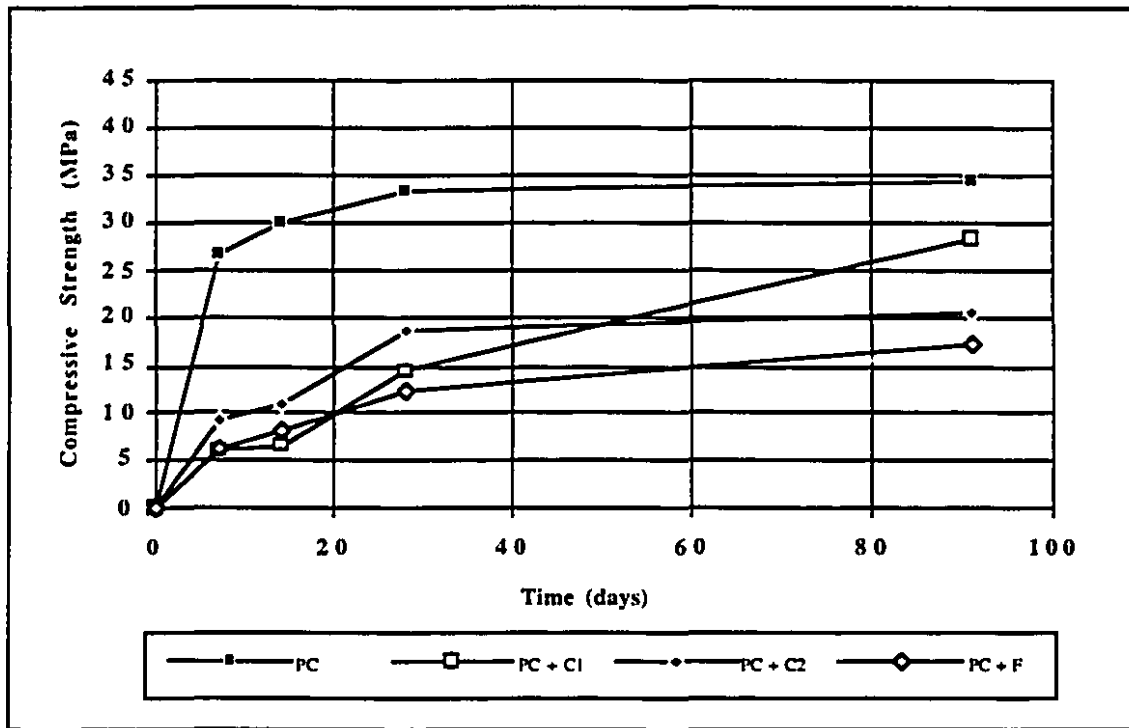


Fig. 7-17 Compressive strength of CSA Type 10 cement grouts with and without fly ash for a water/solids ratio of 0.55

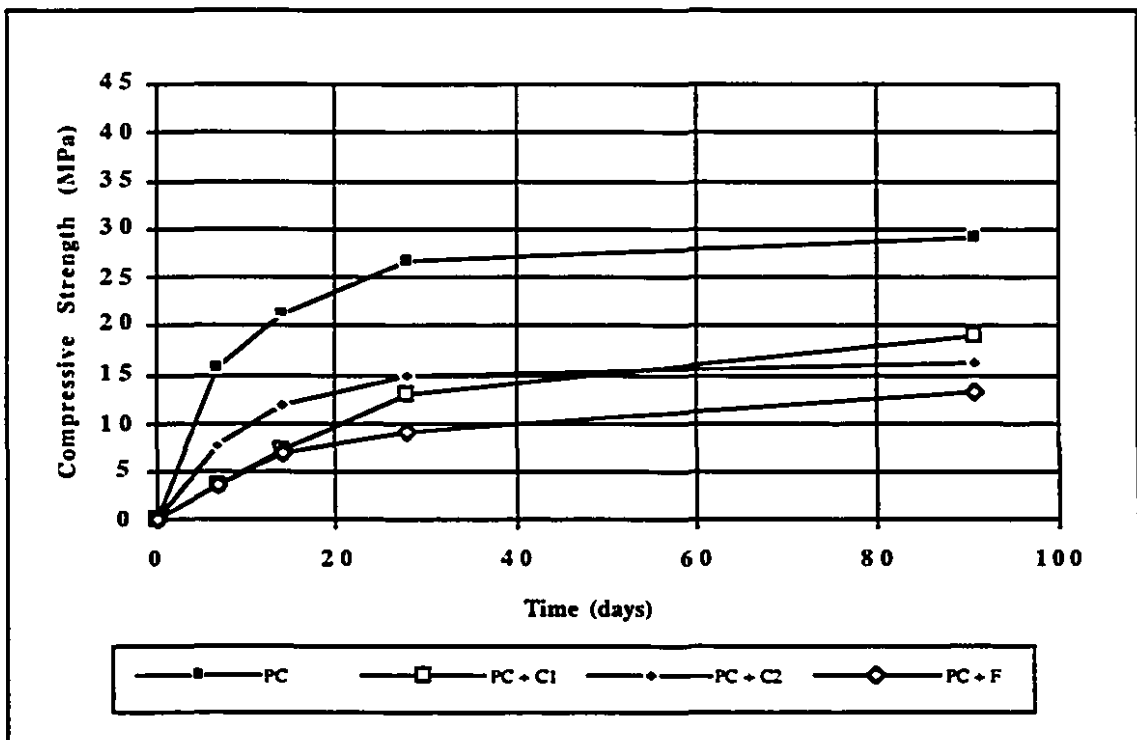


Fig. 7-18 Compressive strength of CSA Type 10 cement grouts with and without fly ash for a water/solids ratio of 0.65

7.3.2 Bond strength

Shear bond strength was measured for CSA Type 10 cement-based grouts with and without fly ash after 14 and 28 days of moist curing (water/solids ratio of 0.5, 0.55, and 0.65). The results are presented in Fig. 7-19 and 7-20, which show the bond strength against the initial water/solids ratio (a tabulated form of the results is presented in Appendix G).

- The results show identical trends in the bond strength development as in the compressive strength.
- The shear bond strength (τ_b) is inversely proportional to the water/solids ratio, and directly proportional to an increase in the curing time (Fig. 7-19 and 7-20).
- As in the case of compressive strength, the shear bond strength of CSA Type 10 cement fly ash grouts is lower than that of the reference cement grout, of an equivalent water/solids ratio, after 14 and 28 days of moist curing (Fig. 7-19 and 7-20).
- The class C fly ashes (C1 and C2) result in a higher bond strength than that of the class F fly ash (F). The C2 fly ash produces a slightly higher bond strength than C1 fly ash. Once again, the difference between the performance of class C and class F fly ashes can be attributed to differences in the chemical compositions, more specifically the CaO content.

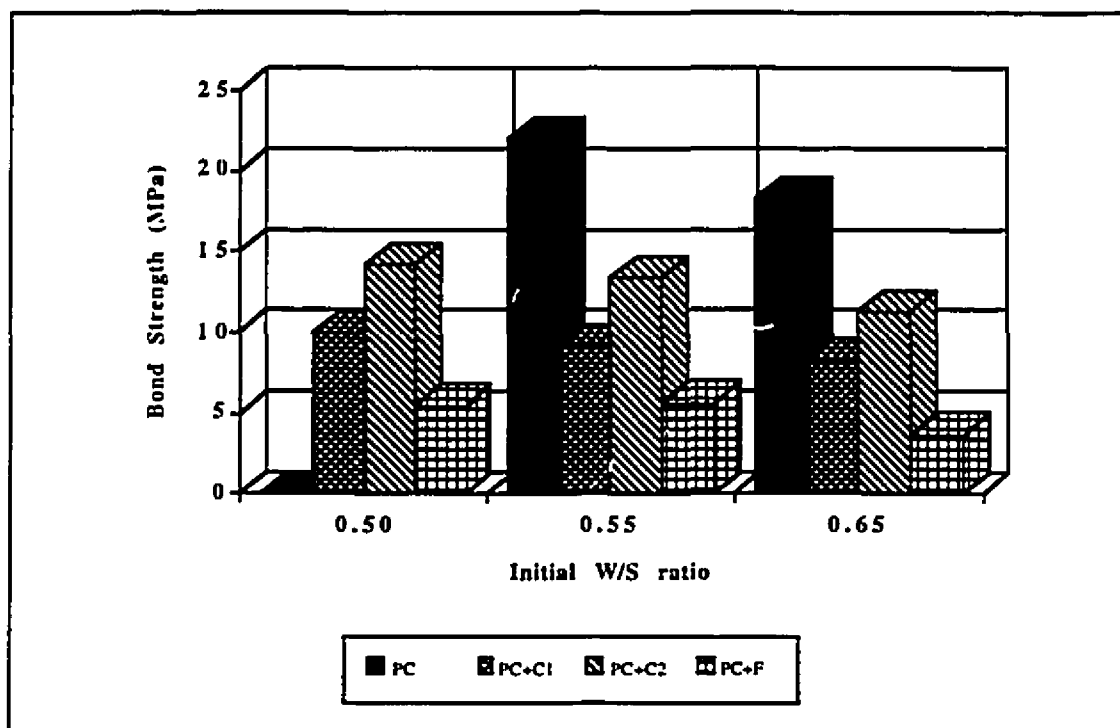


Fig. 7-19 Bond strength of CSA Type 10 cement gouts with and without fly ash at 14 days (bond strength value for PC mixture with a W/S = 0.5 not available)

