CHARACTERIZATION OF CANADIAN FLY ASHES AND THEIR RELATIVE PERFORMANCE IN CONCRETE

G.G. CARETTE and V.M. MALHOTRA

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CHARACTERIZATION OF CANADIAN FLY ASHES AND THEIR RELATIVE PERFORMANCE IN CONCRETE

G.G. Carette* and V.M. Malhotra**

ABSTRACT

Eleven Canadian fly ashes were characterized and evaluated for their relative performance in concrete. Characterization included the determination of mineralogical composition, chemical composition, physical characteristics, and pozzolanic properties. The relative performance of each fly ash in concrete was evaluated through determination of the following properties of fresh and hardened concrete: slump, air content, bleeding, setting time, strength, modulus of elasticity, drying shrinkage, creep, and freezing-and-thawing resistance.

The results indicate a wide range of chemical, physical, and pozzolanic properties for the fly ashes investigated. In spite of this, all the fly ashes evaluated are shown to be suitable for use in concrete. They affect, however, the properties of fresh and hardened concrete in different ways, and this should be taken into account when proportioning concrete containing these fly ashes.

^{*}Materials Engineer and **Head, Construction Materials Section, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada, Ottawa, Canada, KIA OGI.

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CARACTÉRISATION DES CENDRES VOLANTES CANADIENNES ET LEUR PERFORMANCE RELATIVE DANS LE BÉTON

G.G. Carette* et V.M. Malhotra**

RÉSUMÉ

Onze cendres volantes canadiennes ont été caractérisées et évaluées dans le but de déterminer leur performance relative dans le béton. La caractérisation comprenait la détermination de la composition minéralogique et chimique, des caractéristiques physiques et des propriétés pouzzolaniques. La performance relative de chacune des cendres volantes dans le béton a été évaluée en déterminant les propriétés du béton frais et durci, à savoir, l'affaissement, le contenu en air, le ressuage, le temps de prise, la résistance, le module d'élasticité, le retrait au séchage, le fluage et la résistance au gel et au dégel.

Les résultats démontrent la gamme étendue des propriété chimiques, physiques et pouzzolaniques des cendres volantes sur lesquelles a porté la recherche. En dépit des résultats, toutes les cendres volantes étudiées peuvent être utilisées dans le béton. Les cendres volantes peuvent cependant affecter le béton frais et durci de diverses manières et on doit en tenir compte lors du dosage du béton dans lequel elles sont incorporées.

^{*}Ingénieur des matériaux et **Chef, Section des matériaux de construction, Laboratoires des sciences minérales, CANMET, Énergie, Mines et Ressources Canada, Ottawa, Canada, KiA OGI.

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INTRODUCTION

CANMET has an on-going research project dealing with the utilization of supplementary cementing materials in concrete. These materials, which are by-products of mining, metallurgical, and electrical industries, include limestone dust, ferrous and non-ferrous slags, condensed silica fume, and fly ash. The use of these materials in concrete not only helps impart special properties to concrete and reduce the amount of cement requirements but also solves, to a degree, the solid waste disposal problem. A number of research reports have been published on the subject (1-22).

This report is the third in a series on the subject of Canadian fly ashes for use in concrete. The first report of the series was a critical review of the chemical, physical, and pozzolanic properties of fly ash (2); the second report of the series was concerned with a critical evaluation of the published data on the manner in which the inclusion of fly ash affects the mix proportions, the properties of fresh concrete, and the behaviour and durability of hardened concrete (3). This third, and last, report of the series deals with the characterization of Canadian fly ashes and their relative performance in concrete.

SCOPE OF INVESTIGATION

Eleven fiy ashes were investigated in this study. The characterization of the ashes included mineralogical composition, chemical composition, specific gravity, fineness of a 45- μ m sieve, Biaine specific surface area, particle-size distribution, and pozzolanic activity with portland cement and with lime.

The relative performance of each fly ash in concrete was evaluated by incorporating the material as a 20% replacement by mass for cement. Two control and eleven fly ash concrete mixtures were made, each prepared in triplicate batches. The properties of fresh concrete that were determined included slump, air content, bleeding, and setting time. The hardened concrete specimens were subjected to the determination of compressive and flexural strengths, modulus of elasticity, drying shrinkage, creep, and resistance to freezing and thawing.

SOURCES OF FLY ASH

The locations of the sources of the il fly ashes investigated are shown in Figure I. Six ashes (No. i to 6) are derived from bituminous coal, three (No. 7, 8, and 9) are from subbituminous coal, and two (No. 10 and II) are from lignite coal. All sources employ electrostatic precipitators for ash collection, except in one case where mechanical collectors are used. Whenever possible, the samples obtained for the investigation were representative of the material being marketed for use in concrete. Where ash was not being marketed, the sampling was carefully done at the plant to obtain a representative sample.

CHARACTERIZATION OF FLY ASHES

MINERALOGICAL COMPOSITION

Eight of the il fly ashes covering the main source types were examined for their mineralogical composition; their X-ray diffractograms are shown in Figures 2 and 3. A quantitative determination of the major crystalline phases contained in these ashes was also made by a quantitative X-ray diffraction technique (23); the results are given in Table 1, along with calculated values for the glass content. SEM micrographs of a few selected fly ashes are shown in Figures 4(a) and 4(b).

CHEMICAL COMPOSITION

The chemical composition of each fly ash, including major and minor constituents as well as trace elements, is given in Tables 2 and 3. Most constituents were determined by means of inductively coupled argon plasma (ICAP) spectrometry.

PHYSICAL PROPERTIES

SPECIFIC GRAVITY

The specific gravity of fly ash was determined using ASTM C i88 Test Method for Density of Hydraulic Cement. The results are shown in Table 4.

FINENESS BY WET SIEVING ON A 45-um SIEVE

Wet sieving on a 45- μ m sieve was carried out per ASTM method C 430. The percentage residue for each fiy ash after sieve correction factor is

shown in Table 4. The uncorrected percentage residue is also shown in parentheses, since the sieve correction factor is based on a reference cement sample and may not apply exactly for some fly ashes. The correction factor for the sieve used was 16% and, although it had little effect on relatively fine ashes, it did appreciably increase the calculated percentage residue for the coarser ashes.

FINENESS BY DRY SIEVING (ALPINE JET) ON A 45-µm SIEVE

Dry sieving on 45 µm was performed using an Alpine Jet machine, following procedures previously established at CANMET (24). The results of these tests, which are also shown in Table 4, closely reflect those obtained from wet sieving. The dry-sieving results, however, are consistently lower by a difference that is essentially proportional to the percentage of material retained.

BLAINE SPECIFIC SURFACE AREA

The results of Blaine specific surface area, as determined by the air permeability apparatus (ASTM C 204), are given in Table 4.

PARTICLE-SIZE DISTRIBUTION

Three different methods were used to examine the particle-size distribution of fly ash: X-ray sedigraph, coulter counter, and laser particle-size analyzer. The range of particle sizes obtained for the various ashes, using the laser method, is shown in Figure 5. Detailed test results, along with a comparative evaluation of each method, are given elsewhere (25).

POZZOLANIC PROPERTIES

POZZOLANIC ACTIVITY WITH PORTLAND CEMENT

The water requirement and pozzolanic activity with portland cement of the fly ashes were determined using both ASTM C 311 and CSA A23-5 standard test procedures. The two tests are very similar except that the ASTM test involves curing at 38°C for 28 days, whereas the one given in CSA A23-5 is an accelerated test involving curing at 65°C for 7 days. The results of activity tests, including water requirement and activity index, are presented in Table 5. The relationship between the

accelerated 7-day and the 28-day test results is illustrated in Figure 6.

POZZOLANIC ACTIVITY WITH LIME

The pozzolanic activity indices with lime were determined according to ASTM C 311 and the results are also shown in Table 5.

SPECIFICATION REQUIREMENTS

Chemical and physical requirements from both ASTM and CSA specifications on fly ash are shown in Table 6, along with the corresponding values obtained for each fly ash.

CONCRETE MIXES

MATERIALS

CEMENT

Normal portland cement, ASTM Type I, was used. Its physical properties and chemical analysis are given in Table 7.

AGGREGATES

The coarse fraction consisted of 19 mm of crushed limestone and the fine fraction was a local natural sand. To keep the grading uniform for each mixture, both the fine and coarse aggregates were separated into different size fractions that were then recombined to a specific grading.

The specific gravity and absorption of the coarse aggregate were 2.69 and 0.8%, respectively; the corresponding values for the fine aggregate were 2.70 and 1.1%.

AIR-ENTRAINING AGENT

A sulphonated hydrocarbon-type, air-entraining agent was used. Its dosage was adjusted to produce an air content of $6\pm0.5\%$ in all the mixtures.

MIX PROPORTIONING

Concrete mixture proportions are given in Table 8. The two control concrete mixtures were proportioned to have a water-to-cement ratio of 0.50 and a slump of 75 ± 15 mm. The II fly ash

mixtures incorporated 20% fly ash by mass as a partial replacement for cement. The water content of the fly ash mixtures was kept the same as for the control concrete, regardless of any changes in slump. This was done in order to maintain a constant water-to-cementitious-material ratio of 0.50 for all mixtures.

PROPERTIES OF FRESH CONCRETE

All mixtures, including the two control ones, were prepared in triplicate batches to obtain a sufficient number of test specimens for the program requirements. Mixing was carried out in a laboratory counter-current pan mixer, with fly ash being added to the mixer simultaneously with the cement. Total mixing time was six minutes for each mixture. The properties of fresh concrete including unit weight, air content, and slump are given in Table 9, with each value representing the average of three batches. bleeding and setting-time characteristics of concrete were determined from one batch of each mixture. These determinations were made according to ASTM C 232 and ASTM C 403 test procedures. with the exception that the bleeding tests were carried out over a smaller surface of bleeding. The results of both tests are also shown in Table

CASTING AND CURING OF THE TEST SPECIMENS

Fourteen 150- \times 300-mm cylinders and seventeen 90- \times 100- \times 400-mm prisms were cast from the three batches of each mixture. Cylinders were cast in two layers and compacted using an internal vibrator, whereas prisms were cast in two layers and compacted by means of a vibrating table. After casting, the molded specimens were covered with water-saturated burlaps and left in the casting room at 23 \pm 1.7°C for 24 h. They were then demoulded and transferred to a standard moist-curing room until required for testing. The only exceptions were the shrinkage test prisms, which were placed under lime-saturated water at 23 \pm 1.7°C until required for the shrinkage tests.

