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**EFFECT OF CURING ON RESISTANCE TO DEICING SALT SCALING,  
FREEZING-THAWING AND CHLORIDE-ION PENETRATION OF  
FLY ASH CONCRETE**

by

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*to my mom and dad*  
*Hâle and Muzaffer Baştopçu*

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## ABSTRACT

Fly ash is used extensively in concrete as a separately batched material and as an ingredient in blended cement. The use of fly ash in concrete as a replacement material for cement is becoming popular for economy and improvement of the properties of concrete for certain applications. As for normal concrete, when properly cured, fly ash reaction products help fill in the spaces between hydrating cement particles in the cement paste, thus lowering its permeability to water and aggressive chemicals which is strongly related to the durability of concrete.

Concrete performance can be assessed in many ways including durability and mechanical behavior. In this study the durability related performance of normal to high volume fly ash concrete cured differently is investigated along with mechanical properties. Durability characteristics include resistance to deicing salt scaling and freezing-thawing cycles, chloride-ion permeability, capillary absorption whereas the mechanical properties include splitting tensile, compressive, and flexural strengths. The four concrete mixtures used in this study contained 0%, 15%, 30%, and 40% fly ash replacing portland cement by mass.

To determine the effect of curing type, three different curing conditions were used. Curing Type 1 was the uncontrolled curing condition; the specimens were kept inside the laboratory after demolding till the time of testing. Curing Type 2 was the standard curing condition; specimens after demolding were put in 20 °C water for 1 week then kept in 20 °C curing room. Curing Type 3 named as hot curing; specimens after demolding were put in 30 °C water for 1 week and then kept in 20 °C curing room till they are tested at 28 and 56 days of age.

The results revealed that fly ash replacement generally had negative effects on mechanical properties. The negative effects are more distinguishable when the specimens are not cured (Type 1) and less pronounced with moist curing (Type 2) and least with hot curing (Type 3). On the contrary, fly ash replacement generally had positive effects on capillary absorption, chloride permeability (determined by AASHTO T 277), deicing salt scaling (tested according to ASTM C672) and freezing-thawing resistance with the

exception that these properties also were negatively affected in the uncontrolled cured specimens. Highest durability performance was observed in the hot cured (Type 3 ) specimens followed by moist curing (Type 2) ones, and the lowest results were those of the non-cured specimens (Type 1).

## KISA ÖZET

Uçucu Kül'ün beton yapımında kendi başına bir malzeme olarak veya katkı çimentolar içinde kullanımı giderek yaygınlaşmaktadır. Uçucu Külün çimento yerine kullanımının ekonomik yararları ve belirli uygulamalarda beton özelliklerini iyileştirmesinde onu popüler kılmaktadır. Normal betonlarda olduğu gibi, uygun biçimde kür edildiğinde Uçucu Kül reaksiyon ürünleri, betonun çimento hamuru içinde hidratlanan çimento parçacıkları arasındaki boşlukları doldurarak, beton dayanımına doğrudan etki eden su ve zararlı kimyasal madde geçirimsizliğinin azalmasını sağlar.

Betonun performansının belirlenmesinde dayanım ve mekanik testlerin de dahil olduğu pekçok farklı metod kullanılabilir. Bu çalışmada, normal betonlardan başlayarak, yüksek oranda uçucu kül ihtiva eden betonların farklı kür şartları altında dayanıklılığa bağlı performansları, mekanik özellikleri ile birlikte araştırılmıştır. Dayanıklılık özellikleri buz çözücü tuzlara ve donma-çözülme döngülerine karşı direnç, klor iyon geçirgenliği, kılcal geçirgenliği kapsarken, mekanik özellikler ise yarma mukavemeti, eşdeğer küp mukavemeti ve eğilme mukavemeti değerlerinden oluşmaktadır.

Bu çalışmada ele alınan dört farklı beton karışımında çimento miktarı ağırlıkça %0, %15, %30 ve % 40 oranlarında uçucu kül ile değiştirilmiştir. Kür türünün etkisinin belirlenmesi için bu karışımlar üç farklı kür şartına tabi tutulmuştur. "1. Kür Tipi" kontrolsüz kürdür ve numuneler kalıplardan çıkarıldıktan sonra deney zamanına kadar laboratuvar şartlarında bekletilmiştir. "2. Kür Tipi" standart kürdür ve numuneler kalıplardan çıkarıldıktan sonra bir hafta boyunca 20 °C suda bekletilmiş daha sonra deney zamanına kadar 20 °C de kür odasında tutulmuştur. Son kür tipi olan " 3. Kür Tipi"nde ise numuneler kalıptan çıkarılıp bir hafta 30 °C suda bekletilip daha sonra 20 °C kür odasına alınmış ve bütün numuneler 28. ve 56. günlerde test edilmişlerdir.

Deney sonuçları betonda çimento yerine uçucu kül kullanımının mekanik özellikler üzerinde genelde olumsuz etkiler oluşturduğunu göstermiştir. Bu olumsuz etkiler kontrolsüz küre (1. Kür Tipi) tabi tutulan numuneler için daha belirginken, standart kür edilen (2. Kür Tipi) numuneler için daha az etkili ve sıcak kür edilen (3. Kür Tipi) numuneler için en az etkilidir. Bunun aksine, çimento yerine uçucu kül kullanımı kılcal

geçirgenlik, klor geçirgenliđi (AASHTO T 277'e göre belirlenen), buz çözücü tuzlar (ASTM C672 ) ve donma-çözölme döngülerine karşı direnç üzerinde olumlu etkiler sağlamıştır. Burada da kontrolsüz kür edilen numunelerde uçucu külün etkisi olumsuz olmuştur. En yüksek dayanıklılık performansı sıcak küre (3. Kür Tipi) tabi tutulan numunelerde gözlenirken, bunu takiben standart kür (2. Kür Tipi) numuneleri yer almış ve en düşük sonuçlara kontrolsüz kür (1. Kür Tipi) edilen numunelerde karşılaşılmıştır.

## TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENT.....	i v
ABSTRACT.....	v
KISA ÖZET.....	vii
LIST OF FIGURES.....	xii
LIST OF TABLES.....	xviii
1. INTRODUCTION.....	1
1.1. Generation of Fly Ash.....	2
1.2. Properties of Fly Ash.....	3
1.3. Classification of Fly Ash.....	6
1.4. Use of Fly Ash in Concrete.....	7
1.5. Chemical Reactions of Fly Ash in Concrete.....	9
2. FLY ASH IN CONCRETE.....	12
2.1. Effect of Fly Ash in Fresh Concrete.....	12
2.1.1. Workability.....	12
2.1.2. Setting Time.....	12
2.1.3. Bleeding and Finishability.....	13
2.2. Effect of Fly Ash in Hardened Concrete.....	14
2.2.1. Effect of Fly Ash on Concrete Strength.....	14
2.2.2. Effect of Fly Ash on Elastic Properties, Creep, Shrinkage.....	18
2.2.3. Effect of Fly Ash on Durability of Concrete.....	19
2.2.3.1. Capillary Absorption.....	20
2.2.3.2. Chloride Ion Permeability.....	20
2.2.3.3. Carbonation.....	22
2.2.3.4. Sulfate Resistance.....	23
2.2.3.5. Resistance to Freezing-Thawing.....	23
2.2.3.6. Resistance to Deicing Salt Scaling.....	25
2.3. Effect of Curing on Fly Ash Concrete.....	26



2.3.1. Effect of Different Curing Types on Durability of Fly Ash Concrete.....	Page 26
3. EXPERIMENTAL STUDY.....	32
3.1. Materials.....	32
3.1.1. Cement.....	32
3.1.2. Fly Ash.....	33
3.1.3. Aggregate.....	34
3.1.4. Superplasticizer.....	35
3.2. Concrete Mixture.....	36
3.3. Casting and Curing of Test Specimens.....	37
3.4. Test Procedures.....	38
3.4.1. Fresh Concrete Properties.....	38
3.4.2. Hardened Concrete Properties.....	38
3.4.2.1. Mechanical Properties.....	38
3.4.2.2. Capillary Absorption .....	40
3.4.2.3. Chloride Ion Permeability .....	41
3.4.2.4. Deicing Salt Scaling Resistance.....	44
3.4.2.5. Freezing and Thawing Resistance.....	46
4. TEST RESULTS AND EVALUATION.....	48
4.1. Fresh Concrete Properties.....	48
4.2. Hardened Concrete Properties.....	49
4.2.1. Mechanical Properties.....	49
4.2.1.1. Splitting Tensile Strength.....	49
4.2.1.2. Flexural Strength.....	53
4.2.1.3. Equal Cube Strength.....	55
4.3. Capillary Absorption.....	56
4.4. Chloride Ion Permeability.....	59
4.5. Correlation between Initial Current and Total Charge Passed.....	63
4.6. Resistance to Deicing Salt Scaling.....	66
4.7. Resistance to Freezing and Thawing.....	69
4.7.1. Flexural Strength of Beams Subjected to Freezing and Thawing Cycles.....	69

4.7.2. Equivalent Compressive Strength of Beam Sections	Page
after Freezing and Thawing Cycles.....	72
5. CONCLUSION.....	80
APPENDIX.....	83
REFERENCES.....	94

## LIST OF FIGURES

	Page
FIGURE 1.1. - SEM image of fly ash particles at 2000 magnification.	4
FIGURE 1.2. - Idealized model of the effect of fly ash in freshly mixed concrete and the consequent hydrated microstructure.	11
FIGURE 2.1. - Rates of strength gain of portland cement concrete and concrete with fly ash in different percentages.	16
FIGURE 2.2. - Compressive strength development of fly ash concrete.	16
FIGURE 2.3. - Chloride permeability of concretes at 28 days cured at different temperatures.	22
FIGURE 2.4. - Comparison of freeze/thaw test data between OPC and FA concretes.	24
FIGURE 2.5. - Compressive and flexural strength of concrete with fly ash under different curing conditions.	29
FIGURE 2.6. - Effect of curing on coefficient of diffusion and PD index values.	30
FIGURE 2.7. - Importance of extent of curing to durability of concrete with fly ash.	31
FIGURE 3.1. - Aggregate grading curve and zones (according to TS 707).	35
FIGURE 3.2. - Experimental setup for sorptivity test.	41
FIGURE 3.3. - Experimental set-up for Rapid Chloride Permeability Test.	43

	Page
FIGURE 3.4. -The deicing salt scaling specimen with galvanize dike.	46
FIGURE 4.1. - 28 and 56 day splitting tensile strengths of concretes having various percentages of fly ash cured under Curing Condition 1.	52
FIGURE 4.2. - 28 and 56 day splitting tensile strengths of concretes having various percentages of fly ash cured under Curing Condition 2.	52
FIGURE 4.3. - 28 and 56 day splitting tensile strengths of concretes having various percentages of fly ash cured under Curing Condition 3.	52
FIGURE 4.4. - 28 and 56 days diffusion coefficients of concretes having various percentages of fly ash cured under Curing Condition 1.	58
FIGURE 4.5. - 28 and 56 days diffusion coefficients of concretes having various percentages of fly ash cured under Curing Condition 2.	58
FIGURE 4.6. - 28 and 56 days diffusion coefficients of concretes having various percentages of fly ash cured under Curing Condition 3.	58
FIGURE 4.7. - 28 and 56 days chloride permeability of concretes having various percentages of fly ash cured under Curing Condition 1.	61
FIGURE 4.8. - 28 and 56 days chloride permeability of concretes having various percentages of fly ash cured under Curing Condition 2.	61
FIGURE 4.9. - 28 and 56 days chloride permeability of concretes having various percentages of fly ash cured under Curing Condition 3.	61
FIGURE 4.10. - Initial current versus and charge passed in Rapid Chloride Permeability Test at 28 days for concrete having various percentages of fly ash cured and under different curing conditions .	65