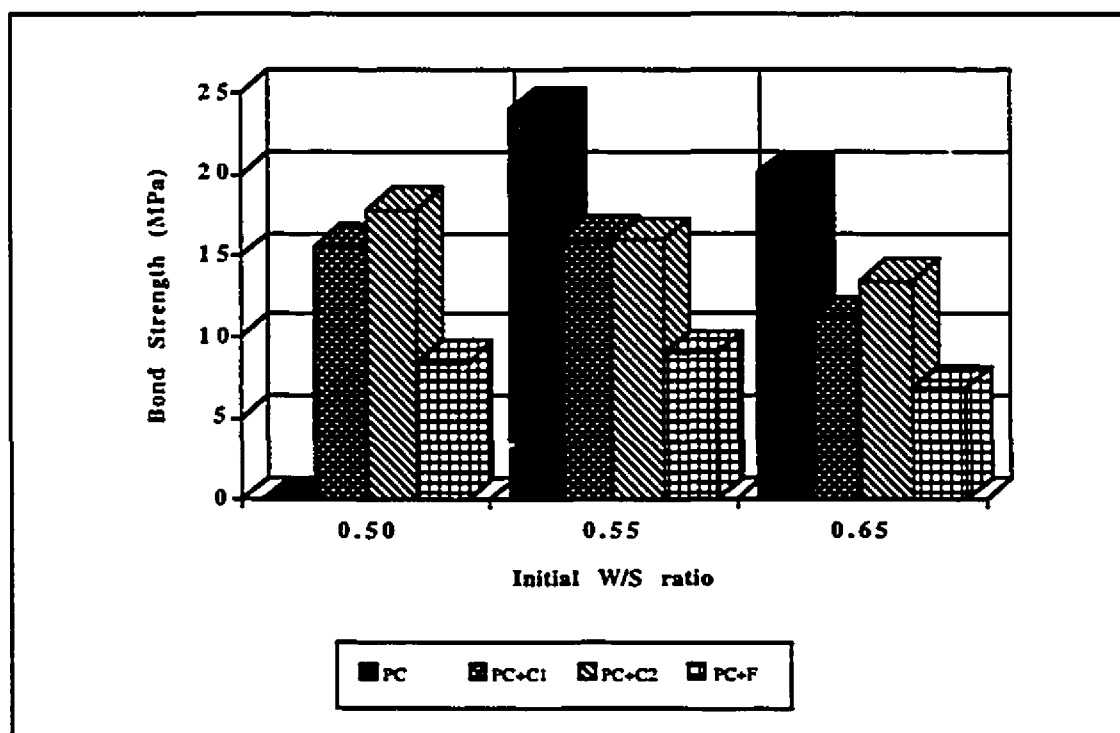


Fig. 7-20 Bond strength of CSA Type 10 cement gouts with and without fly ash at 28 days (bond strength value for PC mixture with a W/S = 0.5 not available)

7.3.3 Modulus of elasticity and Poisson's ratio

The values for the modulus of elasticity and Poisson's ratio were determined for grouts with a water/solids ratio of 0.5, 0.55, and 0.65 after 91 days of moist curing. The results are presented in Fig. 7-21 and 7-22 (also see Appendix H for a tabulated form of the results).

- The modulus of elasticity (E) of CSA Type 10 cement grouts with or without fly ash is inversely proportional to the water/solids ratio (Fig. 7-21).
- The use of fly ash as a SCM results in a reduction in the modulus of elasticity with respect to the reference grout of an equivalent water/solids ratio (Fig. 7-21).
- Grouts containing class C ashes result in a slightly higher modulus of elasticity.
- No apparent trend seems to be present for Poisson's ratio value. However, for the water/solids ratio investigated, the values vary from 0.2 to 0.26. Close examination of Fig. 7-22 reveals that fly ash has little or no influence on the Poisson's ratio value.

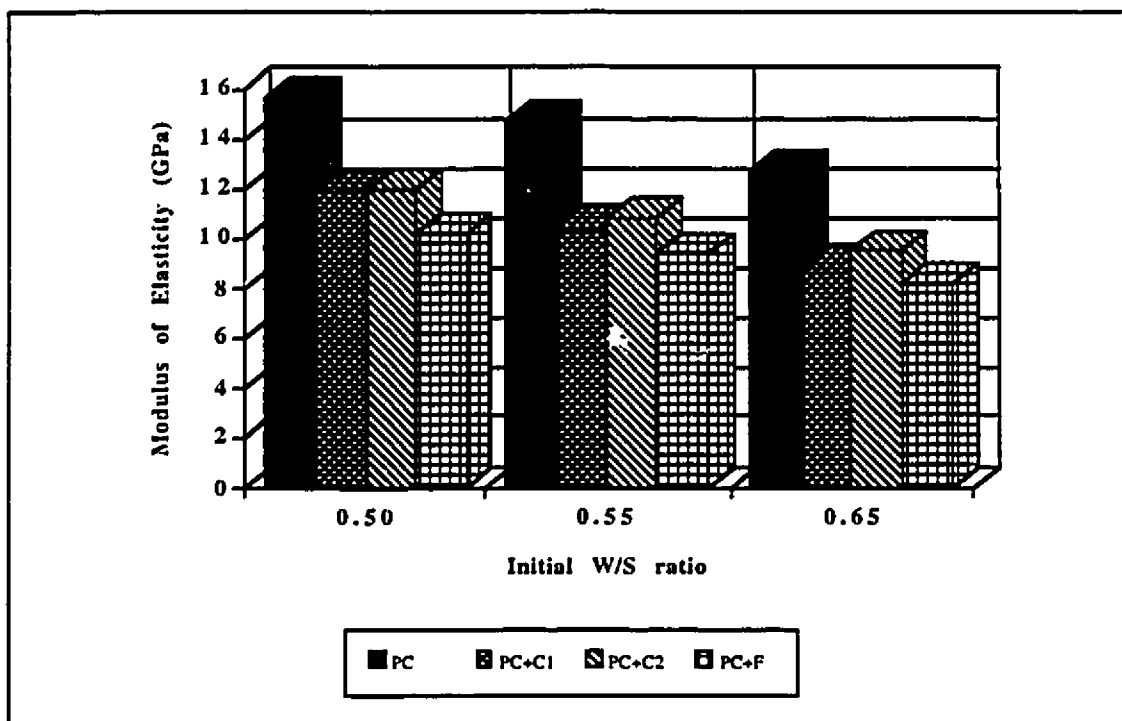


Fig. 7-21 Modulus of elasticity of CSA Type 10 cement-based grouts with and without fly ash

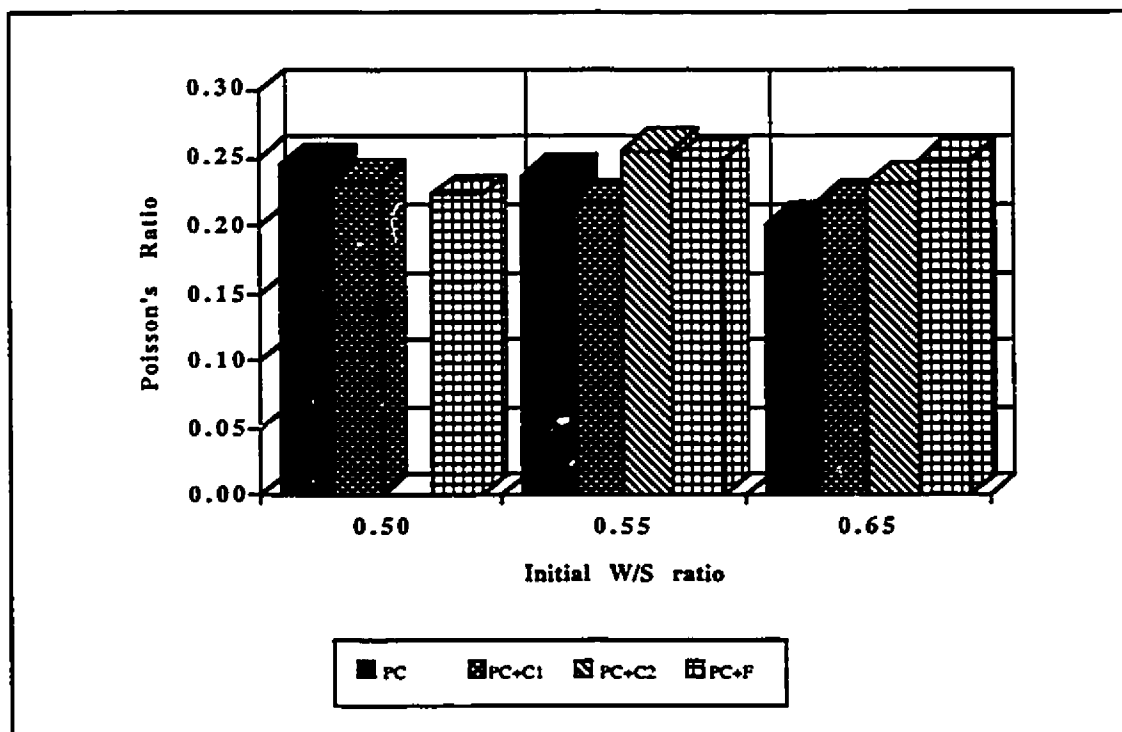


Fig. 7-22 Poisson's ratio of CSA Type 10 cement-based grouts with and without fly ash

7.3.4 Drying-shrinkage

Drying-shrinkage bars (initial water/solids ratio of 0.5, 0.55, and 0.65) were exposed to a dry environment (approximately 30 percent relative humidity, however the ASTM Standard C596-89 specifies a relative humidity of 50 ± 4 percent [Langevin, 1993]). Bars were prepared from CSA Type 10 cement mixtures with and without fly ash. The change in length was measured at 3, 7, 14, 21, 28, 56, 91, and 183 days (shrinkage values are considered negative whereas expansive ones are positive).