TESTING OF SPECIMENS

The testing schedule is shown in Table 10. The cylinders cast from the first batch of each mixture were used to determine the compressive strength development of concrete at ages up to 365 days. Test prisms cast from the second batch

were used to determine the flexural strength at ages up to 91 days. From the same batch, prisms were also cast for determining resistance to freezing and thawing. These tests were performed using Procedure A of ASTM C 666, with the specimens being subjected to a total of 500 rapid cycles of freezing and thawing. From the third batch, cylinders were used for the determination of the modulus of elasticity at 28 days and for creep tests; the latter tests were performed after an initial moist-curing period of 91 days. The creep strains were determined over a period of nine months with a constant applied stress of 9.70 MPa; this, in most cases, was equivalent to about 30% of the compressive strength of the concrete at the age of loading. Also, from the third batch, prisms were subjected to drying shrinkage tests after an initial curing period in water of either 7 or 91 days. Shrinkage strains and moisture losses were monitored over a period of 224 days of air-drying at 23 \pm 1.7°C and 50 \pm 4% of relative humidity. For reference purposes, cylinders were tested in compression at 28 days from both the second and third batches. As far as possible, all tests were carried out according to those ASTM standard test procedures indicated in Table 10.

TEST RESULTS

The compressive and flexural strength test results, along with the modulus of elasticity data for the control and fly ash concrete mixtures, are given in Table II. The ranges of the compressive and flexural strengths obtained with the various fly ash concretes at different ages are shown in Figures 7 and 8. A summary of the shrinkage and creep test results is given in Tables I2 and I3, and the data are illustrated in Figures 9 to II. The durability factors and air-void parameters of hardened concrete are presented in Table I4; the freezing and thawing test results are summarized in Table I5. The detailed freezing and thawing test results are given in Appendix A.

DISCUSSION OF TEST RESULTS

CHARACTERIZATION

MINERALOGICAL COMPOSITION

In general, the mineralogical composition of the fly ashes as determined by X-ray diffraction appeared to be influenced by both the type and

source of ash. In addition to a substantial amount of glassy material, each fly ash examined contained one or more of four major crystalline phases, i.e., quartz, mullite, magnetite, and hematite; however, the relative proportion of each phase varied considerably between ashes (Table I). For example, the percentage of mullite was found to vary between 3 and 24% in bituminous ashes, and between 6 and 12% in subbituminous ashes; such a phase was not detected in the lignite ash. Similarly, magnetite varied between 4 and 17% in the bituminous ashes, whereas it was apparently absent in both the subbituminous and lignite ashes. The glass content, which is generally believed to be an important factor as regards pozzolanic activity, was estimated by difference from the total content of all crystalline phases plus that of LOI. The glass content so calculated was found to vary between 55 and 95%, the lowest values being associated with bituminous ashes having a relatively high LOI content and the highest value being obtained with the lignite fly ash.

CHEMICAL COMPOSITION

The chemical test data indicate a large range of compositions for the fly ashes investigated, reflecting the wide variations in the types of coal and conditions of operations between the various coal-power plants across the country. The six ashes of bituminous origin (No. 1 to 6) are shown to have a high content of combined SiO2, Al2O3, and Fe203, as well as relatively low contents of CaO and MgO (Table 2). Ashes No. 3 and 4 are somewhat unusual in that they contain 40% or more of iron. Ashes No. 5 and 6 have a particularly high LOI content, with values of about 7 and 10%, respectively. The total alkali content (as equivalent Na₂O) of the bituminous ashes varies between 1.15 and 2.62%, with the main component being potassium. The three subbituminous ashes (No. 7 to 9) contain considerably less Fe_2O_3 than the bituminous ashes, but they are somewhat richer Their CaO content is also higher, slightly exceeding 10%, and they are sometimes referred to as high-calcium ashes. The total alkali content of these ashes is quite variable, ranging from 0.77 to more than 5.3%, whereas their LOI content is consistently low, with values below the 1% level in all three cases. The two lignite ashes (No. 10 and 11) are characterized by lower

contents of combined SiO₂ + Al₂O₃ + Fe₂O₃, and higher contents of both CaO and MgO as compared with the above two types of ashes. The CaO level is up to about 13% in each case and these ashes are generally referred to as high-calcium fly ashes. Both of these ashes are further characterized by a high content of total alkali (6.8 and 7.8%), with sodium the main component. Whereas the LOI content of either ash is relatively low (with values below 1%), ash No. II presents a peculiar feature in that it contains more than 7.8% of SO_3 , as compared with a maximum value of 1.4% in other ashes. This high value of SO3, however, was later found to reflect some particular conditions of plant operation and ash collection at the time of sampling, and is believed not to be typical of the material currently produced at this source.

As shown in Table 6, all fly ashes meet the chemical requirements of Canadian Standards (CSA A23.5-M82) except for ash No. II which, as used in the investigation, does not conform to the SO₃ specification limit. As for conformance with ASTM C 618, two additional ashes (No. 5 and 6) fail to meet the chemical requirements of this specification because their LOI content exceeds the 6% limit as set in these standards.

PHYSICAL PROPERTIES

Similar to what was observed with chemical composition, the physical characteristics of the fly ashes were found to vary over a wide range. The specific gravity, for instance, ranged from a low value of 1.90 for subbituminous ash No. 7 to a high value of 2.96 for iron-rich bituminous ash No. 4 (Table 4). All three subbituminous ashes had relatively low specific gravity, with values in the neighbourhood of 2.0, which suggests that hollow particles such as cenospheres or plerospheres (Fig. 4(a)) are present in significant proportions in these ashes. The results of fineness by wet sieving also reflected some large variations between the ashes, with values for the uncorrected residue retained on a 45-µm sieve ranging from less than 2.4% for fly ash No. 11 to more than 39.7% for fly ash No. 9; the corresponding values obtained with alpine jet dry sieving were 2.5 and 33.0%, respectively. There was no apparent relationship between the type of ash and its fineness as determined by a $45-\mu \dot{m}$ sieve,

the latter being probably more influenced by factors such as coal combustion, as well as ash collection and classification, than by the nature of the coal itself. Similarly, the type of ash had no apparent influence on the Blaine specific surface area, which was found to vary between a low value of $130 \text{ m}^2/\text{kg}$ for bituminous ash No. 3 and a high value of more than 581 m²/kg for lignite ash No. II. Moreover, except for the latter ash (which was in a fineness range of its own), there appeared to be very little relationship between the Blaine specific surface area and the fineness by a 45-µm sieve. This is probably a result of different patterns in particle-size distribution of the various fly ashes that, though not illustrated here, were found to exist within the range of particle size shown in Figure 5. For example, the per cent volume of material finer than 10 µm, which can be seen to vary between about 15 and 40% (excluding ash No. 11) and which would largely influence the value of specific surface area, was found to bear little relationship to the per cent of material passing the 45 μ_{m} .

The physical requirements of both CSA A23.5 and ASTM C 618 include a maximum value of 34% for the amount of material retained on a $45\text{-}\mu\text{m}$ sieve using wet sieving (Table 6). In this respect, two ashes (No. 6 and 9) failed to meet the specifications.

POZZOLANIC PROPERTIES

The pozzolanic test results indicate that all fly ashes meet the requirements of CSA for pozzolanic activity, although four of them fail to conform with the corresponding requirements of ASTM (Table 6). In the latter case, it is because of either one or both of the activity indices being slightly below the specified minimum value. As shown in Figure 6, there is a high degree of correlation between the results of the accelerated pozzolanic test (curing at 65°C for 7 days) and those of the standard pozzolanic test (curing at 38°C for 28 days), the correlation coefficient being 0.98. This indicates that the former test is a potential candidate for replacing the latter test in ASTM Specification C 618.

No attempt has been made to establish correlations among various characteristics of fly ash, including the pozzolanic activity index. This, though interesting, yields little useful information for the use of fly ash in practice. Similarly, no attempt has been made to correlate activity indices with strength properties of concrete.

PROPERTIES OF FRESH CONCRETE

Fly ash was incorporated into concrete as a direct replacement by mass for part of the portiand cement, while the water-to-cementitious-material ratio of the mixture was kept constant. This was in order to provide for a uniform basis for the comparative evaluation of the pozzolanic properties of the various fly ashes and their performance in concrete. In practice, a different proportioning approach might have to be considered if the use of a given fly ash is to be fully optimized for a given application.

SLUMP

It is often considered that fly ashes, due to characteristics such as particle shape and size distribution, should normally contribute to some reduction in the water requirement of concrete in which they are incorporated as a partial replacement for cement. To some degree, this was found to be true for most of the fly ashes investigated. The use of a fixed quantity of water did generally result in an increased slump of the fly ash mixtures, the increase over the control mixtures varying between 30 and 70 mm (Table 9). The mixtures that did not show such increase were those made with fly ashes No. 5 and 6, for which the incorporation of the ash appeared to have little effect on the slump of concrete. In both cases, however, this lower slump may be explained by the relatively high LOI content of these ashes.

DOSAGE OF AIR-ENTRAINING ADMIXTURE

There were no problems encountered in maintaining an air content of 6 to 6.5% in any of the concrete mixtures. In general, the required dosage of an air-entraining admixture was little affected by the presence of fly ash. There were nevertheless two exceptions, the mixtures made with bituminous ashes No. 5 and 6, for which the admixture requirement was in each case about four times that of the control mixture (Table 8). This can be attributed, once again, to the high LOI or carbon content of these two ashes.

SETTING TIME

Most fly ashes were found to retard the setting time of concrete, the only two exceptions being a subbituminous and a lignite ash (No. 7 and 11, respectively), which had apparently little effect on either the initial or final set. For all other ashes, the increase in setting time generally ranged between 0.5 and 3 h for the initial set, and between I and 4 h for the final set (Table 9). The set retardation is primarily a consequence of the dilution in the cement factor and increase in the water-to-cement ratio resulting from the incorporation of fly ash in the concrete. However, the wide range of retardation observed also suggests that, in several cases, the chemical characteristics of the ash might have been a significant factor in either inhibiting or further aggravating the retardation.

BLEEDING

The results of the bleeding tests were expressed as a total percentage loss per unit of water originally present in the concrete mixtures. the control concrete, the percentage loss was 2.9%; for the fly ash concretes, it varied between 0.6 and 5.6% (Table 9). Although there was no clear pattern in the results, most fly ash concretes had either comparable or slightly higher bleeding losses than the control concrete. The only exception was concrete made with fly ash No. II, which had a lower bleeding rate, probably because of either the relatively high specific surface area or high alkall and sulphur contents of this ash (Tables 2 and 4). For most other fly ash concretes, the slightly increased bleeding, as compared to that of the control concrete, may result from a generally lower specific surface area of fly ash and/or a higher slump of the concrete.