	Page
FIGURE 4.11. - Plot of the relation between initial current and charge passed in Rapid Chloride Permeability Test at 56 days for concrete having various percentages of fly ash and cured under different curing conditions.	65
FIGURE 4.12.- 28 days flexural strengths of Freezing and Thawing and Control specimens cured under Curing Condition 1 and having various percentages of fly ash.	73
FIGURE 4.13.- 28 days flexural strengths of Freezing and Thawing and Control specimens cured under Curing Condition 2 and having various percentages of fly ash.	73
FIGURE 4.14.- 28 days flexural strengths of Freezing and Thawing and Control specimens cured under Curing Condition 3 and having various percentages of fly ash.	73
FIGURE 4.15. - 56 days flexural strengths of Freezing and Thawing and Control specimens cured under Curing Condition 1 and having various percentages of fly ash.	74
FIGURE 4.16.- 56 days flexural strengths of Freezing and Thawing and Control specimens cured under Curing Condition 2 and having various percentages of fly ash.	74
FIGURE 4.17.- 56 days flexural strengths of Freezing and Thawing and Control specimens cured under Curing Condition 3 and having various percentages of fly ash.	74
FIGURE 4.18.- 28 days equivalent cube strengths of Freezing and Thawing and Control specimens cured under Curing Condition 1 and having various percentages of fly ash.	78

	Page
FIGURE 4.19.- 28 days equivalent cube strengths of Freezing and Thawing and Control specimens cured under Curing Condition 2 and having various percentages of fly ash.	78
FIGURE 4.20.- 28 days equivalent cube strengths of Freezing and Thawing and Control specimens cured under Curing Condition 3 and having various percentages of fly ash.	78
FIGURE 4.21.- 56 days equivalent cube strengths of Freezing and Thawing and Control specimens cured under Curing Condition 1 and having various percentages of fly ash.	79
FIGURE 4.22.- 56 days equivalent cube strengths of Freezing and Thawing and Control specimens cured under Curing Condition 2 and having various percentages of fly ash.	79
FIGURE 4.23.- 56 days equivalent cube strengths of Freezing and Thawing and Control specimens cured under Curing Condition 3 and having various percentages of fly ash.	79
FIGURE A1. - Overall test results of 28 and 56 days chloride permeability of concrete specimens cured under 3 different curing conditions and having various percentages of fly ash and portland cement tested at 56 days.	84
FIGURE A2. - Test specimen at the beginning of the deicing salt scaling test.	85
FIGURE A3. – FA003 after the 5 <sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days).	85
FIGURE A4. – FA003 after the 15 <sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days).	86

	Page
FIGURE A5. – FA003 after the 20 <sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days).	86
FIGURE A6. – FA003 after the 25 <sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days).	87
FIGURE A7. – FA003 after the 30 <sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)	87
FIGURE A8. – FA002 after the 25 <sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)	88
FIGURE A9. – FA152 after the 25 <sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)	88
FIGURE A10. – FA302 after the 30 <sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)	89
FIGURE A11. – FA452 after the 35 <sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)	89
FIGURE A12. – FA002 after the 30 <sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days). (test terminated)	90
FIGURE A13. – FA152 after the 30 <sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days). (test terminated)	90
FIGURE A14. – FA003 after the 30 <sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days). (test terminated)	91
FIGURE A15. – FA153 after the 35 <sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days). (test terminated)	91

	Page
FIGURE A16. – FA001, 151, 301, and 451 after the 10 <sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days).	92
FIGURE A17. – FA002, 152, 302, and 452 after the 20 <sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days).	92
FIGURE A18. – FA003, 153, 303, and 453 after the 15 <sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days).	93
FIGURE A19. – Rapid Chloride Permeability Test Setup.	93



## LIST OF TABLES

	Page
TABLE 1.1. - Fly ash generated in each power plant in Turkey.	3
TABLE 2.1.- Characteristics of Freshly mixed concrete.	13
TABLE 2.2. - Setting time of concrete containing different percentages of fly ash.	14
TABLE 2.3. - Compressive strength at different ages of fly ash concrete.	17
TABLE 2.4. - Compressive strength data for different curing temperatures.	18
TABLE 2.5. - Compressive strength at different ages of fly ash concrete.	19
TABLE 2.6. - 9 and 28 days chloride permeability data, coulombs.	21
TABLE 3.1. – Physical, chemical and strength properties of the cement.	33
TABLE 3.2. - Chemical properties of the fly ash.	34
TABLE 3.3. - Sieve analysis and physical properties of the aggregates.	35
TABLE 3.4. - Specifications of the superplastizer ( SIKAMENT FF-N ).	36
TABLE 3.5. - Mix proportioning of concretes in kg/m <sup>3</sup>	37
TABLE 3.6. - Interpretation of results obtained using RCPT T 277-89.	43
TABLE 3.7. - The rating scale for ASTM C 672.	45
TABLE 4.1. - Properties of fresh concrete.	49

	Page
TABLE 4.2. - Splitting tensile strengths of concretes at 28 and 56 days and percent change in splitting tensile strength with respect to the reference and age.	51
TABLE 4.3. - Flexural strength of control specimens of 28 and 56 days freezing and thawing specimens.	54
TABLE 4.4. - Equivalent cube strengths of beam sections of control specimens broken after freeze-thaw cycles started at 28 and 56 days.	55
TABLE 4.5. - The diffusion coefficient data and percent change of the diffusion coefficient of concretes with respect to the reference and age.	57
TABLE 4.6. - Chloride permeability of concrete specimens, determined by the total charge passed, at the ages of 28 and 56 days.	60
TABLE 4.7. - Percent change of the chloride ion permeability, determined by the total charge passed, with respect to the reference and age.	62
TABLE 4.8. - Initial current measurements at 28 and 56 days.	64
TABLE 4.9. - Slope and correlation coefficients of the relation between initial current and total charge passed at 28 and 56 days.	64
TABLE 4.10. - Results of the deicing salt scaling tests on concrete specimens tested at the ages of 28 and 56 days.	67
TABLE 4.11. - 28 and 56 days results of the deicing salt scaling tests on concrete specimens.	68
TABLE 4.12. - Flexural strength of beams tested after freeze-thaw cycles started at 28 days.	71

	Page
TABLE 4.13. - Flexural strength of beams tested after freeze-thaw cycles started at 56 days.	72
TABLE 4.14. - Equivalent cube strengths of beam sections broken after freezing and thawing cycles started at 28 days.	75
TABLE 4.15. - Equivalent cube strengths of beam sections broken after freeze-thaw cycles started at 56 days.	76

## 1. INTRODUCTION

Today's engineers are seeking new ways, more than ever, to utilize wastes, recycled materials and by-products of industrial processes. This is a part of the joint effort to meet the constantly growing demand as the natural resources of earth are becoming increasingly scarce. As in many fields, this situation is also valid for civil engineering and especially for the field of construction materials.

Turkey, like most other developing countries, generates large amounts of waste materials from a wide variety of industries. Some of these waste materials are potentially recyclable for being used as mineral admixtures in concrete and other uses. Unfortunately, there is little done to utilize this enormous reserve of material in Turkey.

One of the commonly produced (nearly 15 million tons per year) waste materials in Turkey is fly ash, generated by the combustion of pulverized coal in electric power plants [1]. A method for utilizing this material is cement replacement in concrete. Fly ash, being used as a cement replacement material has various advantages, no need to mention the economical one. The aspects to be considered in assessing the advantages or disadvantages are the fresh concrete properties, durability and strength of fly ash concrete.

The fresh concrete properties affected by the presence of fly ash are pumpability, surface finishing, workability and setting time. These are positively affected mainly due to the microstructure of the fly ash particles in the fresh concrete paste. Fly ash particle's spherical structure helps them to act as a lubricant in the paste therefore increasing workability.

Concrete surfaces are vulnerable to deicing salts especially when subjected to repeated cycles of freezing and thawing. Under such conditions surface scaling is nearly inevitable. Freezing and thawing alone may cause considerable damage to concrete causing first micro-cracking leading to total deterioration. Another type of deterioration can be initiated by the ingress of chloride ions from the environment. This is a case where the reinforced concrete structure is harmed as the chloride-induced corrosion of reinforcements is observed in the long run. Other major causes of deterioration are alkali-aggregate

reactivity and sulfate attack. The common aspect of all these is that the deterioration starts and/or is accelerated with the ingress of liquids into the concrete mass. For this reason, despite other measures, lowering the permeability of concrete seems to be the most effective means to obtain more durable concrete for long-term high quality performance.

The strength and permeability of concrete is mainly determined by the microstructure of the paste. Properties of the cementing material and the water-binder ratio are the key factors affecting the hydration reactions forming this paste. Due to their shape, fineness and additional pozzolanic reactions mineral admixtures are effective in forming a denser and less permeable concrete paste. It is these pozzolanic reactions where the influence of curing conditions come to stage and it is found that increased moist curing accelerates these long term pozzolanic reactions of fly ash in concrete.

This study aims to investigate the effect of curing condition on mechanical properties and durability (resistance to deicing salt scaling, freezing and thawing and chloride-ion penetration) of fly ash (Class C) concrete. In this scope, mechanical tests are done, ASTM C 672 was conducted in determining the deicing salt scaling and freezing and thawing resistance with daily cycles ( $-18^{\circ}\text{C}$  -  $+23^{\circ}\text{C}$ ), and AASHTO T 277 was used to determine chloride permeability at the ages of 28 and 56 days.

### 1.1. Generation of Fly Ash

Fly ash is the residue from the combustion of pulverized coal at electric power plants. It is the fine dust, which is carried out of the boiler with the flue gas, and it is removed from the flue gas stream with electrostatic precipitators by means of an electrical charge attraction. Periodically the hopper fees into a pneumatic line, which conveys the fly ash to disposal. The fly ash may be stored in soils for commercial use, wet sluiced to an adjacent ash pond, or damped and trucked to a nearby landfill or utilization facility [2].

In Turkey, there are 12 major thermal power plants burning coal. The amount of coal consumed is nearly 50 million tons per year. This corresponds to 15 million tons of fly ash generated each year [3]. Approximately 50 million tons of fly ash is produced annually

in the United States. About 10 percent of total fly ash production is used in concrete. Unfortunately, this percentage is virtually zero in Turkey. TABLE 1.1 shows a summary of the thermal power plants in Turkey and their annual coal consumption and fly ash generation. [1]. The lignite mines, which are more common in Turkey, located in their vicinity, power all of these plants. Zonguldak's Çatalağızı plant is the only exception burning bituminous coal instead of lignite.

TABLE 1.1. - Fly ash generated in each power plant in Turkey [1]

Power Plant	Location	Coal *10 <sup>3</sup> ton/year	Fly Ash *10 <sup>3</sup> ton/year
Çatalağızı (A)	Zonguldak	650	130
Seyitömer	Kütahya	3500	980
Tunçbilek	Kütahya	2300	607
Yatağan	Muğla	4200	1075
Afşin-Elbistan	Kahramanmaraş	17000	2434
Yeniköy	Muğla	3300	1135
	Ankara	1750	420
Soma	Manisa	300	84
	Manisa	4000	1312
Çatalağızı (B)	Zonguldak	1600	704
Gemlik	Bursa	1539	430
Kangal	Sivas	3600	576

## 1.2. Properties of Fly Ash

Fly ashes are fine powders, heterogeneous in nature. They consist mainly of rounded or spherical glassy particles of varying silica, alumina, and iron oxide content. Irregular and angular particles are also found in fly ashes, which include both unburned coal remnants and mineral particles. Fly ashes may be finer or coarser than ordinary portland cements, depending on the source from which they are obtained. According to ASTM C618 specifications [4], no more than 34% should be retained on the No.325 (45µm) sieve.

Wide variations in coal compositions, combustion conditions, and ash collection systems lead to wide variations in fly ash composition. The shape, fineness, particle size distribution, density, and composition of fly ash particles influence the properties of freshly mixed unhardened concrete and the strength development of the hardened concrete. Fly ashes produced at different power plants or at one plant with different coal sources may have different colors. Fly ash color and the amount used can influence the color of the resulting hardened concrete in the same way as changes in the cement or fine-aggregate color. The fly ash concrete used in this study, having a lighter grey color, was also clearly differentiable from the control concrete. Fly ash color is generally not an engineering concern except that the change in the color of an ash from a particular source may be an indicator of changes in coal source, loss on ignition, iron content, or burning conditions.

Particle size and the shape characteristics of fly ash are dependent upon the source and uniformity of the coal, the degree of pulverization prior to burning, the combustion environment (temperature level and oxygen supply), uniformity of combustion, and the type of collection system used such as mechanical separators, bag filters, or electrostatic precipitators. It was also found that the shape of fly ash particles is also a function of particle size. [5] The SEM image in Fig 1.1 clearly shows the spherical microstructure of the fly ash particles.