Figures 7-23 to 7-25 present the percentage change in the length of the bar as a function of time (the remaining results are presented in Appendix I and J). Table 7-5 presents drying-shrinkage of grouts containing fly ash with respect to the CSA Type 10 reference grout of an equivalent water/solids ratio. The following observation were made from the test results:

- The drying-shrinkage of CSA Type 10 cement grouts with or without fly ash is proportional to the increase in the water/solids ratio (Fig. J-1 to J-4 Appendix J). Identical trends were reported by Mehta [1985] for pure cement pastes.
- Shrinkage was observed with CSA Type 10 cement grouts with or without fly ash exposed to a dry environment. Drying-shrinkage occurs as a result of the loss of physically adsorbed water from CSH, when a grout sample is exposed to ambient humidity which is below saturation [Mehta, 1986].
- As illustrated in Table 7-5, less shrinkage was observed with grouts containing fly ash compared to the reference grouts with an equivalent water/solids ratio (Fig. 7-23 to 7-25). The greatest reduction was observed with C1 fly ash followed by F fly ash.
- As the grout water content increases, the effectiveness of fly ash, in terms of drying-shrinkage reduction, decreases (Table 7-5). For example, at water/solids ratios of 0.5 and 0.65, the relative reduction in shrinkage observed, when C1 fly ash is used as a SCM, is respectively, 41 and 18 percent.

- For grouts containing C1 or F fly ashes, the majority of the drying shrinkage takes place before 91 days (Table 7-5). In other words, the rate of shrinkage is high for the first 91 days and relatively low for the 91 to 183 day interval. On the other hand, the rate of shrinkage of the reference grouts and grouts containing C2 fly ash remains high for the 91 to 183 day interval.

Table 7-2 Apparent change in length of CSA Type 10 cement fly ash grout bars

Initial Water/Solids Ratio	Time (days)	Apparent Change in Bar Length (%)			
		PC	PC+C1	PC+C2	PC+F
0.5	0-91	69.7	52.0	53.8	66.0
	91-183	100.0	58.8	80.8	76.2
0.55	0-91	75.0	73.0	62.6	80.6
	91-183	100.0	80.4	81.4	90.3
0.65	0-91	93.2	79.1	93.0	83.8
	91-183	100.0	82.0	99.4	87.7

* expressed as a percentage of the reference grout of an equivalent water/solids ratio