PROPERTIES OF HARDENED CONCRETE

COMPRESSIVE STRENGTH

The compressive strength development of the control and fly ash concretes is illustrated in Figure 7, which shows the range of strength values obtained with the various fly ash concretes at ages up to 365 days, together with the strength of the control concrete. At ages up to 28 days, the strength of the control concrete was nearly

always higher than that of the fly ash concrete. The only case where the strength of the latter was about equal to that of the control within this period was at 28 days for concrete made with lignite fly ash No. II. For the remaining fly ash concretes, the 28-day compressive strengths ranged from about 70 to 95% of that of the control concrete (Table II).

At 9! days, the two lignite as well as two bituminous ash concretes had strengths equal to, or slightly higher than, that of the control concrete; all other ash concretes had strengths still below that of the control. At 365 days, 6 of the II fly ash concretes had reached strengths exceeding that of the control concrete, whereas the remaining ones had strength values that were still lower than that of the control by 5 to 10%.

To some extent, the above results are in agreement with the commonly reported fact that high-calcium fly ashes do contribute to substantial gain in strength at relatively early ages, as opposed to most low-calcium fly ashes whose contribution to strength becomes most significant only after the age of 56 days or more. Two apparent exceptions to this were concretes made with high-calcium fly ashes No. 7 and 9, for which the early strength development was not found to be significantly different from that of most low-calcium fly ash concretes. This, however, is possibly the result of a particularly large amount of coarse material contained in these two ashes, as indicated by the results of fineness tests (Table 4). The highest level of strength reached after one year of moistcuring belonged to a concrete made with a lowcalcium bituminous fly ash with the highest LOI content. From the results, it appears that, in general, the level of compressive strength achieved may be more dependent upon fineness and particle-size distribution of the ash than upon its composition.

FLEXURAL STRENGTH

Similar to what was observed with compressive strength, the flexural strength of the fly ash concretes was consistently lower than that of the control concrete at ages up to 28 days (Fig. 8). The only exception was, once again, concrete made with lignite fly ash No. II, for which the flexural strength was comparable to that of the control concrete at either ages of 14 or 28 days

(Table II). At 91 days, only the lignite ash concrete referred to above and one subbituminous ash had a flexural strength value slightly exceeding that of the control concrete. For all other ash concretes, the 91-day flexural strength was still below that of the control concrete by about 5 to 10%.

YOUNG'S MODULUS OF ELASTICITY

The 28-day Young's modulus of elasticity values for the fly ash concretes varied between 29.0 and 35.8 GPa as compared with a value of 33.5 GPa for the control concrete (Table II). The data provide no clear indication of significant effect of fly ash or type of fly ash on the modulus of elasticity. The effect, if any, may be masked by the variability of the test.

DRYING SHRINKAGE

Moisture losses and drying shrinkage strains were determined over a period of 224 days, following an initial curing period in lime-saturated water of either 7 or 91 days.

For concrete initially cured for 7 days, the total moisture loss of the fly ash concretes varied between 49.5 and 64.3%, as compared with 55.0% for control concrete. For concrete Initially cured for 91 days, the corresponding range was between 45.4 and 56.3%, with a value of 53.7% for the control concrete. In both cases, there is no clear indication as to whether fly ash might have contributed to variations in moisture loss.

On the other hand, the drying shrinkage strain for concrete initially cured for 7 days in water was apparently little affected by the presence of fly ash in the concrete, the strain values for the control and most fly ash concretes being closely grouped together till after 224 days of drying (Fig. 9). The only significant deviation from this was for concrete made with lignite fly ash No. 10, in which case the total shrinkage strain was found to exceed the average strain by about 50%. For concrete initially cured for 91 days, the pattern was somewhat similar to that observed for concrete initially cured for 7 days except that, in this case, the total shrinkage strain of most fly ash concretes was slightly lower than that of the control concrete (Fig. 10).

CREEP

The creep strain data for control and fly ash concretes are illustrated in Figure II. All fly ash concretes are shown to produce consistently lower creep strains than the control concrete. The strain reduction, which in most cases varies between a value of 20 and 45%, does not appear to be related to the type of ash. The effect of fly ash on creep of concrete is somewhat parallel to that observed for drying shrinkage of concrete initially cured for a period of 91 days. In both situations, the lower strains of the fly ash concretes are possibly a result of a relatively large portion of the ash still remaining unreacted at this age and thus acting as an aggregate material that provides increased restraint.

RESISTANCE TO FREEZING AND THAWING

Freezing-and-thawing tests* were initiated at the age of 14 days for all concretes, and this implies that the strength of many of the fly ash concrete specimens was still significantly lower than that of the control specimens at the age of test. Despite this, all concretes regardless of strength performed equally well under the action of rapid freezing-and-thawing cycles, the durability factor ranging between 95.8 and 98.8% in all cases after 300 cycles (Table 14). This excellent resistance to freezing-and-thawing cycling of both control and fly ash concretes was further confirmed by the relatively small changes recorded in the weight, length, pulse velocity, and resonant frequency measurements as well as the visually unchanged condition of all specimens after an exposure to more than 500 cycles (Table 15). Also, after such exposure, the residual strengths of the control and fly ash concrete specimens were of the same order. Though, in each case, they might appear to indicate a substantial loss of strength of the concrete during exposure, this is actually a result of these residual strengths being expressed as a percentage of the strength of companion moistcured concrete for which maturity is obviously totally different. The behaviour of both control and fly ash concrete specimens, under freezing-and-thawing cycling is undoubtediy the result of an adequate air-void system of the

^{*}ASTM Standard C 666 Procedure A, freezing in water and thawing in water.

concrete in each case, as shown in Table 14, and the fact that concrete had compressive strength of not less than about 20 MPa at the age of cycling (Fig. 7). The spacing factors vary between 0.09 and 0.13 mm for all specimens, which is well below the value of 0.20 mm that is generally agreed upon as being a reasonable limit to ensure frost-resistant concrete (26). From the results, it is clear that the spacing factor is essentially not affected by the presence of fly ash, provided that the dosage of air-entraining admixture is properly adjusted to achieve the same level of air content.

CONCLUSIONS

The physical, mineralogical, chemical, and pozzolanic properties of the fly ashes investigated in this study vary over a wide range, but this is no different from the variability of available fly ashes from other countries. Notwithstanding the above, all the fly ashes investigated are suitable for use in concrete and some of these are already being marketed. Because each fly ash is unique, the users are advised to carry out a trial mixture program to develop proper mixture proportions and water requirements for the fly ash under study. In general, the water demand of fly ash concretes, as evidenced by the increase in the slump of fly ash concretes, is lower than that of control concrete; however, there are exceptions as noted in the report.

The strength properties of fly ash concretes show a gain with time but, even at 365 days, the compressive strengths of some of the fly ash concretes do not reach levels achieved by the control concrete. This is significant and should be taken into account when proportioning fly ash concretes.

All fly ash concretes show satisfactory performance in rapid freezing-and-thawing tests (ASTM Standard C 666 Procedure A). This implies that if concrete is satisfactorily air-entrained and has at least 20 MPa compressive strength at the time of the commencement of the test, the incorporation of fly ash does not adversely affect its resistance to frost action. Also, with one exception, all fly ash concretes exhibit drying shrinkage and creep strains that are either comparable to, or smaller than, those of the control concrete.

The test results reported and the conclusions drawn are for the fly ash samples tested in this investigation and the type of cement used. They may, or may not, be applicable to other types of fly ashes and cements.

REFERENCES

- Malhotra, V.M.; Berry, E.E.; and Wheat, T.A., Editors. Proceedings, Seminar on Energy and Resources Conservation in the Cement and Concrete Industry; CANMET, Energy, Mines and Resources Canada; 1976.
- Berry, E.E. "Fly ash for use in concrete. Part 1 - A critical review of the chemical, physical and pozzolanic properties of fly ash"; CANMET Report 76-25; CANMET, Energy, Mines and Resources Canada; 60 pp.; 1976.
- Berry, E.E., and Malhotra, V.M. "Fly ash for use in concrete - A critical review"; ACI Journal, Proceedings 77:2:59-73; 1980.
- 4. Malhotra, V.M. "Strength and durability characteristics of concrete incorporating a pelletized blast-furnace slag"; ACI Special Publication SP 79 2:891-922; Editor, V.M. Malhotra; 1983.
- Malhotra, V.M.; Carette, G.G.; and Bremner, T.W. "Durability of concrete in marine environment containing granulated blast furnace slag or fly ash or both"; ACI Special Publication SP 651:157-168; Editor, V.M. Malhotra; 1980.
- 6. Mukherjee, P.K.; Loughborough, M.T.; and Malhotra, V.M. "Development of high-strength concrete incorporating a large percentage of fly ash and superplasticizers"; ASTM Cement, Concrete and Aggregates 4:2:81-86; 1983.
- Carette, G.G., and Malhotra, V.M. "Mechanical properties, durability and drying shrinkage of portland cement concrete incorporating silica fume"; ASTM Cement, Concrete and Aggregates 5:1:3-13; 1983.
- 8. Malhotra, V.M., and Carette, G.G. "Silica fume Its use in concrete"; Concrete Construction 27:5:443-446; Chicago, U.S.A.; May 1982.
- 9. Malhotra, V.M., and Carette, G.G. "Silica fume concrete - Properties, applications and limitations"; ACI Concrete International: Design and Construction 5:5:40-46; May 1983.
- 10. Carette, G.G., and Malhotra, V.M. "Early-age strength development of concrete incorporating fly ash and condensed silica fume"; ACI Special Publication SP 79 2:765-786; Editor, V.M. Malhotra; 1984.
- 11. Carette, G.G.; Malhotra, V.M.; and Aitein, P.C. "Mechanical properties of portland cement concrete incorporating blast-furnace slag and condensed silica fume"; Division Report MRP/MSL 84-73 (OP&J); CANMET, Energy, Mines and Resources Canada; 21 pp.; 1984.
- 12. Malhotra, V.M. "Mechanical properties and freezing and thawing resistance of non airentrained, air-entrained, and air-entrained superplasticized concrete using ASTM Test C 666, Procedures A and B"; ASTM Cement, Concrete and Aggregates 4:1:3-23; 1982.
- 13. Carette, G.G.; Wilson, H.S.; and Tan, C.W.
 "Development of super-plasticized lightweight
 high-strength concrete incorporating steel

- fibres, silica fume and fly ash"; Division Report MRP/MSL 84-81 (IR); CANMET, Energy, Mines and Resources Canada; 25 pp.; 1984.
- 14. Nebesar, B., and Carette, G.G. "Variations in the chemical composition, specific surface area, fineness and pozzolanic activity of a condensed silica fume"; Division Report MRP/MSL 84-126 (J); CANMET, Energy, Mines and Resources Canada; 13 pp.; September 1984.
- 15. Malhotra, V.M. "Mechanical properties and freezing and thawing resistance of non airentrained and air-entrained condensed silica-fume concrete using ASTM Test C 666, Procedures A and B"; ACI Special Publication SP 91 2:1069-1093; Editor, V.M. Malhotra; 1986.
- 16. Berry, E.E., and Malhotra, V.M. "Fly ash in concrete"; Division Report MRP/MSL 84-156 (TR); CANMET, Energy, Mines and Resources Canada; 247 pp.; December 1984.
- 17. Berry, E.E., and Malhotra, V.M. "Compilation of Abstracts of papers from recent international conferences and symposia on fly ash in concrete"; 60 pp.; January 1985; Available from the authors at CANMET, Energy, Mines and Resources Canada, Ottawa.
- 18. Carette, G.G.; Malhotra, V.M.; Bédard, C.; De Benedictis, V.; and Plumat, M. "Development of heat-curing cycles for portland cement/fly ash concrete for the precast industry"; ACT Special Publication SP 91 1:249-272; Editor, V.M. Malhotra; 1986.
- 19. Douglas, E., and Mainwaring, P.R. "Hydration and pozzolanic activity of non-ferrous slags"; American Ceramic Society Bulletin 64:5:700-706; 1985.