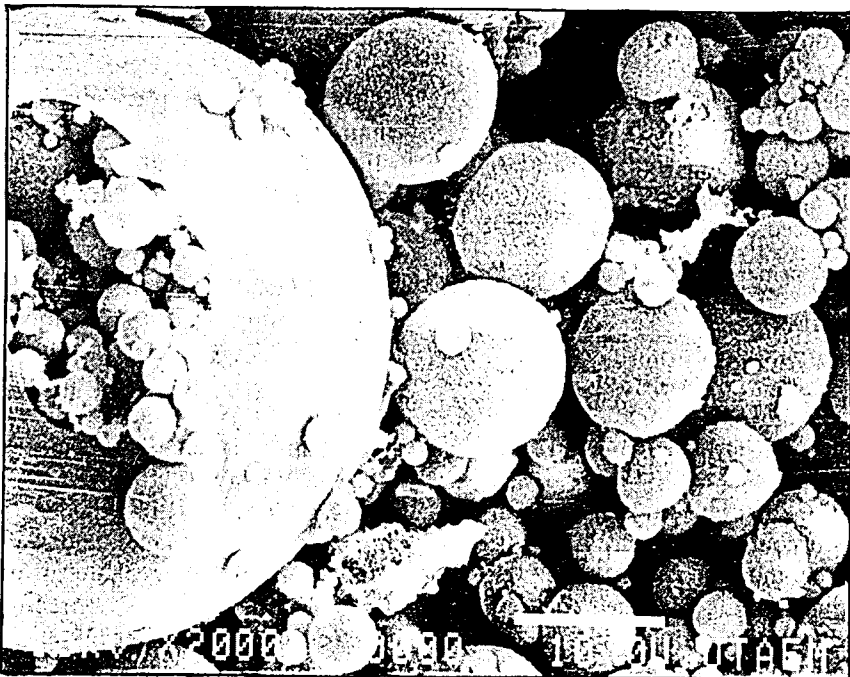


FIGURE 1.1. - SEM image of fly ash particles at 2000 magnification [5]

Individual particles in fly ash range in size from less than 1  $\mu\text{m}$  to greater than 1mm. In older plants where mechanical separators are used, the fly ash is coarser than in more modern plants, which use electrostatic precipitators or bag collectors. In fly ash suitable for use as pozzolan in concrete the majority of the particles pass the No. 325 (45- $\mu\text{m}$ ) sieve. The particle size distribution of fly ash from a particular source will normally remain relatively constant, provided there are no major changes in the coal source, coal grinding, process operation, and plant load.

Fineness of fly ash has a significant influence on its performance in concrete. The results of tests by ASTM C 430, No. 325 (45- $\mu\text{m}$ ) sieve fineness was used [5] as a means to correlate the fineness of Class F fly ash with certain concrete properties. Their data indicate that concrete strength, abrasion resistance, and resistance to freezing and thawing are direct functions of the proportion of the fly ash finer than the No. 325 (45- $\mu\text{m}$ ) sieve. They concluded that fineness is a more consistent indicator of fly ash performance in concrete and the performance improves with increased fineness.

The specific gravity of solid fly ash particles ranges from 1.90 to 3.02 [6], but normally in the range of 2.20 to 2.80. Some fly ash particles are capable of floating on water, indicating a specific gravity less than 1.0. High specific gravity is often an indication of fine particles. Fly ashes high in iron tend to have higher specific gravity [7]. Class C fly ashes tend to have finer particles and fewer cenospheres; thus their specific gravity tends to be higher in the range of 2.4 to 2.8.

Very fine particles have high specific surface areas. For example, a powder consisting of 1- $\mu\text{m}$  diameter spherical particles of the density of portland cement would have a specific surface area of about 2000  $\text{m}^2/\text{kg}$ , about six times that of ordinary portland cements. For materials with a wide range of particle sizes, most of the specific surface area is contributed by the finest fractions.



### 1.3. Classification of Fly Ash

The two main groups of fly ashes (F and C) are recognized by ASTM are obtained from serial different kinds of coal [2]. The reason for the differences among the variety of coals is due to the differences in the coal formation processes over geological time periods [3]. The composition of fly ash produced by combustion depends on the organic and inorganic components in coal. The main constituents of ash are inorganic glassy particles formed from the mineral matter present in the coal. The mineral matter in coal is the result of at least three of the following processes:

- growth of the original plant tissue,
- deposition of minerals during and after peat formation,
- crystallization of secondary minerals which permeated the coal seams

The two main groups given in ASTM C 618 have different sources of coal. Class F fly ash is usually produced by burning of anthracite or bituminous coal while the Class C fly ash is normally produced by burning sub-bituminous coal or lignite. In Turkey nearly all the power plants given in TABLE 1.1 are using lignite for power generation therefore, the fly ash produced in Turkey is generally classified as Class C, high-calcium fly ash.

Bituminous and anthracite coals rarely contain high calcium mineral matter in significant proportion; the sub-bituminous coals and lignites may or may not contain a high amount of calcium [8]. The separation of fly ash into two classes reflects differences in composition, which affect cementitious and pozzolanic properties. Class C (High-calcium) fly ash usually has cementitious properties in addition to pozzolanic properties, while Class F (Low-calcium) fly ash is rarely cementitious when mixed with water [9]. Low calcium fly ash, also called low-lime fly ash has a CaO content less than 10 percent and truly pozzolanic properties where as Class C, high-lime fly ash, has a CaO content of greater than 10 percent and possesses some cementitious properties [10].

#### 1.4. Use of Fly Ash in Concrete

Fly ash is used extensively in concrete as a separately batched material and an ingredient in blended cement. It is used for economy and to improve the properties of concrete used for certain applications.

Fly ash from coal-burning electric power plants became in quantity in the 1930's. In the United States, the study of fly ash for use in portland cement concrete began at about that time. In 1937, R. E. Davis and his associates at the University of California published results of research on concrete containing fly ash [11]. This work served as the foundation for early specifications, methods of testing, and use of fly ash.

Davis and colleagues recognized in subsequent studies the reactivity of fly ash with calcium and alkali-hydroxides in portland cement paste, and therewith the ability of fly ash to act as preventive measure against deleterious alkali-aggregate reactions [11]. The U.S. Army Corps of Engineers, The Bureau of Reclamation, major U.S. engineering firms, and others recognized the beneficial effects of fly ash on the workability of fresh concrete and the advantageous reduction of peak temperatures in mass concrete. The beneficial aspects of fly ash were especially notable in the construction of large concrete dams [12].

The oil crisis of the 1970's led to the greater use of coal to fire electric power plants. Fly ash containing higher levels of calcium oxide became available due to the use of coal and lignite containing calcium compounds in their incombustible fractions. Most such coals in United States are sub-bituminous and lignite. Concurrent with this increased availability of fly ash, extensive research in the United States, Canada and elsewhere has led to better understanding of the chemical reactions involved and improved the technology to economically use the large quantities of fly ash now available to the concrete industry [13].

Fly ash is used in concrete for reasons including economics, improvements and reduction in temperature rise in fresh concrete, workability, and contribution to durability and strength in hardened concrete. Fly ash makes efficient use of the products of the hydration of portland cement: (1) solutions of calcium and alkali hydroxide which exist in

the pore structure of cement paste and (2) the heat generated by hydration of portland cement, an important factor in initiating the reaction of the fly ash.

When fly ash concrete is properly cured, fly ash reaction products help fill in the cement paste fraction of the concrete, thus lowering its permeability to water and aggressive chemicals [14]. The slower reaction rate of many fly ashes is a real help in limiting the amount of early heat generation and the detrimental early temperature rise in massive structures. Using fly ash in concrete saves energy by reducing the amount of portland cement (an energy intensive product) required to achieve the desired concrete properties.

Fly ash generally causes an increase in setting time- both initial and final set of concrete. It normally allows a reduction in the quantity of mixing water in a concrete mixture necessary to produce a target slump. Because of the fineness and rounded shape of the fly ash particles, its use generally improves cohesion and workability of the concrete at a given slump. Segregation and bleeding are often reduced. Fly ash improves the pumpability of concrete mixtures and improves the ease of flat-work finishing operations. This property was perfectly observed during the experiments and the surface finishing of fresh concrete beams containing up to 45% fly ash concrete were considerably easier than normal concrete specimens. As the concrete hardens, the fly ash makes use of the heat developed through portland cement hydration to accelerate pozzolanic reactions and, thereby, promotes the reaction of the fly ash with the available calcium and alkali hydroxides. Using fly ash in concrete generally allows a reduction in the required cement content, and therefore reduces the peak temperature developed in the concrete during curing.

Increased long-term strength through continued pozzolanic reactions is achieved with most fly ashes in concrete, if the concrete is maintained in a moist environment at moderate temperatures [15]. Early strength development at three days age is normally reduced by the use of Class F fly ashes and may be reduced with Class C fly ashes also. However, proper proportioning of the mixture can generally compensate this for.

Continued long-term reaction of fly ash reduces the size of the pore spaces in the cement paste. Permeability and the rate of diffusion of moisture and aggressive chemicals into the concrete is thus reduced, thereby reducing the danger of damage due to sulfate attack, steel corrosion induced by chloride penetration, deicing salt scaling, freezing and thawing deterioration, and alkali-silica reaction.

The performance of a concrete mixture containing fly ash depends upon the same factors that determine quality in non-fly ash concrete. These factors include: (a) the characteristics of the materials incorporated in the mixture; (b) the proportioning of the mixture; (c) the quality assurance programs for material quality and the quality control exercised over concrete production and placement; (d) the quality of workmanship employed in all facets of the concrete construction, and finally (e) the extent of curing.

### **1.5. Chemical Reactions of Fly Ash in Concrete**

Fly ashes are complex in their range of chemical and phase compositions. They consist of heterogeneous combinations of glassy and crystalline phases. Although the constituents are typically not present as oxides, it is customary to express the results of the chemical analysis of the materials in terms of the oxides of the elements, eg., silica ( $\text{SiO}_2$ ), alumina ( $\text{Al}_2\text{O}_3$ ), ferric oxide ( $\text{Fe}_2\text{O}_3$ ), calcium oxide ( $\text{CaO}$ ), magnesium oxide ( $\text{MgO}$ ), and sulfur trioxide ( $\text{SO}_3$ ) [16].

Wide ranges exist in the amounts of the three principal constituents-  $\text{SiO}_2$  (25 to 60 percent),  $\text{Al}_2\text{O}_3$  (10 to 30 percent),  $\text{Fe}_2\text{O}_3$  (5 to 25 percent). If the sum of these three ingredients is 70 percent or greater, the fly ash is technically considered as Class F. Because Class C fly ashes generally contain significant percentages of calcium compounds reported as  $\text{CaO}$ , the sum of the three constituents just mentioned is required only to be greater than 50 percent. The  $\text{MgO}$  content of fly ash is generally not greater than 5 percent. The level of alkali oxides expressed as  $\text{Na}_2\text{O}$  equivalent is generally less than 5 percent in Class F fly ashes, but it may range up to about 10 percent in Class C fly ashes [16].

Active compound in addition to calcium alumino-silicate glass that may be present in Class C fly ash are free lime ( $\text{CaO}$ ), anhydrite ( $\text{CaSO}$ ), tricalcium aluminate ( $3\text{CaO}.\text{Al}_2\text{O}_3$ ), calcium sulfoaluminate ( $4\text{CaO}.3\text{Al}_2\text{O}_3.\text{SO}_4$ ), and rarely, calcium silicates. The strength related properties of concrete containing Class C fly ash, compared to Class F fly ash, are influenced by the cementitious calcium alumino-silicate glass and, in many instances, by the presence of crystalline phases which contribute to the hydration of Class C fly ashes [17].

High-calcium (Class C) fly ashes often react directly with water to form cementitious phases such as C-S-H (calcium-silicate-hydrate), calcium hydroxide, and ettringite. In addition, the glassy phase in these fly ashes, while still predominant, is often less abundant but more reactive than Class F fly ashes. These glassy materials are reactive with the calcium and alkali hydroxides released from the cement-fly ash system.

The principal products of the reactions of fly ash with alkali and calcium hydroxide in concrete are essentially the same as that of the hydration of portland cement (calcium-silicate- hydrate [C-S-H]). The reaction of fly ash depends largely upon breakdown and dissolution of the glassy structure by the hydroxide ions and the heat mobilized during the early hydration of the portland cement fraction. The reaction of the fly ash continues to consume calcium hydroxide to form additional C-S-H as long as calcium hydroxide is present in the pore liquid of the cement paste. These reactions are the key factors affecting the durability of fly ash concrete since they result in a denser concrete mass, less permeable to the ingress of aggressive liquids and prevent calcium hydroxides from leaching and forming a more permeable matrix [16]. An idealized model of the effect of fly ash in freshly mixed concrete and the consequent hydrated microstructure is illustrated in FIGURE 1.2.; (a) freshly OPC concrete; (b) hydrated OPC after 28 day; (c) the same concrete as (a) with fly ash addition; (d) denser , more uniform hydrate microstructure after 28 days [10].