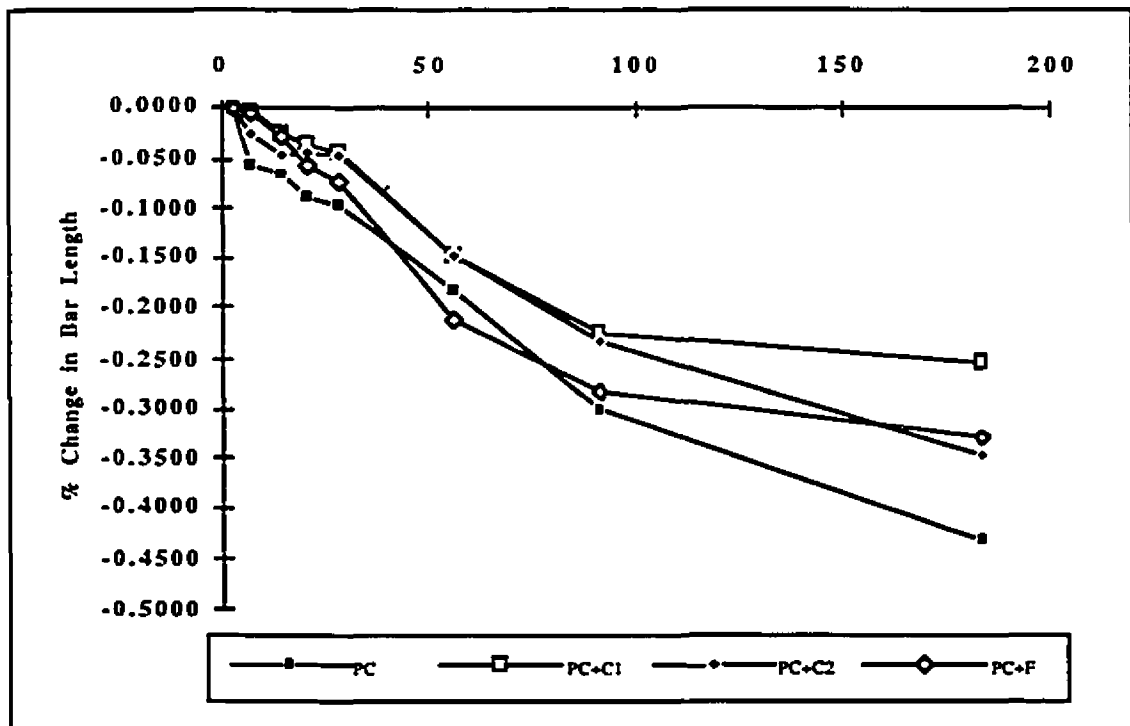


Fig. 7-23 Drying-shrinkage of grouts with a water/solids ratio of 0.5

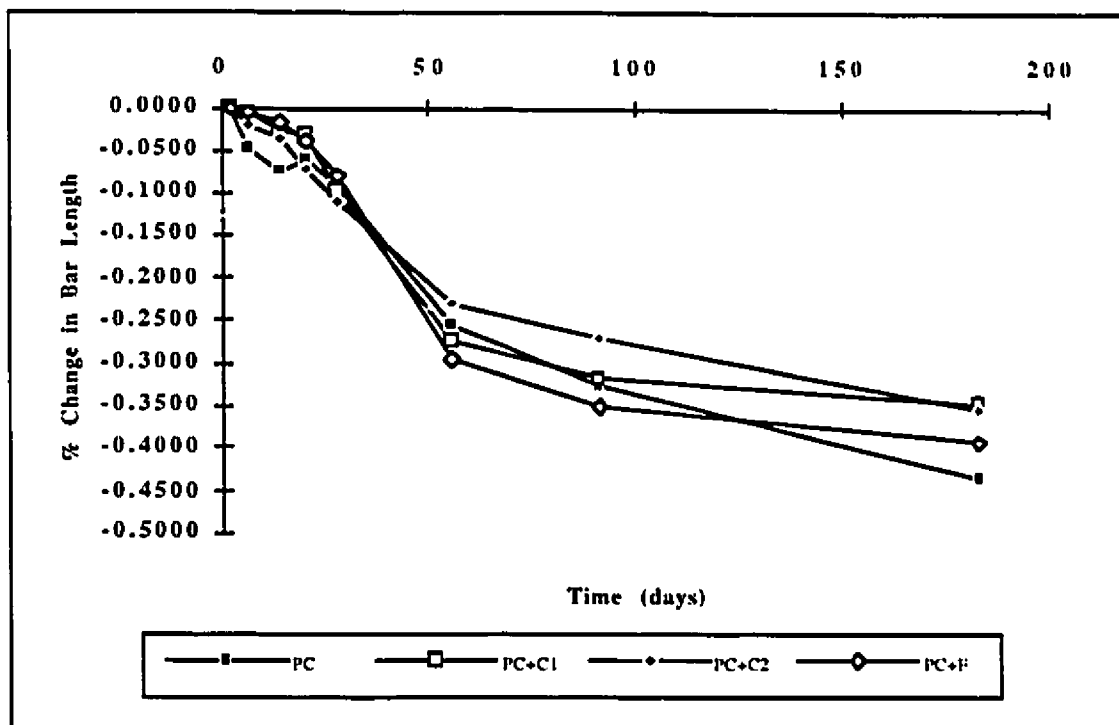


Fig. 7-24 Drying-shrinkage of grouts with a water/solids ratio of 0.55

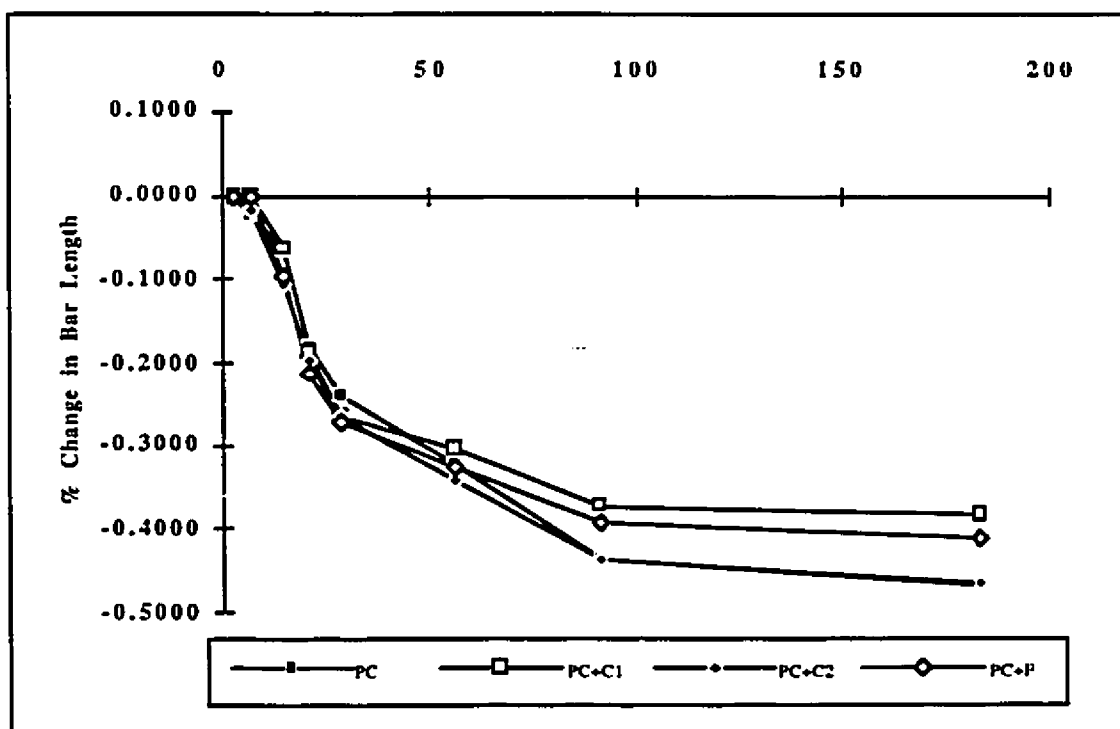


Fig. 7-25 Drying-shrinkage of grouts with a water/solids ratio of 0.65

CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS

The addition of fly ash, particularly high volumes of fly ash, in cement-based grouts has considerable effect on the rheological and mechanical properties of the grouts. In addition, to reduce the flow time of low water/solids ratio grouts and to increase the stability of high water/solids ratio grouts with or without fly ash, the effects of SP as well as the combined effects of SP and AWA were investigated. The following conclusions and recommendations can be made:

- 1 - The particle size distributions of the fly ashes investigated (C1, C2, F) are similar to that of CSA Type 10 cement.
- 2 - Based on the CaO (calcium oxide) content, both C1 and C2 fly ashes are considered high calcium fly ashes (12.45 % and 15.81 % respectively) and are designated class C. On the other hand, the CaO content of F fly ash is relatively low (2.8 %), making this a low-calcium fly ash (class F).
- 3 - The addition of fly ash reduces significantly the flow time of low water/solids ratio (0.4 to 0.65) grouts. C2 fly ash demonstrates the largest reduction. No reduction in flow time is observed at high water/solids ratios (> 0.65).
- 4 - The stability of CSA Type 10 cement grout is improved by the addition of fly ash for water/solids ratios greater than or equal to 0.65. Of the three fly ashes, C1 fly ash demonstrates the largest reduction in bleeding. On the other hand, the addition of fly ash has practically no influence on the stability of CSA Type 10 cement grouts with water/solids ratios lower than 0.65.
- 5 - The slow reactive nature of fly ash is responsible for the prolonged setting time, when compared to a grout with an equivalent cementitious content without fly ash. The low calcium content class F fly ash (F) exhibits the longest setting time.

- 6 - The use of fly ash reduces the early compressive and bond strengths, when compared to the reference grout. However, as they mature, their strengths are closer to those of pure cement grouts.
- 7 - Higher compressive and bond strengths are produced with class C fly ash grouts than with class F fly ash grouts. The low calcium oxide content of the class F fly ash is principally responsible for this phenomenon.
- 8 - The addition of any of the three fly ashes reduces the modulus of elasticity value and has practically no influence on the Poisson's ratio.
- 9 - Fly ash reduces the amount of drying shrinkage compared to pure cement grout with an equivalent water/solids ratio.
- 10 - Superplasticizer decreases the flow time of high solid content grouts. Beyond a water/solids ratio of 0.55, the effect of SP on flow time is neglectable.
- 11 - The stability of CSA Type 10 cement and fly ash grouts is significantly affected by the addition of SP. The addition of SP causes the solid particles to separate into three phases of unknown composition.
- 12 - Anti-washout agents significantly reduce the bleeding of high water/solids ratio cement grouts with and without fly ash. On the other hand, they also increase the flow time and a SP must always be used with them.
- 13 - No benefit is gained by using an SP with low water/solid ratio grouts, since SP destabilizes cement fly ash suspension and the use of AWAs increase the flow time.

CHAPTER 9.0

FUTURE RESEARCH NEEDS

In the light of the results obtained from the experimental program, the following future research needs can be established:

- Determine the effect of low temperature (5 to 10° C) on the rheological and mechanical properties of cement-fly ash grouts. This will predict the behavior of the grout in northern climates.
- Determine the mechanical properties of cement fly ash grouts with water/solids ratios smaller than 0.5 and larger than 0.65.
- Determine the rheological and mechanical properties of:
 - high-volume fly ash (up to 95 percent replacement cement for fly ash) flowable fill or controlled low strength materials (CLSM) used as backfill material around sewer pipes or to fill underground voids (see section 3.1.2.1 for more details);
 - CSA Type 10 Portland cement, fly ash and a third cementing material with highly pozzolanic properties, such as rice-husk ash or silica fume (i.e. ternary system), which might compensate for the loss in early strength which is encountered in cement HVFA grouts;
 - CSA Type 30 Portland cement and high-volume fly ash grouts (compare with CSA Type 10 cement and fly ash mixtures);
 - CSA Type 30 Portland cement, fly ash and rice-husk ash or silica fume grouts (compare with CSA Type 10 cement and fly ash mixtures);
 - high-alumina cement and high-volume fly ash grouts (compare with CSA Type 10 cement and fly ash mixtures);

- high-alumina cement, fly ash and rice-husk ash or silica fume grouts (compare with CSA Type 10 cement and fly ash mixtures);
- high-alumina and rice-husk ash grouts (compare with CSA Type 10 cement and fly ash mixtures).

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APPENDIX A

**FLOW TIME
(TABLE)**

Table A-1 Effect of fly ash and chemical admixtures on the flow time of type 10 cement-based grouts

Mix Description	Flow Time (sec.)							
	Water/Solids Ratio							
	0.4	0.45	0.5	0.55	0.65	0.8	1	1.3
Cement only								
PC		126	80	56	44	39	37	36
PC + SP	71	56	54					
Sundance								
PC+C1	194	92	67	55	45	39	38	36
PC+C1+SP	73	61	56					
PC+C1+SP+AWA	130	80	67			65	56	48
Thunder Bay								
PC+C2	74	56	47	46	41	37	37	46
PC+C2+SP	58	50	44					
PC+C2+SP+AWA	83	63	54			57	49	53
Point Tupper								
PC+F	135	75	54	50	42	38	36	38
PC+F+SP	69	54	46					
PC+F+SP+AWA	102	78	59			60	52	48