- 20. Douglas, E.; Malhotra, V.M.; and Emery, J.J.
 "Cementitious properties of non-ferrous slags
 from Canadian sources"; ASTM Cement, Concrete
 and Aggregates 7:1:3-14; Summer, 1985.
- 21. Douglas, E.; Mainwaring, P.R.; and Hemmings, R.T. "Pozzolanic properties of Canadian non-ferrous slags"; ACI Special Publication SP 91 2:1525-1551; Editor, V.M. Malhotra; 1986.
- 22. Douglas, E., and Malhotra, V.M. "A review of the properties and strength development of non-ferrous slags - portland cement binders"; CANMET Report 85-7E; CANMET, Energy, Mines and Resources Canada; 1986.
- 23. van Roode, M., and Hemmings, R.T. CANMET Contract Report No. 1SQ83-00162; Energy, Mines and Resources Canada; 285 pp.; July 1985.
- 24. Malhotra, V.M., and Wallace, G.G. "A new method for determining fineness of cement"; Mines Branch Investigation Report (IR) 63-119; Energy, Mines and Resources Canada; 12 pp.; December 1963.
- 25. Zerbino, R.; Carette, G.G.; and Malhotra, V.M. "Fineness and Particle Size Determination of Fly Ashes"; Division Report MRP/MSL 86-9 (IR), CANMET, Energy, Mines and Resources Canada; Ottawa, 1986.
- 26. ACI Manual of Concrete Practice, Part 1; American Concrete Institute; Detroit, U.S.A.; 1985.

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TABLES

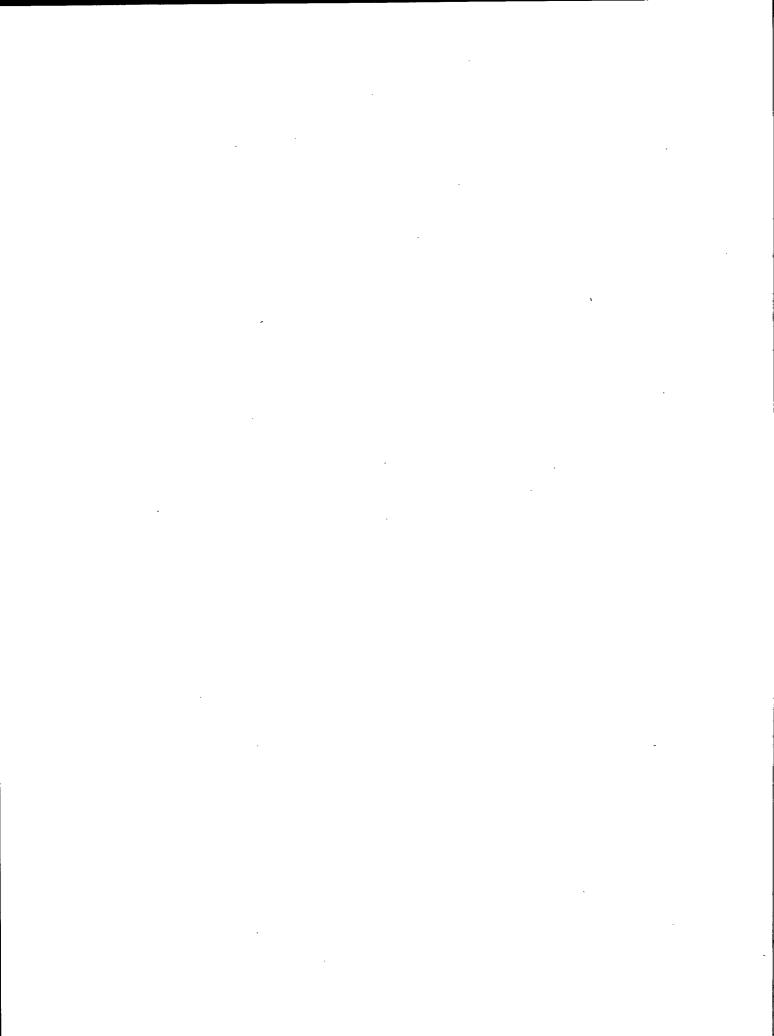


Table 1 - Mineralogical composition of some selected fly ashes

Fly ash	Type of		Phase	compositio	on, per cent		Loss on
source	coal*	Glass	Quartz	Mullite	Magnetite	Hematite	ignition, %
1 4 5 6 7 8	B B B SB SB SB	72.1 70.1 55.6 54.2 90.2 83.9 79.8	4.0 3.2 6.2 8.3 2.9 4.1 8.7	12.6 3.3 19.8 23.5 6.1 10.2 11.5	6.2 17.2 5.6 4.4 	1.6 4.7 3.1 2.1 1.4	3.5 1.5 9.7 7.5 0.8 0.4 0.8
10	L	94.5	4.6				0.9

^{*}B: Bituminous; SB: Subbituminous; L: Lignite.

Table 2 - Chemical composition of fly ashes - major and minor elements

Fly ash	Type of	Chemical composition**, weight per cent												
source	coal*	SiO ₂	A1 ₂ 0 ₃	Fe ₂ 0 ₃	Ca0	Mg0	Na ₂ 0	к ₂ 0	T102	P ₂ 0 ₅	Mn0	Ba0	S0 ₃	T01***
1	В	47.1	23.0	20.4	1.21	1.17	0.54	3.16	0.85	0.16	0.78	0.07	0.67	2.88
2	В	44.1	21.4	26.8	1.95	0.99	0.56	2.32	0.80	0.27	0.12	0.07	0.96	0.70
3	В	35.5	12.5	44.7	1.89	0.63	0.10	1.75	0.56	0.59	0.12	0.04	0.75	0.75
4	В	38.3	12.8	39.7	4.49	0.43	0.14	1.54	0.59	1.54	0.20	0.04	1.34	0.88
5	В	45.1	22.2	15.7	3.77	0.91	0.58	1.52	0.98	0.32	0.32	0.12	1.40	9.72
6	В	48.0	21.5	10.6	6.72	0.96	0.56	0.86	0.91	0.26	0.36	0.21	0.52	6.89
7	SB	55.7	20.4	4.61	10.7	1.53	4.65	1.00	0.43	0.41	0.50	0.75	0.38	0.44
8	SB	55.6	23.1	3.48	12.3	1.21	1.67	0.50	0.64	0.13	0.56	0.47	0.30	0.29
9	SB	62.1	21.4	2.99	11.0	1.76	0.30	0.72	0.65	0.10	0.69	0.33	0.16	0.70
10	L	46.3	22.1	3.10	13.3	3.11	7.30	0.78	0.78	0.44	0.13	1.18	0.80	0.65
11	L [44.5	21.1	3.38	12.9	3.10	6.25	0.80	0.94	0.66	0.17	1.22	7.81	0.82

^{*} B: Bituminous; SB: Subbituminous; L: Lignite.

^{**} By Inductively Coupled Argon Plasma (ICAP) Technique, except for Na $_2^{\rm O}$, K $_2^{\rm O}$, SO $_3$, and LOI.

^{***} Between 105 and 750°C.

Table 3 - Chemical composition of fly ashes - trace elements

Fly ash	Type of		Chemical composition**, ppm												
source	Type of coal*	Cu	Pb	Zn	Ni	Co	Ве	Cd	Мо	Cr	Sr	Th	Zr	V	Ag
1	В	106	198	349	98	12	9	<1	40	110	344	54	135	200	<.5
2	В	128	295	698	295	15	9	2	110	132	526	57	142	215	<.5
3	В	123	< 5	121	76	<5	8	<1	150	57	213	3 9	115	185	<.5
4	В	157	38	155	73	< 5	11	<1	215	56	295	46	99	146	<.5
5	В	103	48	162	106	18	13	<1	30	139	1225	56	137	219	<.5
6	В	69	40	87	58	9	8	<1	35	60	992	54	87	142	<.5
7	SB	28	68	77	50	< 5	6	2	60	3 0	2185	40	192	56	<.5
8	SB	37	73	71	40	<5	6	<1	55	3 0	1085	45	3 09	62	<.5
9	SB	35	40	58	44	<5	5	<1	45	35	738	42	264	81	<.5
10	L	52	58	48	36	<5	6	<1	35	41	3520	26	267	78	<.5
11	L	73	83	120	41	< 5	7	<1	55	56	3435	27	250	125	<.5

^{*} B: Bituminous; SB: Subbituminous; L: Lignite.

^{**} By Inductively Coupled Argon Plasma (ICAP) Technique.

Table 4 - Physical properties of fly ashes

			Physical p	properties	
Fly ash	Type of	Specific gravity	Fineness, % ret	tained on 45 µm	Blaine specific surface
source	coal*	(Le Chatelier Method)	Wet sieving**	Dry sieving (Alpine jet)	area, m ² /kg
1	В	2.53	17.3 (14.9)	12.3	289
2 3	В	2.58	14.7 (12.7)	10.2	31.2
	В	2.88	25.2 (21.7)	18.0	127
4	В	2.96	19.2 (16.6)	14.0	198
5	В	2.38	21.2 (18.3)	16.1	448
6	В	2.22	40.7 (35.1)	30.3	303
7	SB	1.90	33.2 (28.7)	26.4	215
8	SB	2.05	19.4 (16.7)	14.3	326
9	SB	2.11	46.0 (39.7)	33.0	240
10	L	2.38	24.9 (21.5)	18.8	286
11	L	2.53	2.7 (2.4)	2.5	581

^{*} B: Bituminous; SB: Subbituminous; L: Lignite.