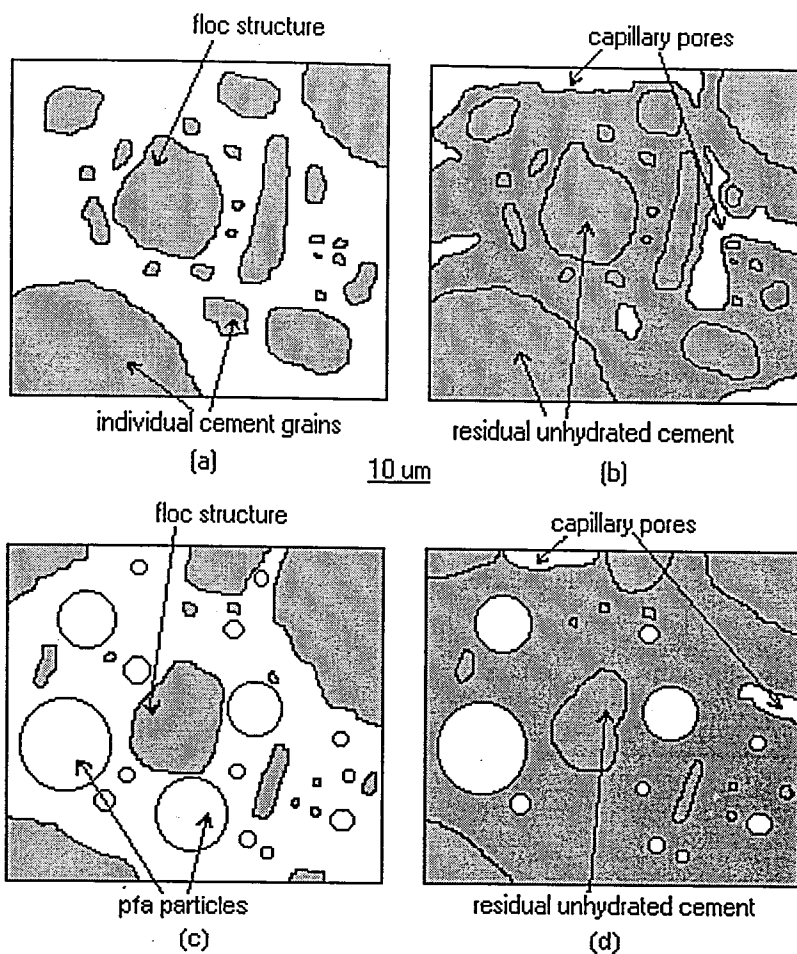


FIGURE 1.2. - Idealized model of the effect of fly ash in freshly mixed concrete and the consequent hydrated microstructure [10]

Clarifying the basic principles of fly ash reaction makes it possible to identify the primary factors, which in practice will influence the effectiveness of the use of fly ash in concrete. These factors include: (a) the chemical and phase composition of the fly ash and the portland cement; (b) the alkali-hydroxide concentrations of the reacting systems; (c) the morphology of the fly ash particles; (d) the fineness of the fly ash and of the portland cement; (e) the development of heat during the early phases of the hydration process; and (f) the reduction in mixing water requirement when using fly ash [16].

## 2. FLY ASH IN CONCRETE

### 2.1. Effect of Fly Ash in Fresh Concrete

#### 2.1.1. Workability

The absolute volume of cement plus fly ash normally exceeds that of cement in similar non-fly ash concrete mixtures. This is because the mass of fly ash used to replace cement is usually equal to or greater than that replaced and the fly ash normally is of lower density. This is also evident from the unit weight measurements of fresh concrete mixes. While it depends on the proportions used, this increase in paste volume produces concrete with improved plasticity and better cohesiveness [18]. In addition, the increase of the  $\text{SiO}_2$  concentration over  $\text{CaO}$  produces an improved stability of the dispersion of the cement and fly ash particles in the highly alkaline fresh paste. [19].

Fly ash changes the flow behaviour of the cement paste [20]; also, the generally spherical shape of fly ash particles normally permits the water in the concrete to be reduced for a given slump [21]. Water reduction by fly ash further increases the solids-to-liquid ratio with additional beneficial results. Especially Class C fly ash generally has a high proportion of particles finer than  $10\text{ }\mu\text{m}$ , which favourably influence concrete workability. [19] Pressures on formwork may be increased with fly ash concrete due to improved workability, slower slump loss, and retarded setting characteristics [22]. Özyıldırım and Halstead [23] also points out that some slump loss is observed with mixes containing Type II cement and Class F fly ash. Their results are given in TABLE 2.1.

#### 2.1.2. Setting Time

The use of fly ash may retard time of setting of concrete. Class F fly ash generally extends, the time of setting, while Class C fly ash may extend, reduce, or have no significant effect on the time of setting. The setting characteristics of concrete are influenced by ambient and concrete temperature; cement type, source, content, and fineness; water content of the paste; soluble alkalies; use of other admixtures; the amount of fly ash; and the fineness and chemical composition of the fly ash [21]. The actual effect

of a given fly ash on time of setting may be determined by testing when a precise determination is needed or by observation when a less precise determination is acceptable.

TABLE 2.1.- Characteristics of Freshly mixed concrete [23].

Batch	Cement type	PC/FA/SF	Air content percentage	Slump, mm	Unit weight, kg/m <sup>3</sup>
1	II	100/0/0	7.5	100	2275
2	II	65/32/3	6.5	95	2280
3	II	65/26/9	8.0	90	2230
4	II	55/40/5	5.6	90	2294
5	II	55/35/10	7.8	80	2236

In a study by Bilodeau and Malhotra [24] it was found that at every water-to-cementitious ratio, the initial and final setting times of high-volume fly ash (Class F) concrete are noticeable retarded compared to those of the reference concretes. This is attributed to the lower cement content of the high-volume fly ash concretes and is in agreement with the other published data. The results of this study are given in TABLE 2.2.

### 2.1.3. Bleeding and Finishability

Using fly ash in air-entrained and nonair-entrained concrete mixtures usually reduces bleeding by providing greater fines volume and a lower water content for a given workability [25]. The fines present from fly ash can compensate for a deficiency in aggregate fines and help block bleed-water channels. Since fly ash concrete typically has a longer time of setting than concrete without fly ash, such mixtures should be finished at a later time than mixtures without fly ash. Failure to do so could lead to premature finishing, which can seal the bleed water under the top surface creating a plane of weakness. Longer times of setting may increase the chances of plastic shrinkage cracking or surface crusting under conditions of high evaporation rates [26].



TABLE 2.2. - Setting time of concrete containing different percentages of fly ash

[24]

Mixture No.	W/(C+FA)	Cement, kg/m <sup>3</sup>	Fly ash,		Time of setting, (h: min)	
			Source	kg/m <sup>3</sup>	Initial	Final
1	0.38	126	L	173	9: 15	12: 50
2	0.31	156	L	215	8: 25	11: 25
3	0.27	180	L	248	7: 45	10: 30
4	0.39	124	S	172	8: 45	12: 05
5	0.31	155	S	214	10: 10	13: 15
6	0.27	182	S	251	9: 20	12: 15
7	0.39	124	R	170	7: 25	10: 30
8	0.31	155	R	213	7: 40	10: 15
9	0.27	181	R	250	6: 20	8: 50
10	0.39	292	-	0	6: 40	8: 30
11	0.31	368	-	0	5: 40	7: 10
12	0.27	428	-	0	5: 15	6: 50

## 2.2. Effects of Fly Ash in Hardened Concrete

### 2.2.1. Effect of Fly Ash on Concrete Strength

Strength at any given age and rate of strength gain of concrete are affected by the characteristics of the particular fly ash, the cement with which it is used, and the proportions of each used in the concrete [16]. There is no known special effect on the relationship of tensile strength to compressive strength for the use of fly ash compared to non-fly ash concrete. The tensile strength of fly ash concrete (splitting cylinder or flexural) in relation to its compressive strength is essentially similar to that of portland cement concrete. Thus, in estimating the tensile strength of concrete from a given compressive strength, the normally accepted relationships can be applied equally to concrete with or without fly ash.

Generally, incorporating high-calcium (Class C) fly ashes tends to be only marginally effective in the rate of strength development in concretes. The strength development of a concrete mix incorporating fly ash designed for a given workability and 28-day standard-cured ( $20^{\circ}\text{C}$  water) strength value is comparable to that of ordinary portland cement concrete designed to the same specifications.

Strength development of concretes with and without high-calcium fly ash ( $\text{CaO} = 30\%$ ) is examined, implementing a simple replacement method. It is found that the rate of strength development of fly ash concrete is comparable to that of the control concrete with or without fly ash [27]. During another study, examining the effects of Class C fly ash, it is observed that the strength development has similar trends for all fly ash replacement percentages under all curing conditions and it is further found that fly ash replacement reduces compressive and tensile strength in all cases [28]. A typical graph of rates of strength gain of fly ash concrete compared to OPC is given in FIGURE 2.1. Examining the effects of Class C (high-calcium) fly ash in concrete it is reported that results showing some increases in compressive strength values are obtained. It should be noted that in this study, the mix-proportioning approach used comprised replacement of fine aggregate by volume, the mass of cement and coarse aggregate being kept constant for each series of determinations [26].

Bilodeau and Malhorta concluded that high-volume fly ash (Class F) concrete with adequate early-age strengths can be produced with cement and total cementitious material as low as 125 and 300  $\text{kg/m}^3$ , respectively [24]. They have also found that the reference concretes exhibits noticeably higher compressive strengths than the fly ash concretes at all ages, and for each  $W/(C+FA)$ . Rapid strength development for the reference concrete between 1 and 7 days compared to that for fly ash concretes was observed. The reference concretes also show higher rates of strength development than the fly ash concretes between 91 days and one year for  $W/(C+FA)$  of 0.31 and 0.27. However, the fly ash concretes developed strength at a noticeably higher rate than the reference concrete between 7 and 28 days for each as presented in TABLE 2.3. and FIGURE 2.2.

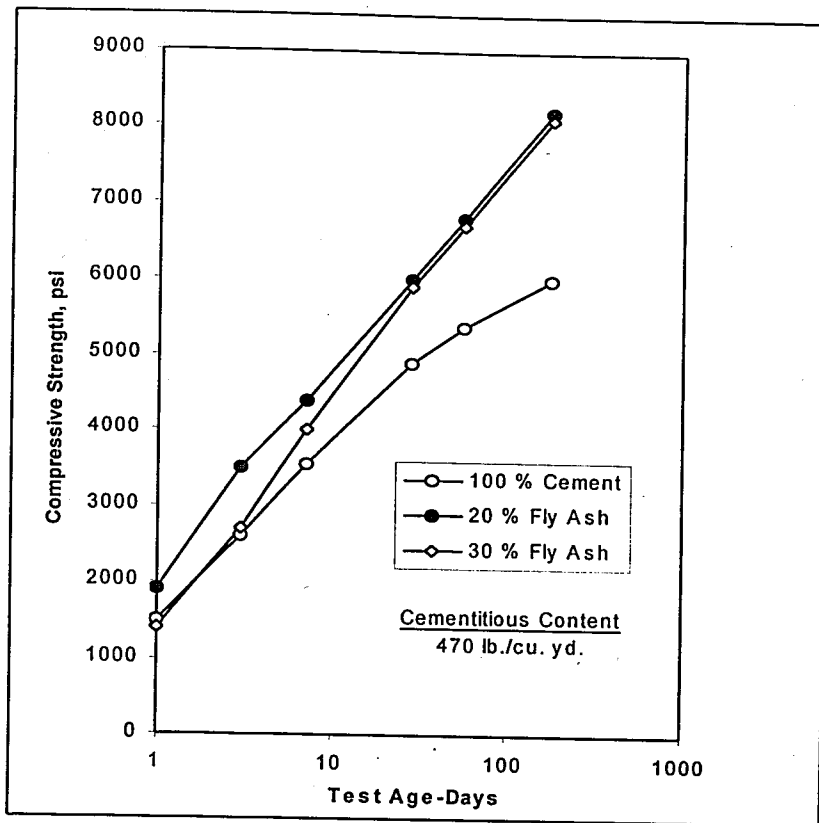


FIGURE 2.1. - Rates of strength gain of portland cement concrete and concrete with fly ash in different percentages [16].