N.F. = no flow

APPENDIX B

**FLOW TIME
(GRAPHS)**

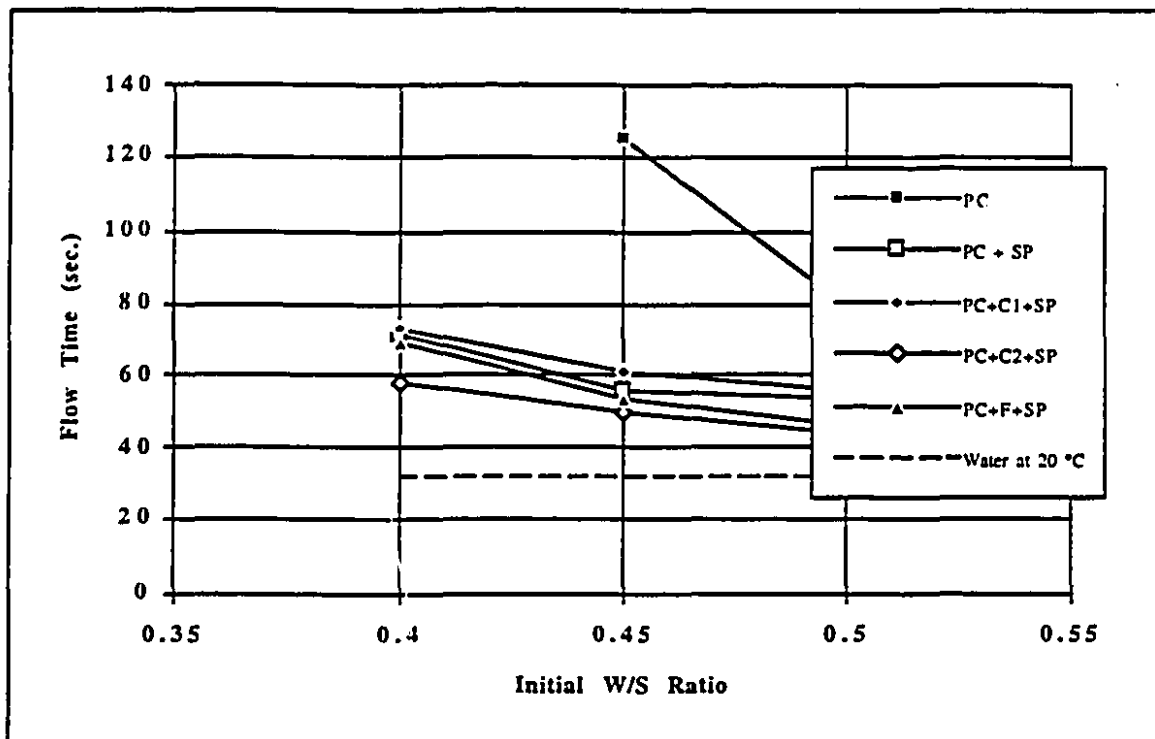


Fig. B-1 Effect of SP on the flow time of low water/solids type 10 cement-based grouts with and without fly ash

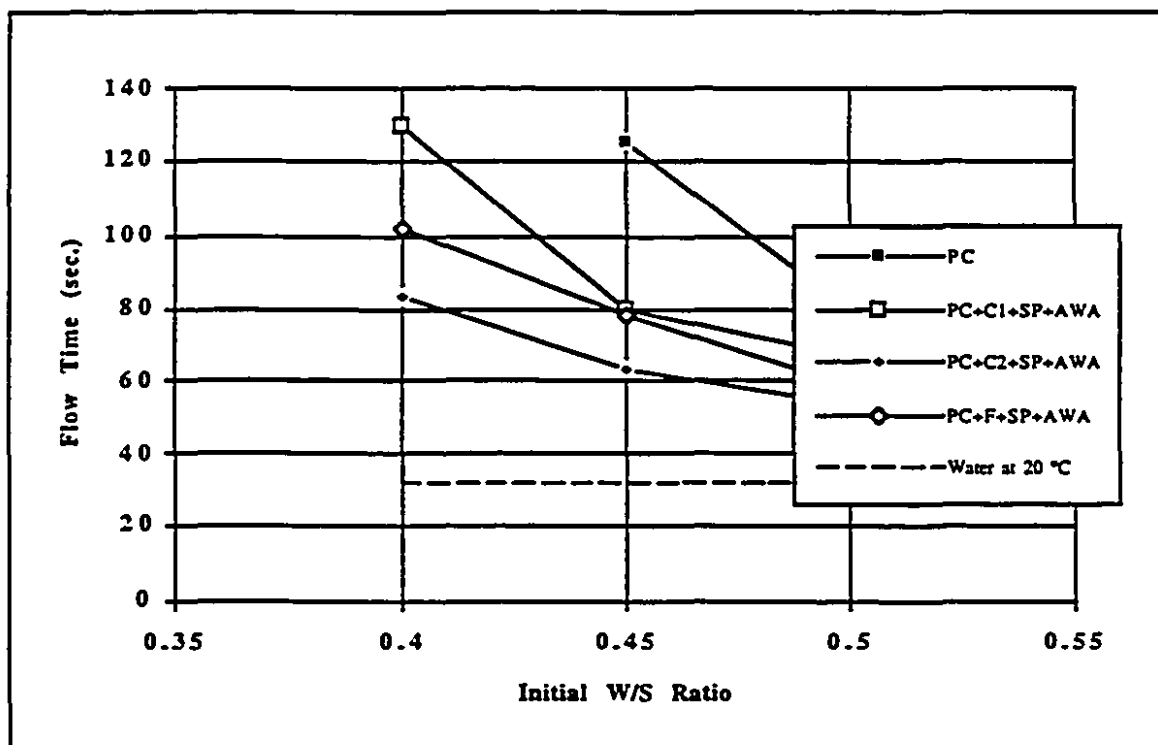


Fig. B-2 Effect of SP and AWA on the flow time of low water/solids type 10 cement-based grouts with and without fly ash

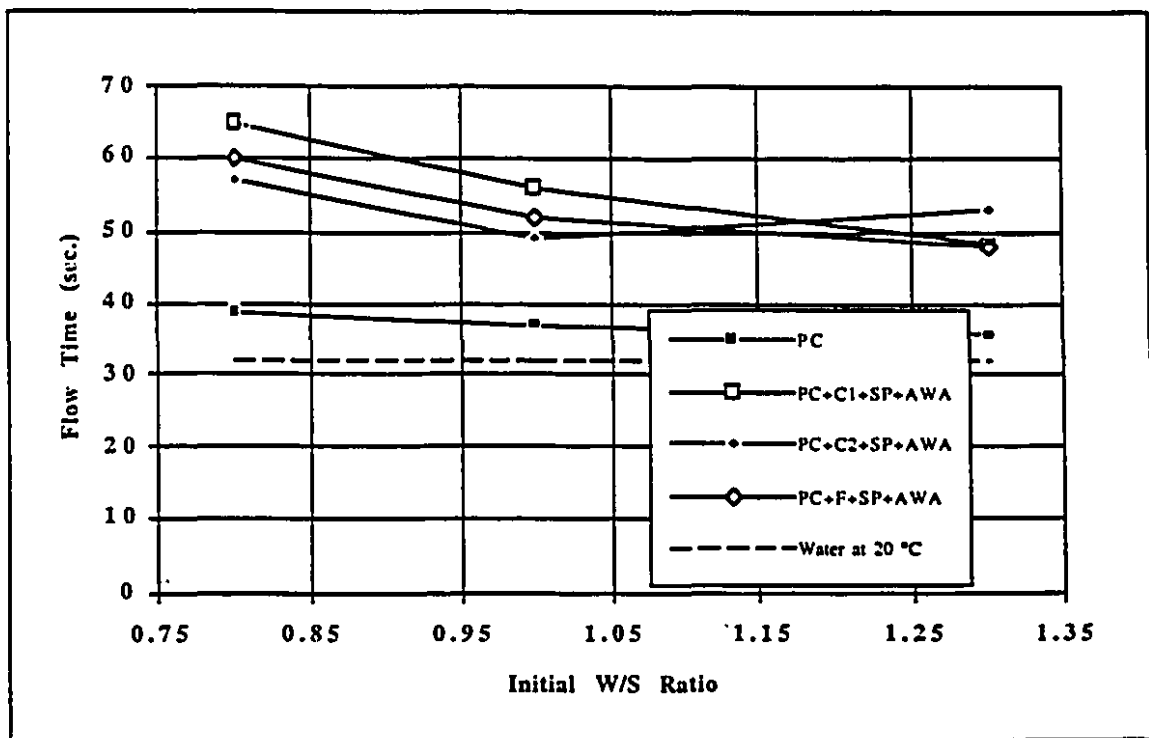


Fig. B-3 Effect of SP and AWA on the flow time of high water/solids type 10 cement-based grouts with and without fly ash

APPENDIX C
VOLUME IN SUSPENSION
(TABLE)

Table C-1 Effect of SP and/or AWA on the VIS of type 10 cement based grout with or without fly ash

Mix Description	Volume in Suspension (%)															
	Water/Solid Ratio															
	0.4		0.45		0.5		0.55		0.65		0.8		1.0		1.3	
	2 hrs.	final	2 hrs.	final	2 hrs.	final	2 hrs.	final	2 hrs.	final	2 hrs.	final	2 hrs.	final	2 hrs.	final
Cement only																
PC	99	99	99	99	98	98	98	98	92	91	78	78	78	69	66	58
PC + 1.5 % SP	99	99	100	100	90	86										
C1 Fly Ash Mixes																
PC+C1	99	99	99	99	99	98	98	98	97	96	86	83	85	76	68	65
PC+C1+1.5 % SP	U.M.	U.M.	U.M.	U.M.	U.M.	U.M.										
PC+C1+1.5 % SP+0.04 % AWA	100	100	100	100	100	100										
PC+C1+1.0 % SP+0.15 % AWA											0	100	0	100	0	100
C2 Fly Ash Mixes																
PC+C2	99	99	99	98	97	97	97	97	84	83	78	78	73	75	62	62
PC+C2+1.5 % SP	98	95	96	89	94	87										
PC+C2+1.5 % SP+0.04 % AWA	100	100	100	100	99	99										
PC+C2+1.0 % SP+0.15 % AWA											0	0	0	0	0	0
F Fly Ash Mixes																
PC+F	99	99	99	99	98	98	97	96	89	89	79	78	78	70	67	59
PC+F+1.5 % SP	96	96	93	93	84	84										
PC+F+1.5 % SP+0.04 % AWA	100	100	100	100	100	100										
PC+F+1.0 % SP+0.15 % AWA											0	0	0	0	0	0