^{**} Values in parentheses do not include sieve correction factor.

Table 5 - Pozzolanic properties of fly ashes

		Pozzolanic a	activity with Por	tland cement **	
Fly ash source	Type of coal*	Water requirement,	Activity index at 28 days,	Accelerated activity index at 7 days,	Pozzolanic activity index with lime at 7 days, MPa
1	В	92	98.2	90.1	6.8
2	В	92	100.0	91.5	6.3
3	В	92	73.2	7 1.7	5.5
4	В	92	92.3	85.6	8.8
5	В	1 00	93.7	87.7	7.6
6	В	104	73.7	71.2	4.5
7	SB	94	73. 3	68.1	4.3
8	SB	92	94.6	85.5	6.3
9	SB	97	77.0	69.3	4.9
10	L	92	86.4	87.3	6.8
11	L	88	132.6	130.7	16.3

^{*} B: Bituminous; SB: Subbituminous; L: Lignite.

^{**} Cement used had composition and properties similar to those shown in Table 7

Table 6 - Compliance of Canadian fly ashes with ASTM and CSA specification requirements

	ASTM C 618-84	CSA A23.5-M82					F	ly Ash	No.				
	requirements*	requirements*	1	2	3	4	5	6	7	8	9	10	11
Chemical Requirements													
$- (SiO_2 + Al_2O_3 + Fe_2O_3), %$	Min 70**		90.5	92.3	92.7	90.8	83.0	80.1	80.7	82.2	86.5	71.5	69.0
- so ₃ , %	Max 5.0	Max 5.0	0.67	0.96	0.75	1.34	1.40	0.52	0.38	0.30	0.16	0.80	7.81
- 1.01, %	Max 6.0	Max 12.0***	2.88	0.70	0.75	0.88	9.72	6.89	0.44	0.29	0.70	0.65	0.82
Physical Requirements													
- % Retained on 45 µm by wet sieving	Max 34	Мах 34	17.3	14.7	25.2	19.2	21.2	40.7	33.2	19.4	46.0	24.9	2.7
- Water requirement, %	Max 105		92	92	92	92	100	104	94	92	97	92	88
- Pozzolanic activity index with Portland cement, %													
- CSA accelerated at 7 days		Min 68	90.1	91.5	71.7	85.6	87.7	71.2	68.1	85.5	69.3	87.3	130.7
- ASTM at 28 days	Min 75		98.2	100.0	73.2	92.3	93.7	73.7	73.3	94.6	77.0	86.4	132.6
- Pozzolanic activity with lime, MPa	Min 5.5		6.8	6.3	5.5	8.8	7.6	4.5	4.3	6.3	4.9	6.8	16.3

^{*} For both Class F and C fly ashes unless otherwise indicated.

^{**} Min 60 for Class C.

^{***} Max 6.0 for Class C.

Table 7 - Physical properties and chemical analysis of cement*

Physical propert	ies	Chemical analys	sis	
Fineness				
- Passing 75-µm sieve		Silicon dioxide		22.02%
- Passing 45-µm sieve		Calcium oxide (total)		
- Blaine	: 363 m ² /kg	Alumina		3.99%
		Ferric oxide	:	2.76%
Normal consistency	: 22.0%	Magnesia	:	3.30%
		Sulphur trioxide	:	3.01%
Vicat setting time		Sodium oxide	:	0.49%
		Potassium oxide	:	0.54%
- Initial	: 140 min	Loss on ignition	:	1.59%
- Final	: 230 min	Insoluble residue	:	0.14%
Autoclave expansion	: 0.09%	Compound composition		
Compressive strength of		C3S	•	48.8%
50-mm mortar cubes		C ₂ S	•	26.3%
(W/C:0.485;Flow:127%)		C_3^2 A		5.9%
(11,000,400,110,1127/8)		C ₄ AF		8.4%
- 3 days	: 20.8 MPa	1 04	•	0 1 1/0
	: 25.2 MPa			
- 28 days	: 33.2 MPa			

^{*} Data supplied by manufacturer.

Table 8 - Concrete mixture proportions

				Bato	ch quantiti	ies, kg	/m ³	
Mixture No.	Fly ash source	Replacement of cement by fly ash, % by weight	W/(C+F)*	Cement	Fly ash	Fine agg.	Coarse agg.	A.E.A., mL/m ³
Control 1		0	0.50	297		791	1094	200
Control 2	_	ő	0.50	295		782	1082	170
F1	1.	20	0.50	236	59	780	1.077	320
F2	1 2 3	20	0.50	237	59	782	1080	200
F3	3	20	0.50	237	59	786	1088	200
F4	4	20	0.50	238	59	792	1094	160
F5	5	20	0.50	237	59	782	1080	690
F6	6	20	0.50	238	59	784	1082	660
F7	7	20	0.50	239	59	780	1077	370
F8	8	20	0.50	236	59	775	1069	230
F9	9	20	0.50	236	59	775	1070	240
F10	1.0	20	0.50	237	59	781	1079	290
F11	1.1.	20	0.50	237	59	782	1080	150

^{*} Water/(cement + fly ash) by weight.

Note: Control mixture No. 1 was made at the start of the mixing program (Feb. 1984) and control mixture No. 2 was made at the end of the program (April 1984).

Table 9 - Properties of fresh concrete

		Properties of fresh concrete							
Mixture	W/(C+F)*	Unit weight,	Slump,	Air	Bleeding,	Setting time, h:min			
No.		kg/m ³	mm	content,	%	Initial	Final		
Control 1	0.50	2320	70	6.5	*** ***				
Control 2	0.50	2320	70	6.4	2.9	4:10	6:00		
F1	0.50	2300	100	6.2	3.1	4:50	8:00		
F2	0.50	2310	105	6.2	4.6	7:15	10:15		
F3	0.50	2310	100	6.2	5.1	5:20	8:10		
F4	0.50	2320	110	6.3	4.3	6:20	8:25		
F5	0.50	2310	65	6.4	2.7	5:15	8:55		
F6	0.50	2300	75	6.5	2.6	4:30	6:50		
F7	0.50	2300	100	6.1	2.9	4:15	6:20		
F8	0.50	2300	115	6.2	5.6	5:10	7:30		
F9	0.50	2280	100	6.4	4.4	5:25	9:00		
F10	0.50	2290	130	6.5	2.5	4:45	7:00		
F11	0.50	2290	140	6.6	0.6	4:00	6:05		

^{*} Water/(cement + fly ash) by weight.

Note: The values for the unit weight, slump, and air content are the averages of three batches. The bleeding and setting time values are for one batch only.

Table 10 - Testing schedule for hardened concrete

Batch	Type of		Age of	f testing, day	s	
No.	testing	7	14	28	91	365
1	Compression (ASTM C 39)	2 cylinders	2 cylinders	2 cylinders	2 cylinders	2 cylinders
2	Flexure (ASTM C 78)		3 prisms	3 prisms	3 prisms	
	Freezing and thawing (ASTM C 666)	_	ere exposed to E 14 days of mo		es of freezing	and thawing
	Compression (ASTM C 39)			2 cylinders		
3	Modulus of elasticity (ASTM C 469)			2 cylinders		
	Creep (ASTM C 512)	Four cylinder moist-curing	rs were tested	for creep at	the end of 91 o	lays of
	Drying shrinkage (ASTM C 157)		ere exposed to end of 7 and 91			er cent R.H.,
	Compression (ASTM C 39)			2 cylinders		

Note: For each mix, three concrete batches were made to obtain all the specimens needed.

Table 11 - Summary of compressive and flexural strength, and Young's modulus of elasticity test results

Mixture	Compressive strength* of 150 x 300-mm cylinders, MPa				Flexural strength** of 75 x 100 x 400-mm prisms, MPa			Modulus of elasticity* of 150 x 300-mm cylinders, GPa
No.	7-day	28-day	91-day	365-day	14-day	28-day	91-day	28-day
Control 1 Control 2 F1 F2 F3 F4 F5 F6 F7 F8 F9 F10 F11	23.4 22.1 18.4 16.9 14.4 17.8 20.1 18.4 16.7 17.9 16.7 19.2 21.1	30.6 28.6 25.7 25.2 21.0 23.3 28.0 24.8 24.1 27.7 24.9 28.5 29.4	34.9 32.5 31.4 34.8 27.6 32.3 33.9 31.8 29.1 29.0 31.1 33.7 35.3	39.2 36.5 38.3 37.0 34.4 36.9 44.3 39.2 35.7 40.4 35.6 39.7 40.1	4.9 4.3 4.4 3.9 4.0 4.1 3.5 3.5 3.9 4.6 4.3 4.1 4.8	5.4 4.7 4.4 4.8 5.0 4.4 4.6 4.5 5.0 4.2 5.1 5.3	5.9 5.9 5.4 5.5 5.3 5.2 5.3 5.6 5.4 6.1 5.7 5.8 6.6	33.5 33.0 30.2 30.0 31.5 33.0 29.0 33.0 34.1 35.8 31.3 32.9

^{*} Each value is the average of two tests.

^{**} Each value is the average of three tests.

Table 12 - Summary of shrinkage test results

	Duration of drying, days	Shrinkage measurements						
			ally cured for mys in water	Initially cured for 91 days in water				
Mixture No.		Moisture* loss, %	Drying shrinkage, X10 ⁻⁶	Moisture*	Drying shrinkage, X10 ⁻⁶			
Control 2	224	55.0	422	53.7	453			
F1	224	57.5	447	47.9	365			
F2	224	57.3	364	45.4	280			
F3	224	56.9	411	56.2	405			
F4	224	54.7	379	49.2	387			
F5	224	58.8	404	51.1	403			
F6	224	60.6	475	56.4	454			
F7	224	64.3	397	54.1	433			
F8	224	56.3	400		327			
F9	224	58.2	390	49.3	361			
F10	224	58.4	642	55.2	500			
F11 .	224	49.5	454	48.9	362			

^{*}As a percentage of total original water.

Table 13 - Summary of creep test results

Mixture No.	Age at loading, days	Applied stress, MPa	Stress/ strength ratio*, %	Duration of loading, days	Initial elastic strain, X10-6	Creep strain**, XlO ⁻⁶
Control 1	91	9.70	28	259	269	585
Control 2	91	"	30	244	275	562
F1	91	"	31	255	248	357
F2	90	"	28	255	247	331
F3	91	**	35	262	299	537
F4	90	**	30	254	273	458
F5	99	"	29	260	255	367
F6	91	"	31	264	252	492
F7	90	"	33	260	263	404
F8	90	"	33	258	237	320
F9	91		31	252	237	36 0
F10	91	"	29	239	262	544
F11	91	"	27	254	264	367

^{*}Applied stress as a percentage of compressive strength of companion specimens at time of loading.