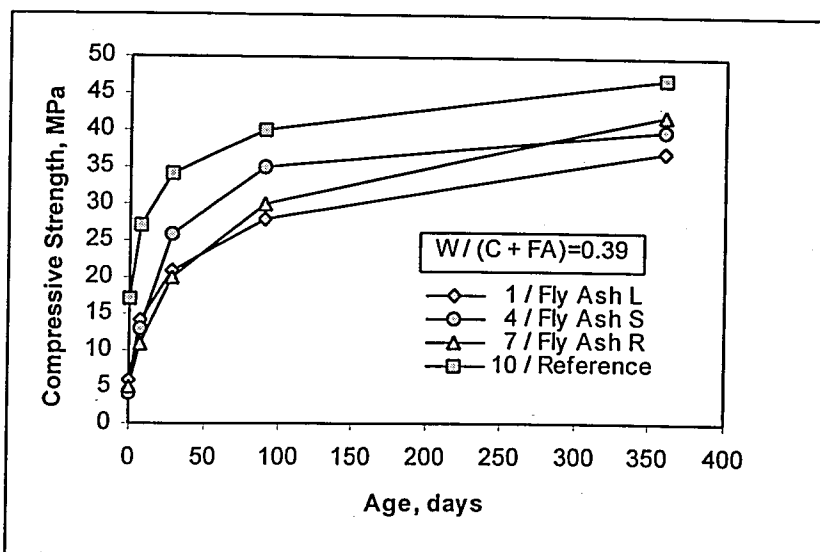


FIGURE 2.2. - Compressive strength development of fly ash concrete [24].

TABLE 2.3. - Compressive strength at different ages of fly ash concrete [24].

Mixture No.	W/(C+FA)	Cement, kg/m <sup>3</sup>	Fly ash,		Compressive strength, MPa				
			Source	kg/m <sup>3</sup>	1 d	7 d	28 d	91 d	365 d
					Batch A	Batch A	Batch A	Batch A	Batch A
1	0.38	126	L	173	3.9	11.2	21.1	30.1	39.4
2	0.31	156	L	215	6.8	16.0	28.0	37.1	47.1
3	0.27	180	L	248	8.6	20.5	27.5	44.1	51.9
4	0.39	124	S	172	3.2	13.2	33.7	35.9	41.1
5	0.31	155	S	214	6.5	22.0	36.9	46.0	51.4
6	0.27	182	S	251	9.9	26.4	41.0	50.3	56.8
7	0.39	124	R	170	3.9	11.2	20.2	30.9	42.7
8	0.31	155	R	213	7.0	17.0	29.3	41.2	51.4
9	0.27	181	R	250	9.2	20.9	34.8	44.2	55.0
10	0.39	292	-	0	16.4	27.8	34.6	40.3	47.1
11	0.31	368	-	0	27.8	43.3	55.1	66.4	78.9
12	0.27	428	-	0	37.2	51.2	61.3	71.8	83.7

Özyıldırım and Halstead has shown that control specimens under different curing conditions generally showed higher compressive strength values than concretes containing Class F fly ash [23]. Their results are presented in TABLE 2.4.

In a study by Nasser and Lai, it is revealed that generally all concretes with Class C fly ash showed a loss in strength at the age of 7 days. However, with an increase in age, concrete with 20 to 35% fly ash gave a 28-day strength similar to that of the control specimens while concrete with 50% fly ash suffered a reduction in strength of about 30% of the control concrete [29].

A study on flexural strength of Class F fly ash concrete showed that in general the flexural strength values follow the same trend as compressive strengths. With minor exceptions, the plain portland cement concretes exhibit somewhat higher flexural strengths at all ages for a given W/(C+FA) [23]. The results are shown on TABLE 2.5.

TABLE 2.4. - Compressive strength data for different curing temperatures [23].

C/FA/SF	Cured at 6 C			Cured at 23 C			Cured at 38 C		
	1 d	7 d	28 d	1 d	7 d	28 d	1 d	7 d	28 d
100/0/0	4.14	26.61	37.85	18.68	29.58	36.40	22.34	25.58	38.13
65/32/3	1.65	18.96	25.44	10.07	20.48	35.03	13.31	28.68	41.30
65/26/9	3.93	17.44	22.75	10.82	23.72	35.85	17.17	32.96	36.89
55/40/5	1.17	12.27	18.27	7.58	17.65	32.13	12.00	26.89	38.96
55/35/10	1.52	11.58	17.93	9.51	19.03	36.34	16.62	33.44	34.96

TABLE 2.5. - Compressive strength at different ages of fly ash concrete [24].

Mixture No.	W/(C+FA)	Cement, kg/m <sup>3</sup>	Fly ash,		Flexural strength, MPa			
			Source	kg/m <sup>3</sup>	14 d	91 d	365 d	91 d Modulus of Elasticity, GPa
1	0.38	126	L	173	2.2	4.0	4.7	37.3
2	0.31	156	L	215	2.8	4.3	4.8	40.4
3	0.27	180	L	248	3.8	5.0	5.0	43.7
4	0.39	124	S	172	3.9	5.7	5.7	39.1
5	0.31	155	S	214	3.7	5.9	5.9	40.8
6	0.27	182	S	251	-	5.3	5.3	40.3
7	0.39	124	R	170	-	4.4	4.4	38.2
8	0.31	155	R	213	-	4.5	4.5	40.7
9	0.27	181	R	250	-	5.6	5.6	40.7
10	0.39	292	-	0	4.5	5.3	5.3	40.3
11	0.31	368	-	0	6.3	7.5	7.5	45.1
12	0.27	428	-	0	7.2	7.1	7.1	46.2

### 2.2.2. Effect of Fly Ash on Elastic Properties, Creep, Shrinkage

The modulus of elasticity of fly ash concrete, as well as its compressive strength, is somewhat lower at early ages and a little higher at later ages than similar concretes without fly ash. The effect of fly ash on modulus of elasticity is not as significant as the effects of

fly ash on strength and durability. It should be noted that the cement and aggregate characteristics have a greater effect on modulus of elasticity than the use of fly ash [30].

It is also found that Young's modulus of elasticity, creep, and drying shrinkage of high-volume fly ash concrete are comparable to those of the plain portland cement concrete [24]. Modulus of elasticity results of concretes incorporating fly ash are presented in TABLE 2.6.

A study on drying shrinkage [24] revealed that the shrinkage strains were comparable for fly ash (Class F) and reference concretes and that expansion shrinkage strains of prisms at 448 days were insignificant for all high-volume fly ash concretes, whereas noticeable expansions occurred in the prisms cast from the reference concretes.

Another study by Tikalsky et al. [31] points out that concrete containing 35 percent Class F fly ash under hot-dry conditions has 28 percent less shrinkage than portland cement concrete after 120-days. Under moderate conditions, an 11 percent reduction was measured after 220 days. Class C fly ash concrete exhibited similar long-term shrinkage under hot-dry and moderate conditions as the control concrete without fly ash. The same study shows that concrete containing Class C fly ash showed a 5-percent reduction in creep compared to that of portland cement concrete without fly ash.

Similar research attributed the low creep strain of high-volume fly ash concrete to the unhydrated fly ash particles in the concrete acting as fine aggregate, and thus providing restraints against creep [24].

### **2.2.3. Effect of Fly Ash on Durability of Concrete**

Durability of portland cement concrete defined as its ability to resist weathering action, chemical attack, abrasion, or any other process of deterioration. Durable concrete will retain its original form, quality, and serviceability when exposed to its environment. In this respect, incorporating pozzolanic admixtures, fly ash in this context, is investigated for improved durability characteristics.

2.2.3.1. Capillary Absorption. Concrete is permeable to water to the extent that it has interconnecting void spaces. Permeability of concrete is governed by variables such as amount of cementitious material, water content, aggregate grading, consolidation, and curing efficiency.

Permeability of concrete is critical for it's the determining factor for the ingress of aggressive chemicals in addition to water. As the permeability is reduced the resistance of concrete against such deterioration is increased substantially. The incorporation of supplementary cementing materials such as fly ash, furnace slag, rice husk ash, silica fume, and natural pozzolans in concrete results in fine pore structure and changes the aggregate/paste interface, leading to a decrease in permeability.

The effect of fly ash on permeability is mainly due to its chemical reactions in the cement paste. Calcium hydroxide liberated by hydrating cement is water-soluble and may leach out of hardened concrete, leaving voids for the ingress of water. Through its pozzolanic properties, fly ash chemically combines with calcium, potassium, and sodium hydroxide to produce C-S-H, thus reducing the risk of leaching calcium hydroxide. As a result permeability is reduced. [14]. Moreover, the reduced permeability of fly ash concentrate can decrease the rate of ingress of water, corrosive chemicals, and oxygen.

According to the findings of E. Güneysi [32], concrete specimens containing 10% fly ash showed 52 to 65% decrease in sorptivity with respect to the reference concrete without fly ash. The decrease was much more significant when fly ash was incorporated along with silica fume.

2.2.3.2. Chloride Ion Permeability. The ingress of chlorides into concrete can have a major destructive effect on reinforcing steel, and is one of the most serious problems encountered in reinforced concrete structures worldwide. A recent report for the Department of Transport concluded that over 75% of concrete bridges in England and Wales might be affected by chloride attack [33]. In a study investigating chloride permeability of Class F fly ash concrete it is been found that the high-volume fly ash concrete shows excellent resistance to chloride-ion penetration and outperforms plain portland cement concrete. The total charge in coulombs at 91 days, a measure of the

resistance to the chloride-ion penetration determined by AASHTO T277-83 ranges from 278 to 1078. The corresponding values for reference concrete range from 1003 to 2313. The extension of the curing period from 28 to 91 days in this study reduced the total charge passed through all concrete, with the difference much more marked for the fly ash concretes than for plain portland cement concrete [24].

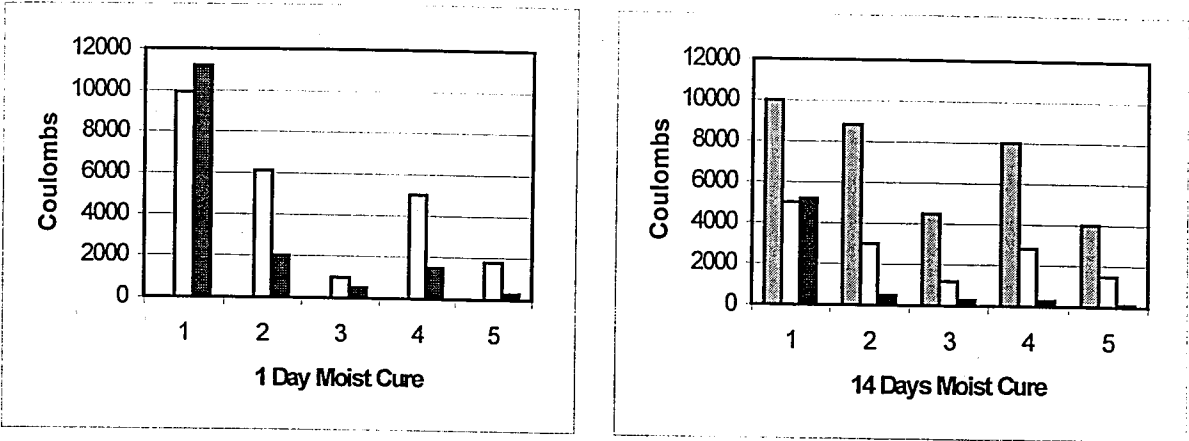
In a similar study by Özyıldırım and Halstead [23], the results show that, in all cases, when cured under the same conditions, concretes with Class F fly ash had a lower coulomb value than the control concretes made with only portland cement. FIGURE 2.3. [23] illustrates the results of this study. Moist curing for 14 days reduced the chloride permeability (lowered coulomb values) in all cases. Increasing the curing temperature from 23 C greatly decreased the coulomb values in specimens containing fly ash, but effects on the control specimens were minimal. When moist cured at 38 C for either 1 or 14 days, all specimens with fly ash had a coulomb value less than 1000, indicating very low permeability. Specimens moist cured for 2 weeks at 6 C generally had a coulomb value in the high permeability range. These results are presented in TABLE 2.6.