U.M. = unstable mix

APPENDIX D

**TWO HOUR VOLUME IN SUSPENSION
(GRAPHS)**

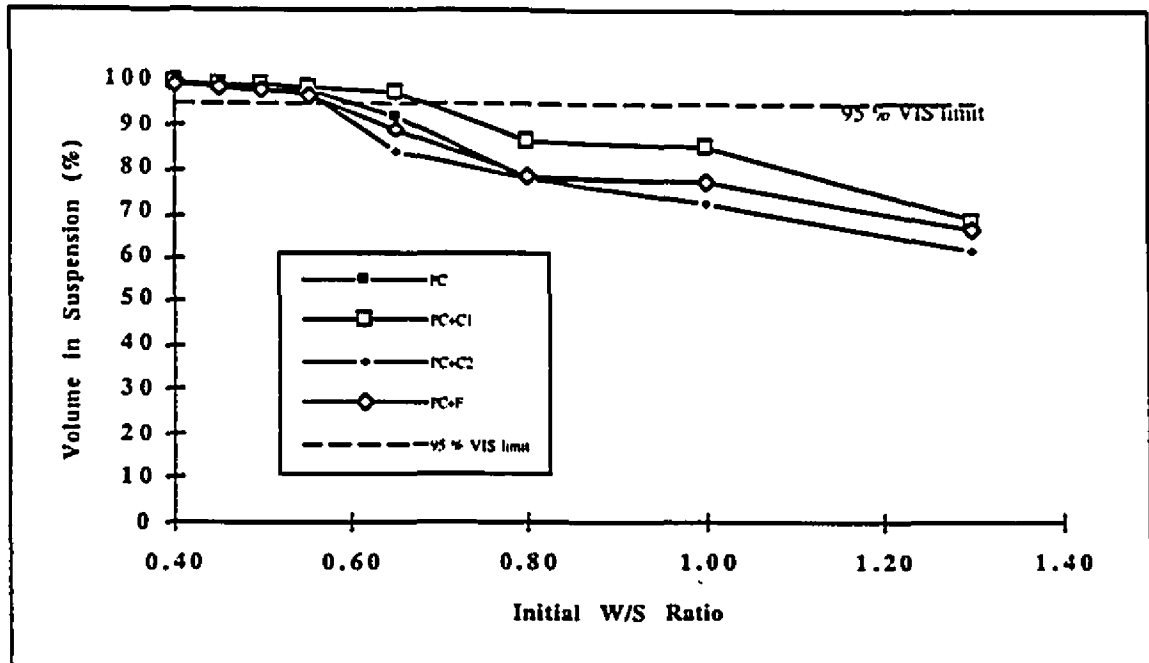


Fig. D-1 Effect of fly ash on the two hour bleeding of type 10 cement-based grouts

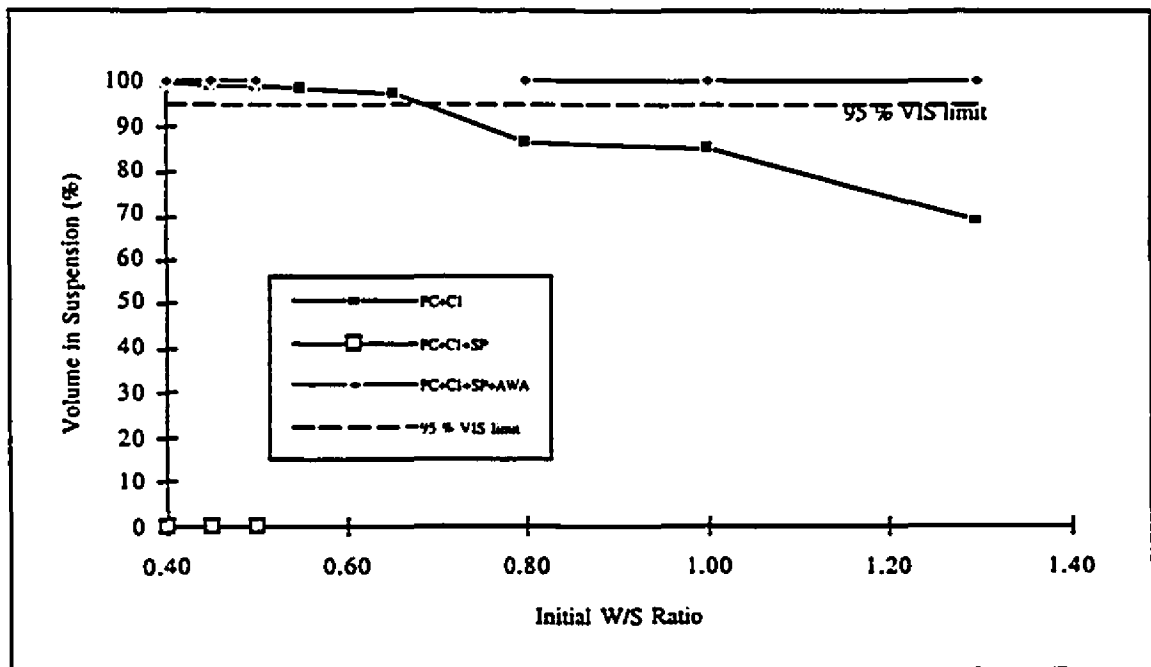


Fig. D-2 Effect of fly ash on the two hour bleeding of type 10 cement and C1 fly ash grouts

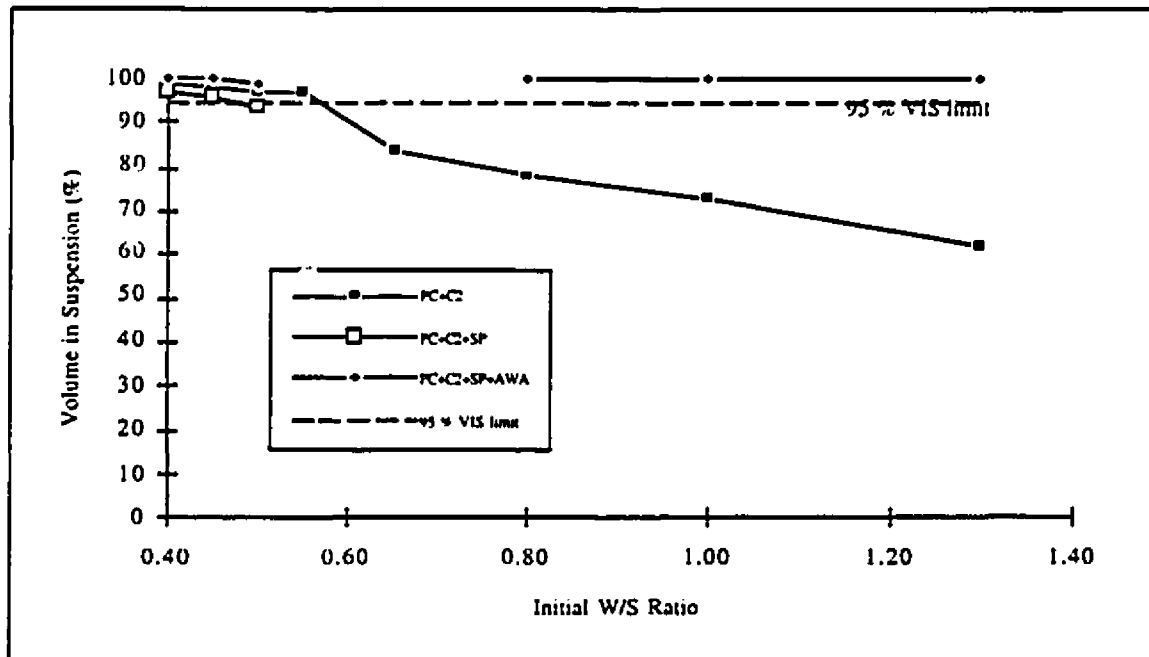


Fig. D-3 Effect of fly ash on the two hour bleeding of type 10 cement and C2 fly ash grouts