^{**}Creep strain = total load-induced strain - initial elastic strain.

Table 14 - Durability factors and air-void parameters of hardened concrete for control and fly ash mixtures

Mixture No.	Durability factor after 300 cycles, %*	Air content,**	Specific surface (a), mm ⁻¹	Spacing factor (\bar{L}) , mm
Control 1	97.7	7.2 (6.5)	22.3	0.135
Control 2	98.1	7.2 (6.4)	25.2	0.108
F1	96.4	8.0 (6.2)	20.1	0.132
F2	98.8	8.4 (6.2)	20.4	0.124
F 3	96.8	8.3 (6.2)	23.4	0.108
F4 '	98.8	8.7 (6.3)	19.5	0.108
F5	97.2	7.9 (6.4)	26.1	0.104
F6	96.8	- (6.5)	38.7	0.105
F7	97.6	7.0 (6.1)	27.2	0.110
F8	96.9	7.1 (6.2)	23.5	0.109
F9	97.6	9.1 (6.4)	20.0	0.121
F10	97.2	7.5 (6.5)	34.8	0.094
F11	95.8	7.6 (6.6)	26.5	0.108

^{*}Determined in accordance with ASTM C 666 (Procedure A).

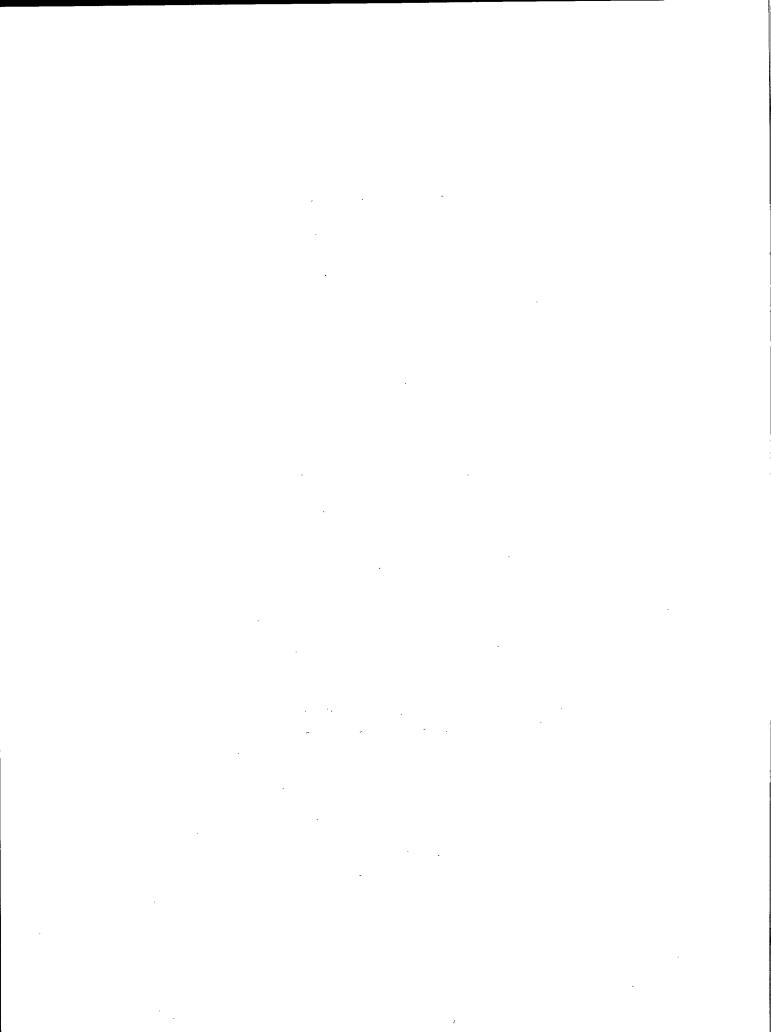
^{**}Values in parentheses refer to the air content of fresh concrete.

Table 15 - Summary of results of freezing and thawing tests

			Summary of test results after 500 cycles of freezing and thawing							
Mixture No.	W/(C+F)*	Air content,	Weight change,	Length change,	Pulse Velocity change, %	Fundamental longitudinal frequency change, %	Relative dynamic modulus, %	Residual flexural strength,**	Residual modified cube strength,**	
Control 1	0.50	6.5	-0.44	+0.007	0.0	-0.9	98		83	
Control 2	0.50	6.4	+0.06		+1.5	-0.4	99	70	72	
F1	0.50	6.2	+0.06	+0.006	+2.8	-1.4	97	[74	
F2	0.50	6.2	-0.39	+0.016	-0.2	-1.4	97	59	80	
F3	0.50	6.2	-0.37	+0.006	+0.9	-1.4	97	73	69	
F4	0.50.	6.3	-0.88	+0.003	+1.6	-0.4	99 98	82	65	
F5	0.50	6.4	-0.22	+0.018	+0.9	-1.0	98	71	76	
F6	0.50	6.5	-0.99	+0.018	+2.0	-1.2	98	64	74	
F7	0.50	6.1	-0.49		+2.1	-0.4	99 97	67	70	
F8	0.50	6.2	-0.19	-0.002	0.0	-1.4	97	71	72	
F9	0.50	6.4	-0.40	+0.011	+0.2	-1.2	98 97 97	70	75	
F10	0.50	6.5	-0.38	+0.021	+2.0	-1.4	97	64	74	
F11	0.50	6.6	-0.26	+0.015	-3.0	-1.7	97	66	76	

^{*}Water/(cement + fly ash) by weight.

^{**}This value is a percentage of the strength of companion test prisms that had been moist-cured for a period equivalent to the completion of 500 cycles of freezing and thawing.



FIGURES

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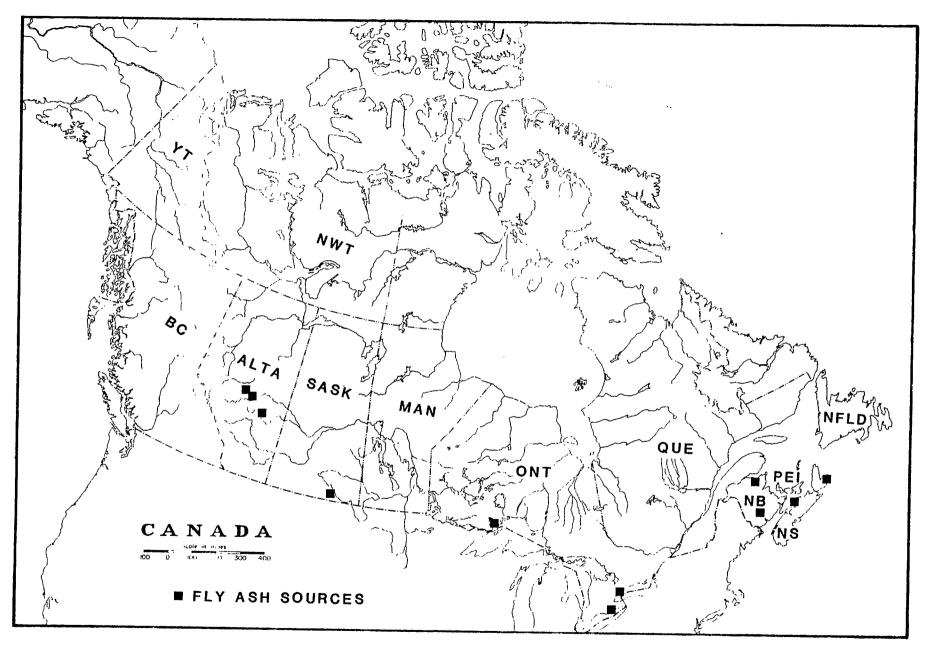


Fig. 1 - Locations of fly ash sources

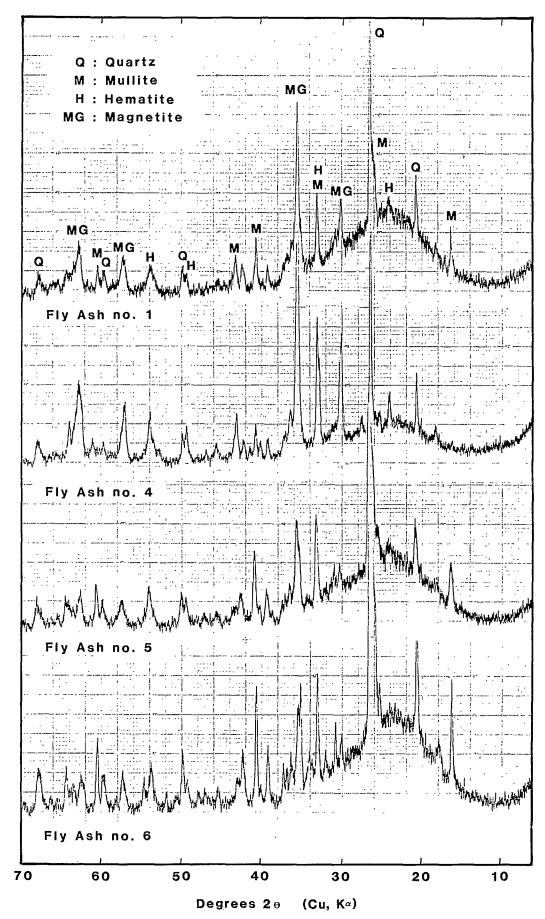


Fig. 2 - X-ray diffractograms for bituminous fly ashes

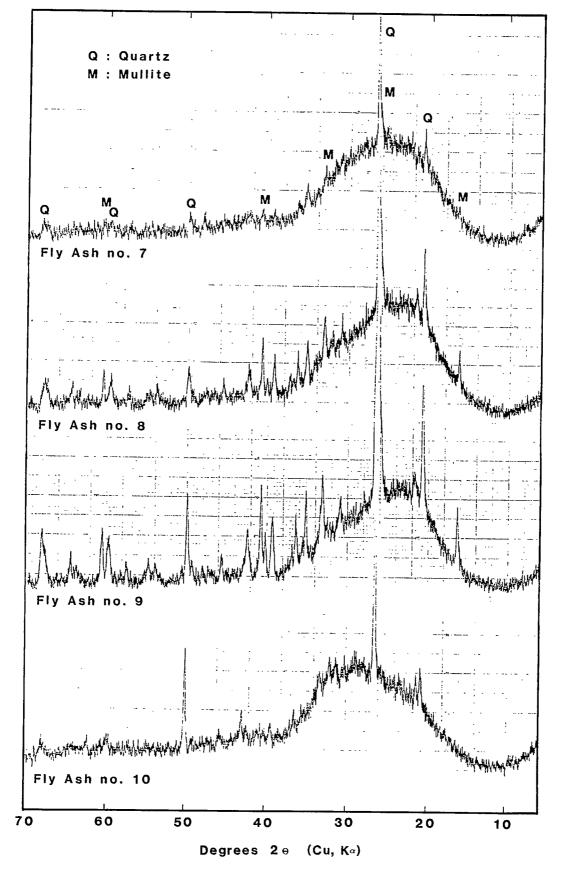


Fig. 3 - X-ray diffractograms for subbituminous and lignite fly ashes.