TABLE 2.6. - 9 and 28 days chloride permeability data, coulombs [23].

C/FA/SF	Moist-cured, 1 day		Moist-cured, 14 days		
	23 C	38 C	6 C	23 C	38 C
100/0/0	9820	11150	10300	5340	5560
65/32/3	6270	1970	8940	3240	500
65/26/9	990	500	4410	1200	280
55/40/5	5180	1440	7950	2880	320
55/35/10	1740	310	3960	1390	110

In another study investigating chloride diffusion coefficients of concretes [33] it is found that both the potential difference (PD) and the concentration difference (CD) methods show lower diffusion values for concretes containing fly ash.





batch no.	mixture		
	cement (%)	fly ash (%)	silica fume (%)
1	100	0	0
2	65	32	3
3	65	26	9
4	55	40	5
5	55	35	10

6 °C  
 23 °C  
 38 °C

FIGURE 2.3. - Chloride permeability of concretes at 28 days cured at different temperatures.

2.2.3.3. Carbonation. Carbonation occurs through the following process; first calcium hydroxide and alluminates in hydrated portland cement react in a moist environment with carbon dioxide from the atmosphere to form calcium carbonate. This process is observed in all portland cement concretes. The factors determining the rate of carbonation are permeability of concrete, degree of saturation with water, and the mass of calcium hydroxide available for reaction. Proper curing and compaction at a low (W/C) ratio might be sufficient to form an impermeable concrete and eliminate carbonation to insignificant levels.

However, fly ash can be used to solve the question from a different approach. Fly ash reacts with the calcium hydroxides available in the paste therefore eliminates carbonation reactions to a certain extent. According to Ho and Lewis [34], concrete containing fly ash showed a significant improvement in quality when curing was extended from 7 to 90 days. With a further curing to 90 days, concrete containing fly ash showed a slower rate of carbonation as compared to plain and water-reduced concretes.

2.2.3.4. Sulfate Resistance. Concrete can be severely damaged by the reactions of water-soluble sulfates. This occurs as the following; first sulfate reacts with  $\text{Ca(OH)}_2$ , commonly known as lime, present in the paste to form calcium sulfate (gypsum), then gypsum reacts with  $\text{C}_3\text{A}$ , one of the main constituents of cement, and forms calcium sulfoaluminate. This reaction yields products of greater volume than those of the original reactants, resulting in expansion and finally, this expansion deteriorates the concrete paste [35].

Fly ash is generally accepted to improve sulfate resistance. The increase in sulfate resistance for some is believed to be due in part to the continued reactions of fly ash with hydroxides in concrete to continue form additional calcium silicate hydrates (C-S-H), which fills in capillary pores in the cement paste, reducing permeability and the ingress of sulfate solutions.

2.2.3.5. Resistance to Freezing-Thawing Cycles. Concrete has long been known to be vulnerable to frost attack. As the temperatures of saturated hardened concrete is lowered, the water held in the capillary pores in the cement paste freezes in a manner similar to the freezing of capillaries in rock, and expansion of the concrete takes place. If subsequent thawing is followed by re-freezing, further expansion takes place, so that repeated cycles of freezing and thawing have a cumulative effect, and one can envisage an analogy between this and fatigue failure.

The resistance to damage from freezing and thawing of concrete made with or without fly ash depends upon the adequacy of the air-void system, the soundness of the aggregates, age of concrete, degree of hydration (maturity), strength of cement paste, and moisture condition of the concrete. Studies on air-entrained concrete, incorporating Class F and Class C fly ash, suggest that the concrete containing fly ash represent no greater risk from frost attack than the corresponding reference concrete without fly ash [36].

Evaluating freezing and thawing tests, Yuan and Cook [37] have reported that concrete with 20% Class C fly ash is more durable than control whereas air-entrained concrete with 50% fly ash exhibited worse performance than reference concrete without fly ash. It is worth noting, these are the final results after 1200 cycles and surprisingly, all of the fly ash concretes (containing 20, 30, and 50% fly ash) show better performance than

control concretes for the first 900 cycles and then 30 and 50% fly ash concretes exhibited a gradual decrease in performance till the 1200<sup>th</sup> cycle as presented in FIGURE 2.4. [36]

In a study on resistance of fly ash (Class C) concrete to freezing and thawing, Nasser and Lai [29] concluded that the use of high percentages of fly ash in concrete (35 and 50%) reduced its resistance to freezing and thawing even though it contained about 6% air and was cured in water for 80 days. However, concrete containing 20% fly ash gave satisfactory performance provided its air content and strength were comparable to control concrete, which contained no fly ash.

Results from the SEM examination show that the decrease in resistance of fly ash concrete to freezing and thawing may be due to the slow migration of Portlandite (microcrystalline  $\text{Ca}(\text{OH})_2$ ) and fibrous ettringite hydrate crystals from the dense C-S-H zones to air voids during cycling. Concrete with fly ash was less susceptible to gradual displacement of Portlandite to air voids because it contained less Portlandite in its structure, but its air voids contained more fibrous hydrates displaced from the surrounding dense C-S-H, which may have led to an increase in paste porosity [29].

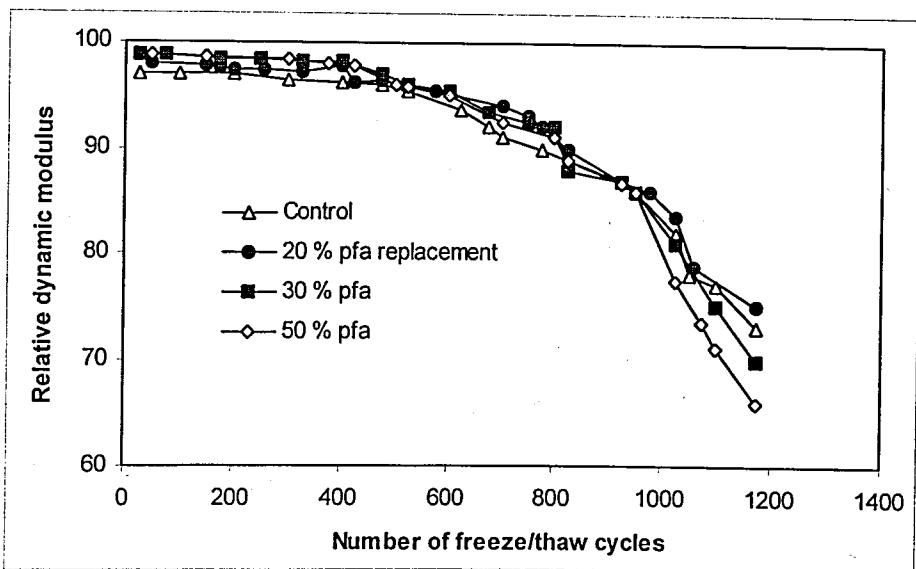


FIGURE 2.4. - Comparison of freeze/thaw test data between OPC and FA concretes [36]

2.2.3.6. Resistance to Deicing Salt Scaling. In the case of road slabs, frost not only affects the durability of concrete but leads also to the use of deicing salts, which exert an adverse effect on concrete by increasing the severity of the frost action. The salts commonly used are NaCl and  $\text{CaCl}_2$  and their repeated application with intervening periods of freezing or drying results [38] in surface scaling of the concrete. The salts produce osmotic pressure and cause movement of water toward the top layer of the slab where freezing takes place.

One action of the deicers is to enhance the corrosion of steel [38]. The deicer melts the snow or ice, which is often ponded by adjacent ice the resulting liquid is absorbed and, because of its lowered freezing point, remains liquid. As more ice melts, the melt water becomes diluted until its freezing point rises to near the freezing point of water. Freezing then occurs. Thus, freezing and thawing occur as often as without the use of deicers, or even more often since a possibly insulating layer of ice has been destroyed. In consequence, deicers can be said to increase saturation, possibly to increase the number of cycles of freezing and thawing, and to promote corrosion of steel.

The main effect of fly ash in concrete for resistance to deicing salt scaling is the lowered permeability for the ingress of salty water to initiate the deterioration process described above. The critical factor in the performance of fly ash concrete for deicing salt scaling resistance is proper curing and finishing and nearly all research takes these factors into consideration when evaluating the results.

According to a study by Marchand et al. [39] on deicing salt scaling resistance of roller-compacted pavements, incorporating Class C fly ash in concrete has noticeably increased the deicing salt scaling resistance of pavements.

Surprisingly, the use of fly ash (Class C), in contradiction with results obtained in the previous study, did not increase the durability of RCC in another study by the same research group [40]. The poor durability of the fly ash concretes was noted as unexpected, particularly considering the good compressive strength of these mixtures. The discrepancy between the two series of results was attributed to variations in the material properties, even if the fly ash was from the same source as that in the previous tests. Fly ash properties are known to vary significantly from one period of the year to another. A slight

modification in the particle size distribution of the ash could, for instance, significantly modify the structure of the cement paste, and subsequently alter the frost durability of RCC mixtures, without necessarily reducing the compressive strength which is influenced by the mineralogical composition. It is also possible that the surface of the fly ash mixtures was more affected by the compaction operations than other parts of the test sections [40].

Bilodeau and Malhotra [24] observed relatively poor performance for high-volume Class F fly ash concrete under deicing salt scaling and abrasion tests. In this study, final visual evaluation of the test slabs subjected to the deicing salts scaling test show that all high-volume fly ash concretes performed poorly and the reference concretes performed better than the fly ash concretes.

As mentioned in the Manual of Concrete Practice [16], application of deicers were observed by Perenchio and Klieger to cause a slightly higher loss of surface mortar from the concrete containing fly ash. This loss of mortar is confined to a thin top surface and has not been shown to be neither progressive nor related to internal disruption. It may represent, in part, sub-optimum finishing or curing of the surface of the concrete containing fly ash, a difference in carbonation due to a lesser amount of calcium hydroxide in the paste, or both.

### **2.3. Effect of Curing on Fly Ash Concrete**

#### **2.3.1. Effect of Different Curing Types on Durability of Fly Ash Concrete**

Availability of moisture and ambient temperature poses a critical role in the strength development and durability properties of concrete, either with or without fly ash. Here strength development and durability properties are mentioned together since they both are determined by the chemical reactions in the paste, which are largely affected by the curing conditions. This concept of proper curing is of greater importance for fly ash concrete and is been investigated for a long time. This is mainly due to the fact that pozzolanic reactions active in fly ash concrete are slow to start, and therefore in the

absence of adequate availability of moisture the strength development from the source is accordingly limited.

Under higher early curing temperature fly ash is shown to develop higher strength for all ages. For example, fly ash concrete exhibits higher strength values when cured above 20 °C than the values attained below 20 °C [28]. This beneficial effect of early high-temperature curing on pozzolanic reactions of fly ash is well suited for precast structural concrete applications.

Generally fly ash concrete is considered to be more sensitive to poor curing than portland cement concrete without fly ash since the pozzolanic reactions of fly ash fails to contribute to the development of strength when concrete is inappropriately cured. In this respect, prolonged curing as well as higher curing temperatures are critical factors in curing fly ash concrete [28].

Idorn [25] has suggested that, in general, fly ash reaction with portland cement in modern concrete is a two-stage reaction. Initially, and during the early curing, the primary reaction is with alkali hydroxides, and subsequently the main reaction is with calcium hydroxide. This phase distinction is not apparent when research is conducted at room temperature, at room temperature the slower calcium-hydroxide activation prevails and the early alkali activation is minimized. As was shown to be the case for portland cement by Verbeck [41], the pozzolanic reaction of fly ashes with lime and water follows Arrhenius' law for the interdependence of temperatures and the rates of reaction. An increase in temperatures causes more than a proportionate increase in the reaction rate.

However, the activation energy of fly ash is different from the activation energy of portland cement [16]. The concept of activation energy includes the energy needed for the reactants, mechanical energy from fine grinding, and thermal energy from the hydration or an outside heat source. Therefore, calculations of rates of strength development in concrete during the early curing phase require calorimetric-based determinations of the rate of heat development with the particular fly ash-portland cement combinations [25].

The behaviour of high-lime ashes (Class C) is sensitive to temperature: specifically, in mass concrete when a rise in temperature occurs, the products of reaction may not be of high strength. However, the development of strength is not simply related to temperature, being satisfactory in the region of 120 to 150 °C, but not at about 200 °C when the products of reaction are substantially different [38].