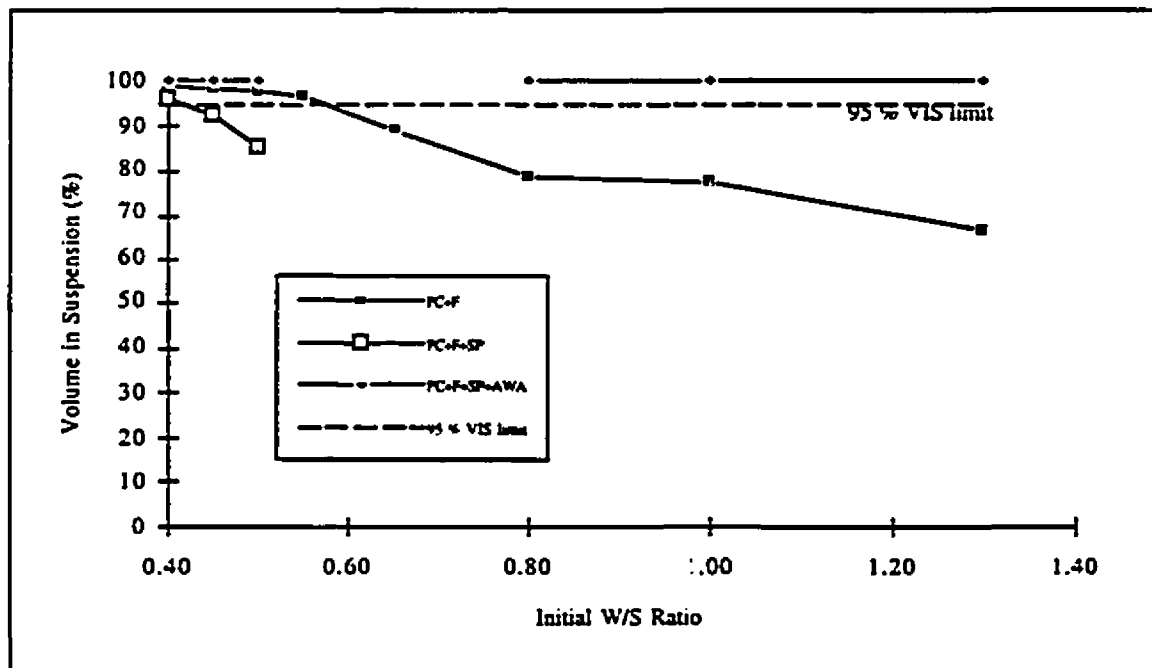


Fig. D-4 Effect of fly ash on the two hour bleeding of type 10 cement and F fly ash grouts

APPENDIX E
SETTING TIME
(TABLE)

Table E-1 Effect of fly ash on setting time of type 10 cement based grouts

Initial Water/Solid Ratio	Initial Setting Time (hrs.)				Final Setting Time (hrs.)			
	PC	PC+C1	PC+C2	PC+F	PC	PC+C1	PC+C2	PC+F
0.5	6	10	9	13	9	13	13	21
0.55	7	10	10	13	13	15	14	21
0.65	8	14	13	21	15	25	24	26

APPENDIX F

COMPRESSIVE STRENGTH
(TABLE)

Table F-1 Effect of fly ash on the compressive strength of type 10 cement-based grouts

Initial Water/Solid Ratio	Time (days)	Compressive Strength (MPa)			
		PC	PC + C1	PC + C2	PC + F
0.50					
	0	0	0	0	0
	7	31.3	8.5	11.7	7.7
	14	36	13.8	12.5	9.6
	28	38	17.5	20.2	15.5
	91	42.6	28.9	26	24.6
0.55					
	0	0	0	0	0
	7	26.8	6.1	9.2	6.2
	14	30	6.5	10.8	8.3
	28	33.5	14.3	18.5	12.2
	91	34.4	28.3	20.6	17.4
0.65					
	0	0	0	0	0
	7	15.7	3.7	7.6	3.7
	14	21.3	7.1	12	6.8
	28	26.7	13	14.8	9.2
	91	29.2	18.8	16.1	13.3

APPENDIX G
BOND STRENGTH
(TABLE)

Table G-1 Effect of fly ash on bond strength of type 10 cement based grouts

Initial Water/Solid Ratio	Bond Strength (MPa)							
	14 Day Bond Strength				28 Day Bond Strength			
	PC	PC+C1	PC+C2	PC+F	PC	PC+C1	PC+C2	PC+F
0.50	N/A	10	14	6	N/A	16	18	9
0.55	22	9	13	6	24	16	16	9
0.65	18	8	11	4	20	11	14	7

APPENDIX H

MODULUS OF ELASTICITY AND POISSON'S RATIO
(TABLE)

Table H-1 Effect of fly ash on the modulus of elasticity and Poisson's ratio value of type 10 cement based grouts

Initial Water/Solid Ratio	Modulus of Elasticity (GPa)				Poisson's Ratio			
	PC	PC+C1	PC+C2	PC+F	PC	PC+C1	PC+C2	PC+F
0.50	16	12	12	10	0.25	0.23		0.22
0.55	15	10	11	9	0.24	0.22	0.26	0.25
0.65	13	9	10	8	0.20	0.22	0.23	0.25

APPENDIX I
DRYING-SHRINKAGE
(TABLE)

Table I-1 Drying-shrinkage of type 10 cement-based grouts with and without fly ash

Initial Water/Solids Ratio	Time (days)	% Change in Bar Length			
		PC	PC + C1	PC + C2	PC + F
0.50					
	3	0.0000	0.0000	0.0000	0.0000
	7	-0.0573	-0.0032	-0.0243	-0.0050
	14	-0.0655	-0.0250	-0.0460	-0.0270
	21	-0.0885	-0.0360	-0.0433	-0.0570
	28	-0.0973	-0.0425	-0.0450	-0.0733
	56	-0.1808	-0.1478	-0.1473	-0.2110
	91	-0.3001	-0.2238	-0.2314	-0.2840
	183	-0.4305	-0.2533	-0.3480	-0.3280
0.55					
	3	0.0000	0.0000	0.0000	0.0000
	7	-0.0477	-0.0055	-0.0205	-0.0067
	14	-0.0748	-0.0260	-0.0365	-0.0183
	21	-0.0605	-0.0325	-0.0708	-0.0380
	28	-0.0915	-0.0970	-0.1098	-0.0788
	56	-0.2555	-0.2725	-0.2295	-0.2935
	91	-0.3259	-0.3173	-0.2721	-0.3502
	183	-0.4345	-0.3495	-0.3535	-0.3925
0.65					
	3	0.0000	0.0000	0.0000	0.0000
	7	-0.0213	0.0002	-0.0168	-0.0010
	14	-0.0648	-0.0615	-0.1040	-0.0953
	21	-0.1818	-0.1885	-0.1958	-0.2135
	28	-0.2395	-0.2650	-0.2635	-0.2700
	56	-0.3223	-0.3038	-0.3405	-0.3240
	91	-0.4367	-0.3708	-0.4358	-0.3930
	183	-0.4688	-0.3843	-0.4660	-0.4113

APPENDIX J
DRYING-SHRINKAGE
(GRAPHS)