Bituminous ash (No. 1)



Subbituminous ash (No. 7)

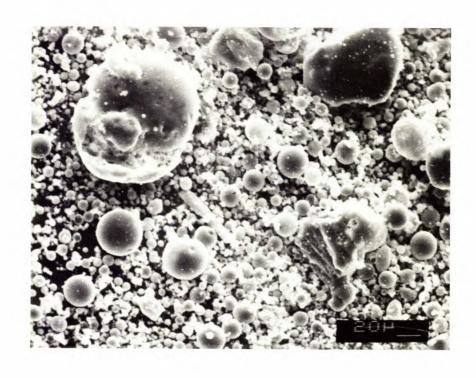


Bituminous ash (No. 6)



Lignite ash (No. 10)

Fig. 4(a) - SEM micrographs of selected fly ashes. (Backscattered electron images of polished sections of dispersed samples.)



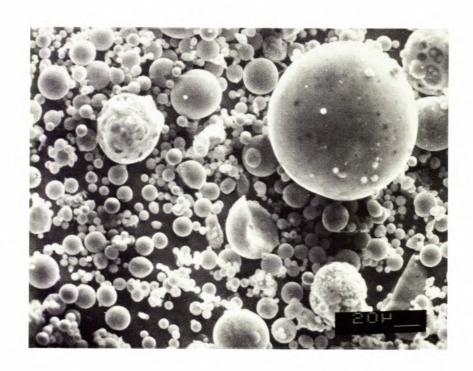


Fig. 4(b) - Secondary electron SEM images of fly ash particles (Bituminous ash No. 1)

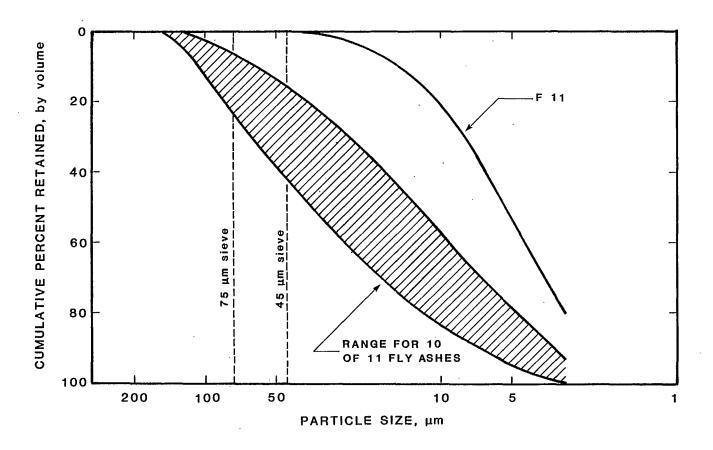


Fig. 5 - Range of particle size of fly ashes (Laser Method)

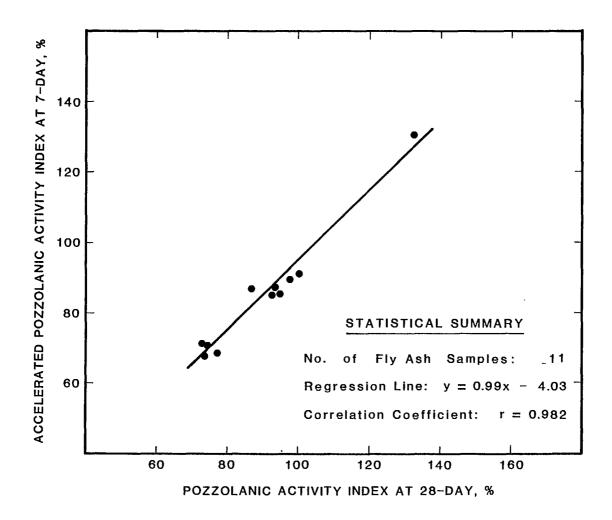


Fig. 6 - Relationship between 7-day accelerated and 28-day pozzolanic activity tests