After the rate of strength contribution of portland cement slows, the continued pozzolanic activity of fly ash contributes to increased strength gain at later ages if the concrete is kept moist; therefore, fly ash concrete with equivalent or lower strength at early ages may have equivalent or higher strength at later ages than concrete without fly ash [42]. This higher rate of strength gain will continue with time and result in higher later age strengths that can be achieved by using additional cement. The extension of the curing period from 28 to 91 days in another study is observed to reduce the total charge passed through all concrete, with the difference much more marked for the fly ash concretes than for plain portland cement concrete [24].

Increasing the curing temperature from 23 °C greatly decreased the coulomb values in specimens containing fly ash, but effects on the control specimens were minimal. When moist cured at 38 °C for either 1 or 14 days, all specimens with fly ash had a coulomb value less than 1000, indicating very low permeability. Specimens moist cured for 2 weeks at 6 °C generally had a coulomb value in the high permeability range (see TABLE 2.7) [23]. The deterioration of the specimens, under scaling tests, cured with a membrane forming curing compound is found to be always less than that of water-cured companion specimens containing Class F fly ash [40].

According to Özyıldırım and Halstead [23] for Class F fly ash concrete, because the improved properties depend greatly on the reduced permeability of concrete resulting from the pozzolanic reaction that occurs over time, such concretes are subject to delays in developing low permeabilities under low temperatures or conditions where limited moisture is present. Strength development is also affected by the above criteria. Their results also demonstrate a positive effect of increased temperature from 23 to 38 °C on the behaviour of concretes containing fly ash. However, the adverse effect of cold temperature (6 °C) on the development of strength of concretes with fly ash requires attention.

In study investigating the effect of different curing conditions on the flexural and compressive strength of Class C fly ash concrete [31] (presented in FIGURE 2.5.) it is been found that for all curing conditions studied, including hot-dry conditions (38 °C, 32 percent relative humidity), moist-cured (24 °C, 100 percent RH), and cold cured (4 °C, 55 percent RH), concrete containing Class C fly ash showed lower flexural and compressive strength than similar concrete without fly ash under same curing conditions. It is further noted that the pozzolanic reaction in concrete containing fly ash will realize greater strengths at ambient temperatures when a moist environment is present; the membrane-curing compound used was an inadequate moisture barrier for concrete containing fly ash.

In a study on long term strength of high fly ash concretes Hansen [43] concludes that high fly ash concretes continue to gain considerable strength beyond 28 days when cured in water at 20 °C. However, in this study the concrete strength development came to an almost complete stop after 3 ½ years.

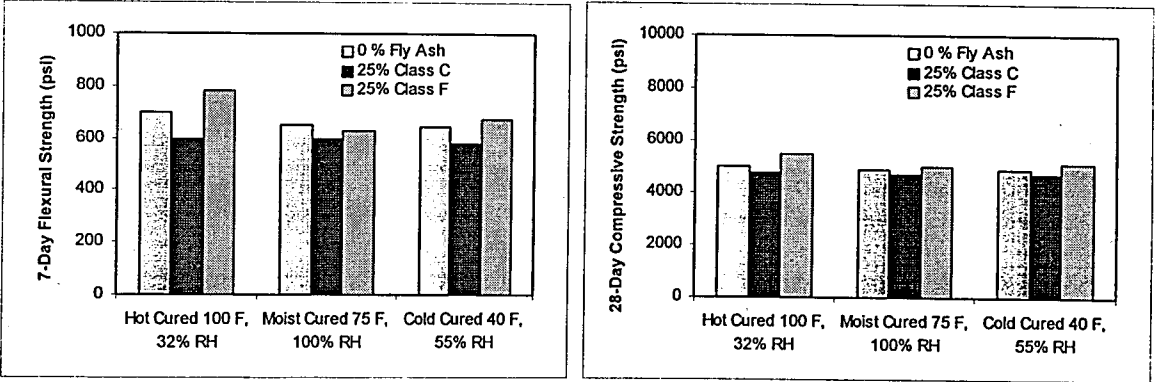


FIGURE 2.5. – Compressive and flexural strength of concrete with fly ash under different curing conditions.



In another study investigating chloride diffusion coefficients of concretes [33] it is found that both the potential difference (PD) and the concentration difference (CD) methods show lower diffusion values for concretes containing fly ash. The effect of different curing types exhibit that the lowest diffusion values are observed for the fly ash concrete specimens cured in 20 °C water for 28 days and then air cured for 3 months (E4), secondly specimens cured in 20 °C water for 3 days followed by air curing (E1), thirdly specimens air cured for 28 days (E2), and lastly the highest values of diffusion are for specimens air cured for 28 days (E3) [33] as illustrated in FIGURE 2.6 [33].

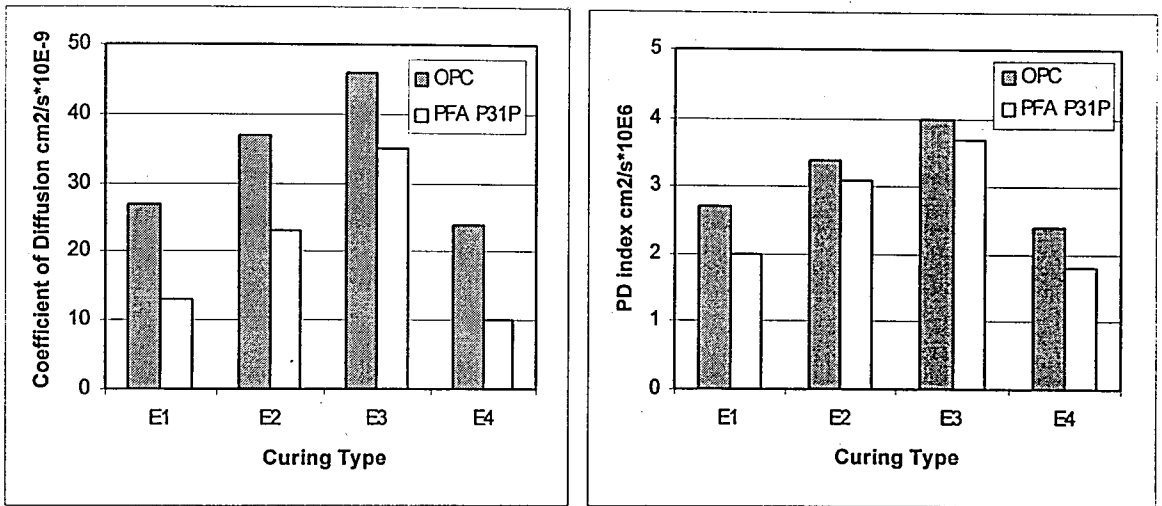


FIGURE 2.6. - Effect of curing on coefficient of diffusion and PD index values.

Nasser and Lai [29] unexpectedly noted that prolonged curing did not improve durability of concrete containing Class C fly ash and that concrete incorporating 35 to 50% fly ash was susceptible to frost resistance even though cured for 80 days.

According to a previous study [28] using the same mix proportions, same materials, and same three curing types (uncontrolled curing, moist-curing and hot-curing) as this study, it is reported that increased curing temperature generally increases tensile, compressive, and flexural strength of the specimens. This increase is much more noticeable in comparing concretes having high percentages (30 and 45%) of fly ash and cured in different conditions.

According to Ho and Lewis [34], based on a common 28-day strength, concrete containing fly ash showed a significant improvement in quality when curing was extended from 7 to 90 days. With a further curing to 90 days, concrete containing fly ash showed a slower rate of carbonation as compared to plain and water-reduced concretes.

In another research [16] done on freezing and thawing durability of concrete containing fly ash fog curing at 21 °C is found to be the most effective method for attaining highly durable concrete. The improvement in the durability performance of fly ash concrete was very significant in the case of prolonged curing (180 days). Simulated field conditions were also studied and once again proved that fly ash concrete is more susceptible to poor curing. The results are given in FIGURE 2.7.

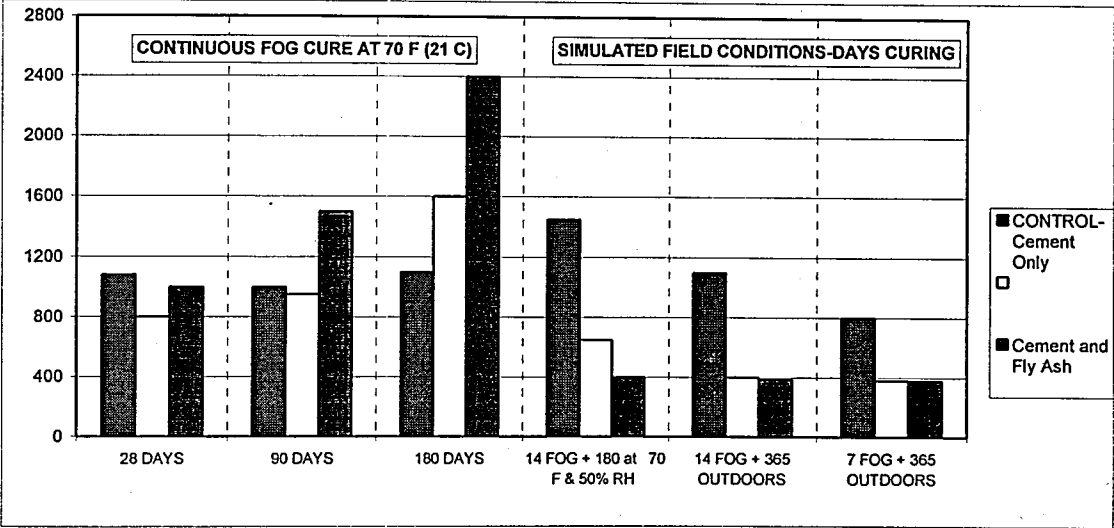


FIGURE 2.7. - Importance of extent of curing to durability of concrete with fly ash [16].

### **3. EXPERIMENTAL STUDY**

This study was carried out in order to investigate the effect of different curing conditions on the mechanical properties and durability of concrete containing different percentages of fly ash replacing cement. Control concrete specimens without fly ash were also produced as references. Three different curing conditions and four different fly ash percentages with other properties being unchanged were chosen in order to evaluate the performance of concrete with respect to various mechanical tests as well as durability tests.

The mechanical tests consisted of equivalent cube compressive strength, splitting tensile strength, and flexural strength whereas sorptivity, chloride ion permeability, deicing salt scaling, as well as freezing and thawing were implemented as durability tests. The results of these different tests for concretes containing fly ash were compared with the respective reference concretes containing only portland cement.

#### **3.1. Materials**

##### **3.1.1. Cement**

The cement used in this study is classified as ASTM Type I portland cement (PC 52.5). The data for the physical and chemical properties and rate of strength gain for this particular cement are given in TABLE 3.1.

TABLE 3.1. – Physical, chemical and strength properties of the cement

Analysis Report	Portland Cement (PC 52.5)
SiO <sub>2</sub> (wt. per cent)	21.1
Al <sub>2</sub> O <sub>3</sub> (wt. per cent)	4.7
CaO (wt. per cent)	63.71
Fe <sub>2</sub> O <sub>3</sub> (wt. per cent)	4.01
MgO (wt. per cent)	1.36
SO <sub>3</sub> (wt. per cent)	2.52
Insoluble Residue (wt. per cent)	0.43
Loss on Ignition (wt. per cent)	1.22
Specific Gravity (g/cm <sup>3</sup> )	3.12
Blaine Fineness (cm <sup>2</sup> /g)	3435
Initial Setting Time (hr)	2.45
Final Setting Time (hr)	3.25
f <sub>cc</sub> (1 day) (MPa)	15.7
f <sub>cc</sub> (2 days) (MPa)	26.4
f <sub>cc</sub> (7 days) (MPa)	45.8
f <sub>cc</sub> (28 days) (MPa)	60.8

### 3.1.2. Fly Ash

The fly ash used in this study is classified as Class C according to ASTM C 618. The ash has a lignite coal source used in the Soma Thermal Power Plant in the Aegean region of Turkey and it is this coal source which makes it Class C fly ash with a relatively high calcium content and cementitious properties. The specific gravity for this particular fly ash is 2.40 g/cm<sup>3</sup>. The properties of this fly ash are summarized in TABLE 3.2.