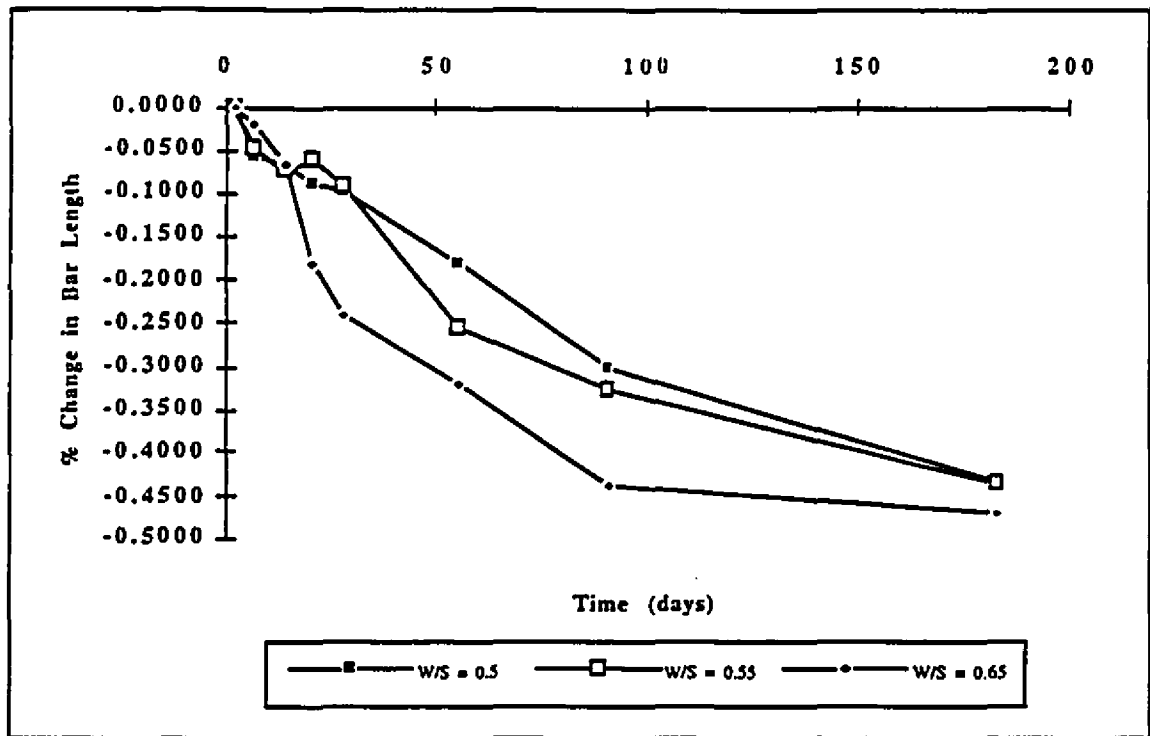


Fig. J-1 Drying-shrinkage of type 10 cement grouts

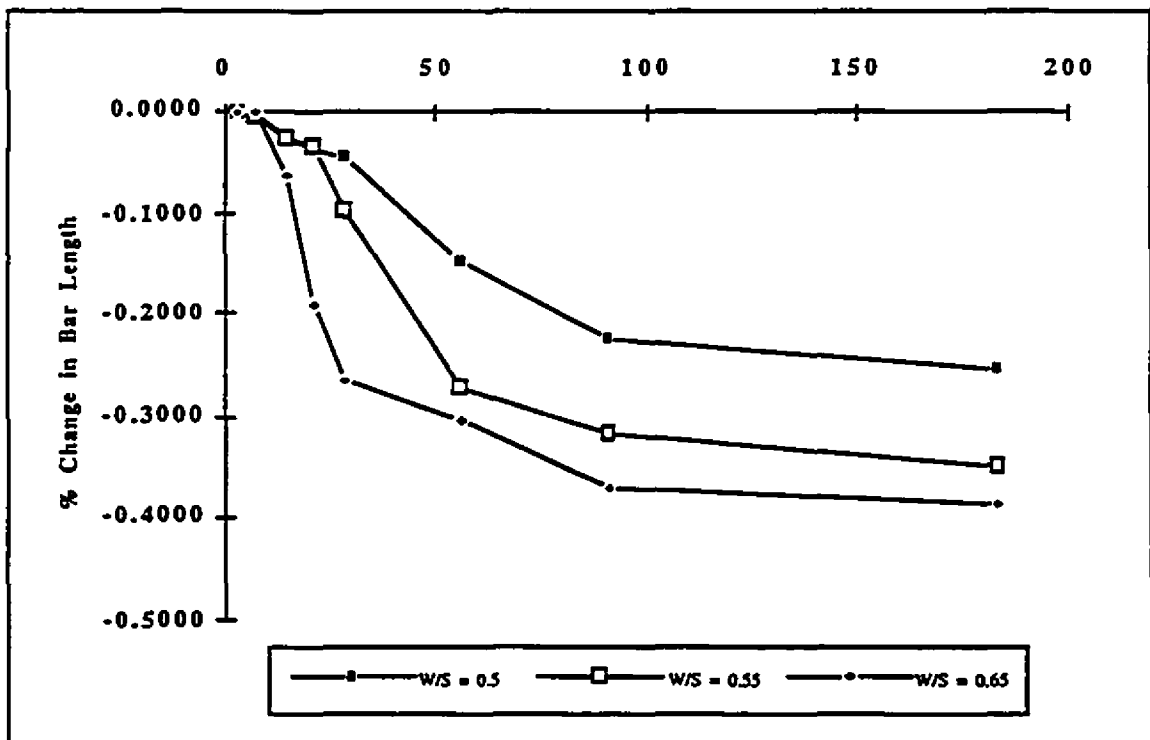


Fig. J-2 Drying-shrinkage of type 10 cement grouts containing C1 fly ash

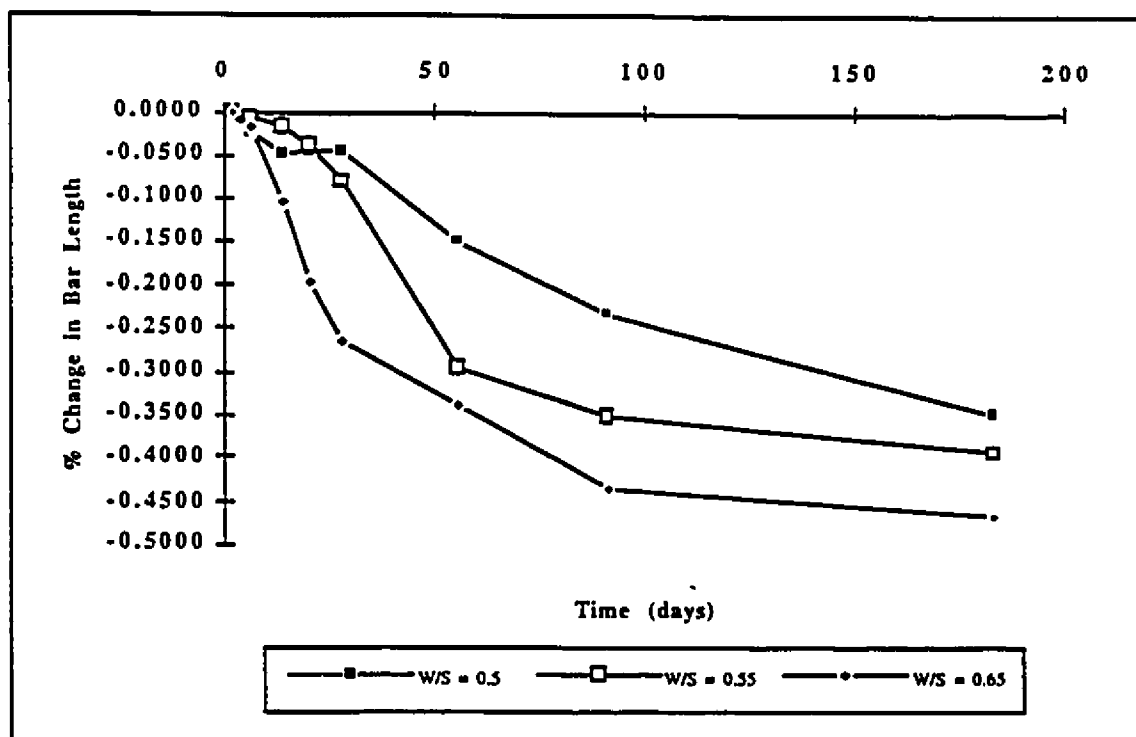


Fig. J-3 Drying-shrinkage of type 10 cement grouts containing C2 fly ash

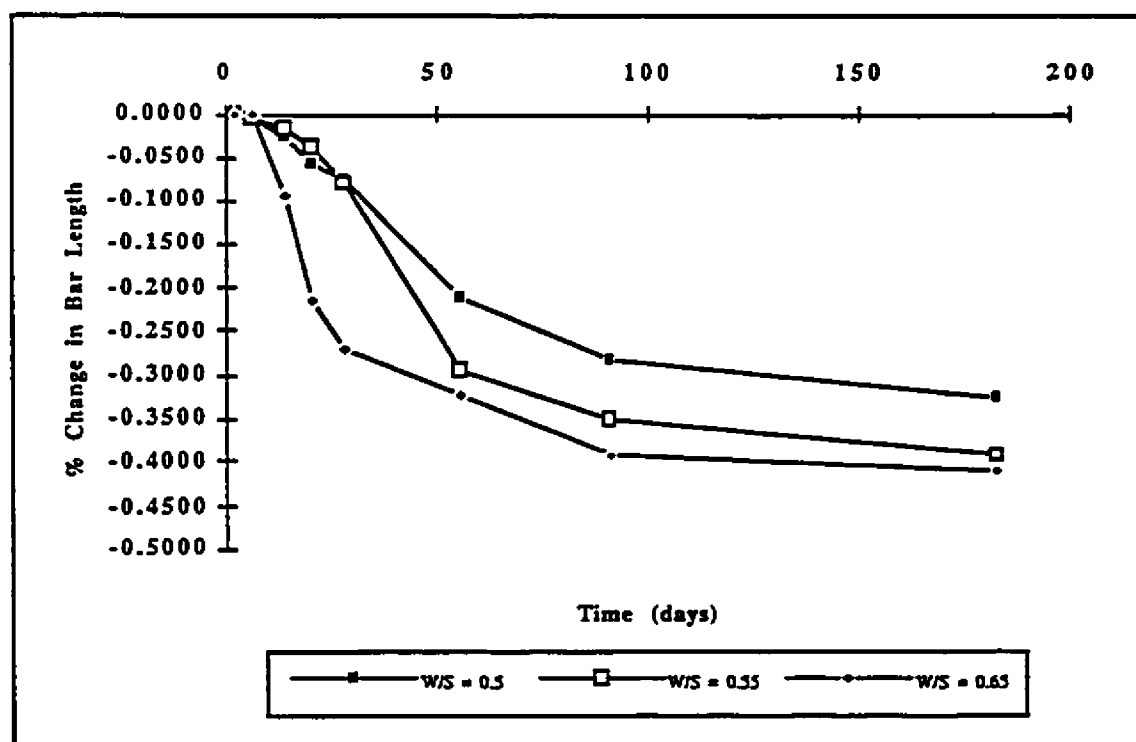


Fig. J-4 Drying-shrinkage of type 10 cement grouts containing F fly ash