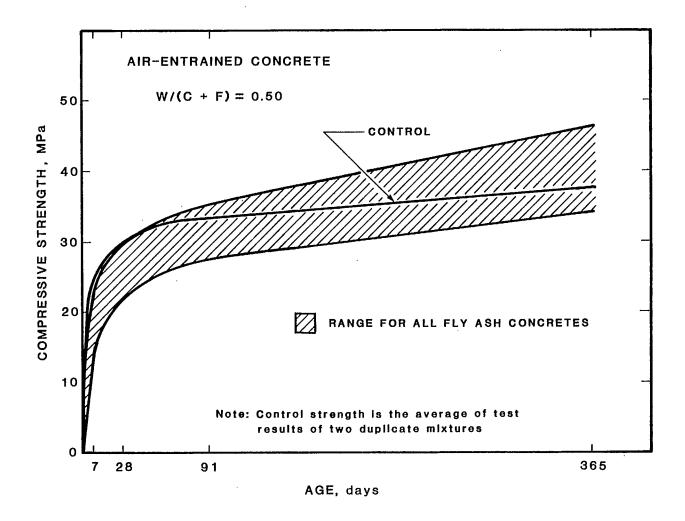


Fig. 7 - Compressive strength development with age

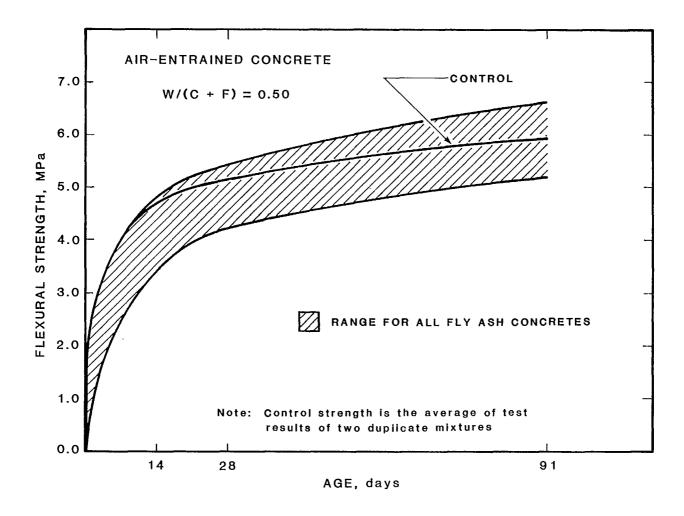


Fig. 8 - Flexural strength development with age

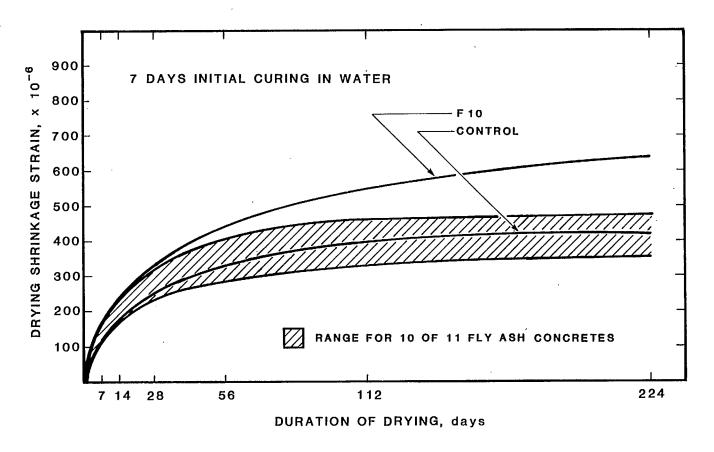


Fig. 9 - Drying shrinkage strains after initial curing of 7 days in water

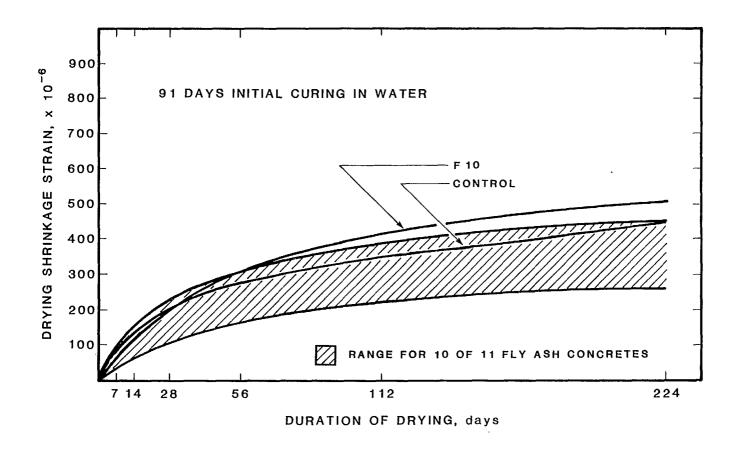


Fig. 10- Drying shrinkage strains after initial curing of 91 days in water

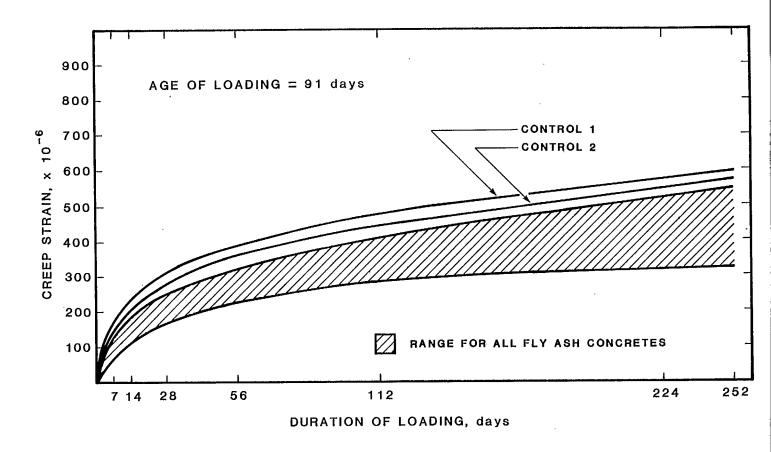


Fig. 11- Creep strains after initial moist-curing of 91 days

APPENDIX A DETAILED FREEZING AND THAWING TEST RESULTS

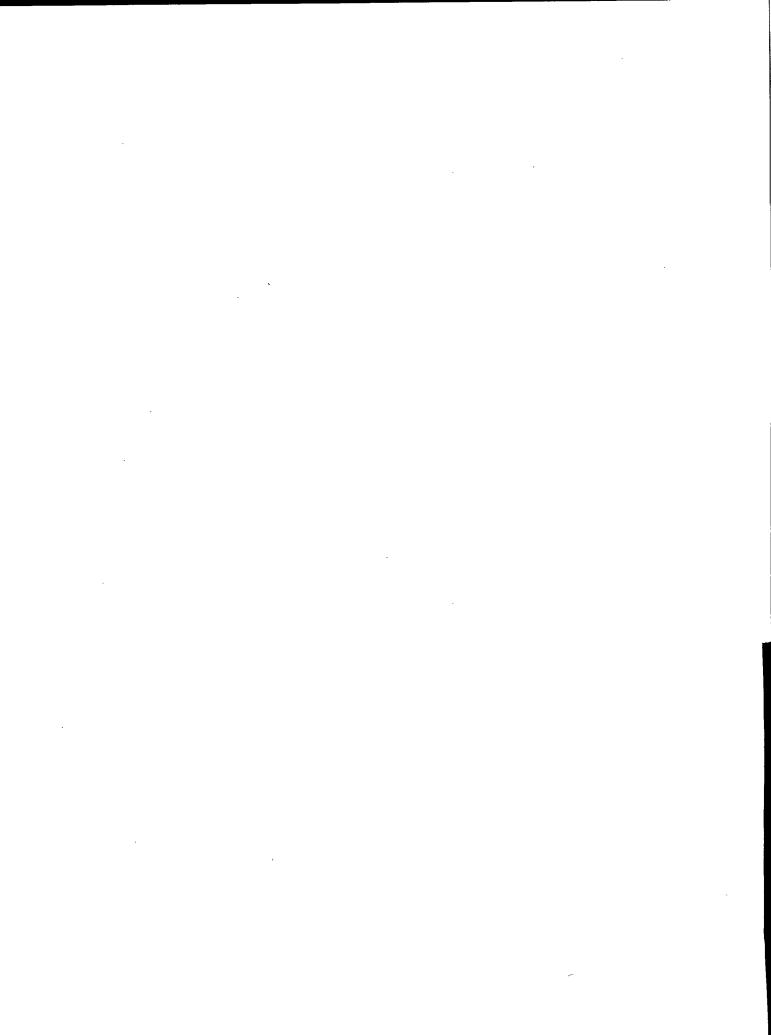


Table Al - Weight changes of reference moist-cured and test prisms exposed to freeze-thaw cycling

Mixture W/C+F)*		Weight of 76 x 102 x 390-mm prisms, kg								
	W/C+F)*	Air content, %	Reference moist-cured prisms			Test prisms subjected to freeze-thaw cycling				Relative change,
			At 14 days	At the end of cycling,**	Change, %	At O cycles	At 300 cycles	At 500 cycles	Change, % (after 500 cycles)	
Control-1 Control-2 F1 F2 F3 F4 F5 F6 F7 F8 F9 F10	0.50 0.50 0.50 0.50 0.50 0.50 0.50 0.50 0.50 0.50	6.5 6.4 6.2 6.2 6.3 6.5 6.5 6.1 6.2 6.5 6.5	7.186 7.125 6.970 7.124 7.088 7.147 7.067 7.077 7.052 7.024 7.034 7.017 7.287	7.202 7.159 7.010 7.147 7.112 7.164 7.109 7.113 7.087 7.045 7.044 7.041 7.306	+0.22 +0.48 +0.57 +0.32 +0.34 +0.24 +0.59 +0.51 +0.50 +0.30 +0.43 +0.34 +0.26	7.228 7.129 7.024 7.174 7.023 7.173 7.127 7.099 6.988 6.973 6.993 7.032 7.183	7.213 7.130 7.051 7.051 7.167 7.011 7.142 7.128 7.083 6.972 6.983 6.993 7.035 7.187	7.196 7.133 7.028 7.146 6.997 7.110 7.111 7.029 6.954 6.960 6.965 7.005 7.164	-0.44 +0.06 +0.06 -0.39 -0.37 -0.88 -0.22 -0.99 -0.49 -0.19 -0.40 -0.38 -0.26	-0.66 -0.42 -0.51 -0.71 -1.12 -0.81 -1.50 -0.99 -0.49 -0.83 -0.72

^{*} Water/(cement + fly ash) by weight.

^{**} Corresponding to the equivalent of 500 cycles, i.e., to about 90 days.

Table A2 - Length changes of reference moist-cured and test prisms exposed to freeze-thaw cycling

Mixture W/C+F)*		Effective Length of 76 x 102 x 390-mm prisms,** mm								
	W/C+F)*	Air content, %	Reference moist-cured prisms			Test prisms subjected to freeze-thaw cycling				Relative change,†† %
		At 14 days	At the end of cycling;†	Change ;†† %	At O cycles	At 300 cycles	At 500 cycles	Change,†† % (after 500 cycles)		
Control-1 Control-2 F1 F2 F3 F4 F5 F6 F7 F8 F9 F10	0.50 0.50 0.50 0.50 0.50 0.50 0.50 0.50	6.422234572456 6.6666666666666666666666666666666666	3.216 3.513 3.404 3.373 2.865 3.112 2.720 3.510 3.762 3.444 3.487 3.294 3.096	3.213 3.594 3.439 3.421 2.891 3.127 2.758 3.554 3.792 3.475 3.548 3.330 3.122	-0.001 +0.022 +0.010 +0.013 +0.007 +0.004 +0.010 +0.012 +0.008 +0.009 +0.017 +0.010 +0.007	3.419 3.556 3.279 3.073 3.487 3.241 3.675 3.287 3.559 3.439 3.470	3. 432 3. 528 3. 312 3. 089 3. 515 3. 261 3. 716 3. 289 3. 559 3. 461 3. 485	3.444 3.576 3.338 3.096 3.498 3.307 3.741 3.281 3.597 3.514 3.526	+0.007 +0.006 +0.016 +0.003 +0.018 +0.018 -0.002 +0.011 +0.021 +0.015	+0.008 -0.004 +0.003 -0.001 -0.001 +0.008 +0.006 -0.011 -0.006 +0.011 +0.008

^{*} Water/(cement + fly ash) by weight.

^{**} Gauge length = 358 mm.

[†] Corresponding to the equivalent of 500 cycles, i.e., to about 90 days.

 $[\]dagger\dagger$ As a percentage of total effective length including gauge length of 358 mm.

Table A3 - Changes in ultrasonic pulse velocity of reference moist-cured and test prisms exposed to freeze-thaw cycling

Mixture W/C	_	Air content, %	Ultrasonic pulse velocity of 76 x 102 x 390-mm prisms, m/s							
	W/C+F)*		Reference moist-cured prisms			Test prisms subjected to freeze-thaw cycling				Relative change, %
	; ;		At 14 days	At the end of cycling,**	Change,	At 0 cycles	At 300 cycles	At 500 cycles	Change, % (after 500 cycles)	
Control-1 Control-2 F1 F2 F3 F4 F5 F6 F7 F8 F9 F10	0.50 0.50 0.50 0.50 0.50 0.50 0.50 0.50	6.5 6.4 6.2 6.2 6.3 6.4 6.5 6.1 6.5 6.5	4670 4550 4340 4500 4470 4460 4380 4390 4380 4460 4410 4510	4850 4760 4670 4780 4720 4760 4720 4690 4650 4720 4670 4750 4780	+3.8 +4.6 +7.6 +6.2 +5.6 +6.7 +7.8 +6.8 +6.2 +5.8 +5.9 +5.3 +2.8	4690 4520 4310 4530 4430 4460 4470 4440 4330 4480 4470 4500 4640	4670 4560 4380 4520 4460 4510 4480 4480 4390 4520 4490 4460 4580	4690 4590 4430 4520 4470 4530 4510 4530 4420 4480 4480 4590 4500	0.0 +1.5 +2.8 -0.2 +0.9 +1.6 +0.9 +2.0 +2.1 0.0 +2.1 -0.2 +2.0 -3.0	-3.8 -3.1 -8.8 -6.4 -4.7 -5.1 -6.9 -4.8 -4.1 -5.7 -3.3 -5.8

^{*} Water/(cement + fly ash) by weight.

^{**} Corresponding to the equivalent of 500 cycles, i.e., to about 90 days.

Table A4 - Changes in fundamental longitudinal frequency of reference moist-cured and test prisms exposed to freeze-thaw cycling

Mixture W/C+F)*		Fundamental longitudinal frequency of 76 x 102 x 390-mm prisms, Hz								
	W/C+F)*	Air content,	, Reference moist-cured prisms			Test prisms subjected to freeze-thaw cycling				Relative change, %
		At 14 days,	At the end of cycling,**	Change,	At O cycles,	At 300 cycles,	At 500 cycles,	Change, % (after 500 cycles)		
Control-1 Control-2 F1 F2 F3 F4 F5 F6 F7 F8 F9 F10	0.50 0.50 0.50 0.50 0.50 0.50 0.50 0.50	6.5 6.4 6.2 6.2 6.3 6.4 6.5 6.1 6.4 6.5 6.4	5250 5120 4910 5030 4970 5060 4960 5000 4940 5100 4990 5090 5240	5470 5350 5290 5400 5320 5350 5360 5370 5370 5320 5360 5430	+4.2 +4.5 +7.7 +7.0 +5.7 +8.1 +6.8 +6.7 +5.3 +6.6 +5.3	5290 5120 4980 5050 4990 5040 5050 5030 4920 5100 5020 5020 5200	5230 5070 4890 5020 4910 5010 4980 4950 4860 5020 4960 4950 5090	5240 5100 4910 4980 4920 5020 5000 4970 4900 5030 4960 4950 5110	-0.9 -0.4 -1.4 -1.4 -0.4 -1.0 -1.2 -0.4 -1.4 -1.7	-5.1 -4.9 -9.1 -8.8 -8.4 -6.1 -9.1 -8.0 -7.1 -6.7 -7.8 -6.7 -5.3

^{*} Water/(cement + fly ash) by weight.

^{**} Corresponding to the equivalent of 500 cycles, i.e., to about 90 days.