TABLE 3.2. - Chemical properties of the fly ash

Analysis Report	Class C Fly Ash
SiO <sub>2</sub> (wt. per cent)	50.48
Al <sub>2</sub> O <sub>3</sub> (wt. per cent)	23.74
CaO (wt. per cent)	9.28
Fe <sub>2</sub> O <sub>3</sub> (wt. per cent)	5.84
MgO (wt. per cent)	2.58
SO <sub>3</sub> (wt. per cent)	1.41
Cl <sup>-</sup>	0.02
Na <sub>2</sub> O	0.30
K <sub>2</sub> O	1.13
H <sub>2</sub> O	.....
CaCO <sub>3</sub>	16.50
Insoluble Residue (wt. per cent)	.....
Loss on Ignition (wt. per cent)	2.10

### 3.1.3. Aggregate

Fine aggregate used in this study is river sand (0-8 mm). Crushed basaltic stone obtained from Karatepe basalt quarry was chosen as coarse aggregate. Both of these aggregates were sieved, washed and surface dried before use. The maximum particle size in the mixture of fine and coarse aggregates was 31.5 mm. Sieve analysis and physical properties of the aggregates used in the concrete mix are presented in TABLE 3.3. The sieve analysis results of the final mix design are given in FIGURE 3.1.

TABLE 3.3. - Sieve analysis and physical properties of the aggregates

Sieve size (mm)	Sand	No. 10 Basalt	No. 20 Basalt
31.5	100.00	100.00	100.00
16.0	100.00	100.00	72.46
8.0	100.00	44.18	0.57
4.0	99.45	10.12	0.29
2.0	75.91	0.60	0.18
1.0	57.52	0.44	0.14
0.5	29.06	0.36	0.14
0.25	4.41	0.29	0.14
Fineness Modulus	2.14	5.30	6.18
Specific Wt. (Mg/m <sup>3</sup> )	2.60	2.90	2.90

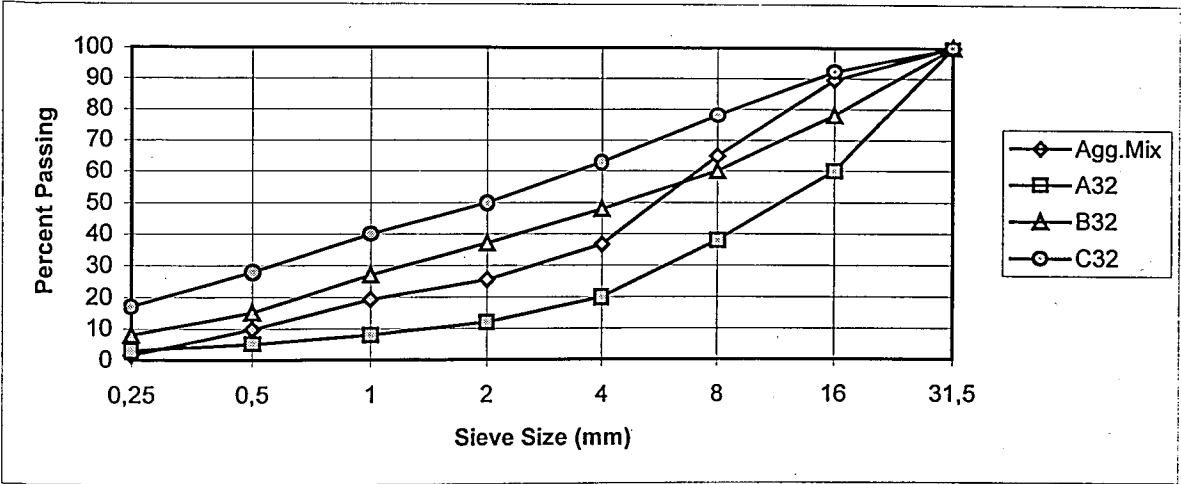


FIGURE 3.1. - Aggregate grading curve and zones (according to TS 707)

### 3.1.4. Superplasticizer

Melamine based SIKAMENT FF-N was used at 0.6 percent by weighth of cement. It has a specific gravity of 1.21 kg/ltr and other properties are given in the TABLE 3.4.

TABLE 3.4. - Specifications of the superplastizer ( SIKAMENT FF-N )

Type	Melamine based polymer dispersion
Color	Dark Brown
State	Liquid
Specific Gravity ( $\text{g/cm}^3$ )	1.21 – 1.22 kg/lt
Ph value	~ 9
Freezing Point	-4 ° C
Chloride Content	None
Nitrate Content	None

### 3.2. Concrete Mixture

During the experiments, a total of four different concrete mixtures have been produced. One of these was a control concrete without fly ash replacement. The other three had varied percentages of fly ash replacing portland cement in the mix. So the concretes tested had 0, 15, 30, and 45 percent fly ash replacement levels and other mixture contents are presented in TABLE 3.5.

It should be noted that the different concrete mixtures in this study are labelled according to a simple code. The letters FA stand for fly ash, the following two integers indicate the replacement percentage of cement by fly ash, and the last integer indicates the curing type applied to the specimen. In some of the tables this coding is not used and fly ash percentages are given explicitly. A constant water-cement ratio of 0.5 is used for all of the mixes. Total cementitious material (portland cement + fly ash) is also kept nearly constant at  $430 \text{ kg/m}^3$  for all concretes. Sikament FF-N was incorporated into the mixture, as a superplastizer, to attain a flowing consistency with a slump of  $21 \pm 2 \text{ cm}$ .

TABLE 3.5. - Mix proportioning of concretes in kg/m<sup>3</sup>.

Mix Code	Cementitious Materials		Water	W/C	W/ (C + FA)	Sand	Crushed Basalt Stone		S. P.*
	Cement	Fly Ash					Fine	Coarse	
FA001	430	0	214	0,50	0,50	669	562	564	2,15
FA002	430	0	214	0,50	0,50	669	562	564	2,15
FA003	430	0	214	0,50	0,50	669	562	564	2,15
FA151	365	65	215	0,59	0,50	658	553	557	2,2
FA152	365	65	215	0,59	0,50	658	553	557	2,2
FA153	365	65	215	0,59	0,50	658	553	557	2,2
FA301	300	130	214	0,71	0,50	652	548	550	2,11
FA302	300	130	214	0,71	0,50	652	548	550	2,11
FA303	300	130	214	0,71	0,50	652	548	550	2,11
FA451	235	195	214	0,91	0,50	640	539	540	2,12
FA452	235	195	214	0,91	0,50	640	539	540	2,12
FA453	235	195	214	0,91	0,50	640	539	540	2,12

\* S.P: superplastizer (Sikament FF-N)

### 3.3.Casting and Curing of Test Specimens

Each concrete mixture were produced in batches of 0.04 m<sup>3</sup> and mixing was done by power-driven revolving pan mixer in accordance with ASTM C 192. Four 100x200 mm cylinder moulds, and six 100x100x500 mm prism moulds were used for each batch of concrete mixture. Casting was done in three layers and each layer was compacted by a vibrating table for 10 seconds. All of the specimens were finished with a steel trowel after casting. Special attention was given to the finishing of prismatic specimens, prepared for deicing salt scaling and freezing and thawing tests, for its obvious significance in these tests. The finishing of the salt scaling specimens were done in accordance with related ASTM C 672. Since fly ash concrete typically has a longer time of setting than concrete without fly ash, such mixtures were finished at a later time than mixtures without fly ash. It



is reported that failure to do so could lead to premature finishing, which can seal the bleed water under the top surface creating a plane of weakness [26].

Plastic sheets were used to cover the specimens after finishing to prevent excessive moisture loss. The specimens were kept in laboratory conditions and demolded after 24 hours. After demolding the specimens are subjected to three different curing conditions;

1- In curing condition 1, the specimens were kept in the laboratory conditions after demolding till the time of testing. The relatively high humidity and low temperature environment of the laboratory is assumed as the uncontrolled curing condition.

2- In the second curing condition which is referred as moist curing, the specimens were put in 20°C water for 1 week after demolding. After this first week they were transferred to the curing room at  $20 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  R.H. and kept there till the time of testing.

3- In the third curing condition, the specimens were this time put in 30°C water for the first week after demolding and again transferred and kept in the curing room under same conditions as Curing 2. This type of curing will be referred as hot curing.

### 3.4. Test Procedures

#### 3.4.1. Fresh Concrete Properties

Fresh concrete properties, such as unit weight and slump value, were determined immediately after mixing. These measurements were done in accordance with ASTM C 138 for unit weight and ASTM C 143 for slump value determination. For each fresh mix characteristics such as workability, surface finishing, bleeding, and setting time were also noted for making comparisons between control and fly ash concretes.

#### 3.4.2. Hardened Concrete Properties

3.4.2.1. Mechanical Properties. Compressive strength, splitting tensile strength, and flexural strength are the parameters investigated as mechanical properties of the specimens.

Compressive and flexural strength values were also used to evaluate the effects of freezing and thawing tests on the specimens by comparing them with their references.

Splitting tensile strength determination was done on the top and bottom sections of 100x200 mm cylinder specimens which had their 50 mm thick middle section removed for chloride ion permeability tests. So, for each concrete mix four cylinder sections cut from two cylinder specimens were tested at the age of 28 and 56 days. The splitting tensile strength determination was carried out in accordance with the ASTM C 496. The tensile strength was calculated by the equation;

$$f_t = \frac{2P}{\pi DL} \quad (3.1)$$

where  $f_t$  is the tensile strength, in MPa;  $P$  is the maximum load applied, in N; and  $D$  and  $L$  the diameter and the length of the cylindrical specimen, in mm.

Flexural strength tests were performed on the 100x100x500 mm-prismatic specimens. In this test, two different sets of results were evaluated. First set of results belonged to the reference concretes, which were not exposed to freezing and thawing and deicing salt scaling tests. These reference specimens were tested when their counterpart, exposed to freezing and thawing and deicing salt scaling tests, has reached a predetermined deterioration rating given by ASTM C 672. These specimens were exposed to freezing and thawing cycles at ages of 28 and 56 days. When they had deteriorated to a certain extent, they were tested together with their same batch reference specimens on the same day and theirs constitute the second set of results. The flexural strength tests were carried out in accordance with ASTM C 293 and the flexural strength is calculated using following simple relation;

$$\sigma_f = \frac{Mc}{I} \quad (3.2)$$

where  $\sigma_f$  is the flexural stress, in MPa;  $M$  is the maximum moment (  $PL / 4$  ) in this case), in Nmm;  $c$  is the largest distance from the neutral surface (  $(h / 2)$  in this case), in mm; and

$I$  is the moment of inertia of the element ( $bh^3 / 12$ ) for beams), in  $\text{mm}^4$ . Here  $b$  is the average width of the specimen and  $h$  is the average depth of the specimen. These are constant for the reference concrete specimens whereas, the depth of those specimens, which were exposed to deicing salt scaling test, was slightly reduced due to apparent surface scaling. This reduction is taken into account when computing the flexural strength of these specimens.

As for the compressive strength determination, the same two sets of results are evaluated. Actually, the compressive strength determination was conducted on the portions of beams broken in flexure. This was done in accordance with ASTM C 116 Standard Test Method for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure. So, the equivalent cube strengths of beam portions broken in the above flexural strength tests were determined. The reference specimens and respective freezing and thawing specimens were first broken in flexure then tested as  $100 \times 100 \times 100$  equivalent cubes using bearing plates as required by the standard. Likewise, in the equivalent cube strength determination the scaled off surface was taken into account when calculating the cross-sectional area for compressive strength.

3.4.2.2. Capillary Absorption. Capillary absorption (sorptivity) test was carried out using the set up illustrated in FIGURE 3.2. Like splitting tensile test, water sorptivity determination was done on the top and bottom sections of  $100 \times 200$  mm cylinder specimens which had their 50 mm thick middle section removed for chloride ion permeability tests. So, for each concrete mix two cylinder sections cut from two cylinder specimens were tested at the age of 28 and 56 days.

Before testing, the lateral faces of the specimens are coated with paraffin in order to assure that the water was absorbed only through the bottom surface, but not the lateral surface. Then the specimens are placed on rods in a glass container filled with enough water as to cover the 5 mm level above the base of specimens. The cumulative water absorbed was recorded as the specimens were weighed at specific time intervals up to 2 hours. Before weighing the surface water on the base of the specimens were removed using a damp piece of cloth. Finally, the results were plotted in a graph of absorbed water versus

square root of time and the slope (S) of that graph was found which in turn gave the sorptivity (diffusion coefficient (D)) by the following relation;

$$D = S^2 \text{ (m}^2 \text{ / min)} \quad (3.3)$$

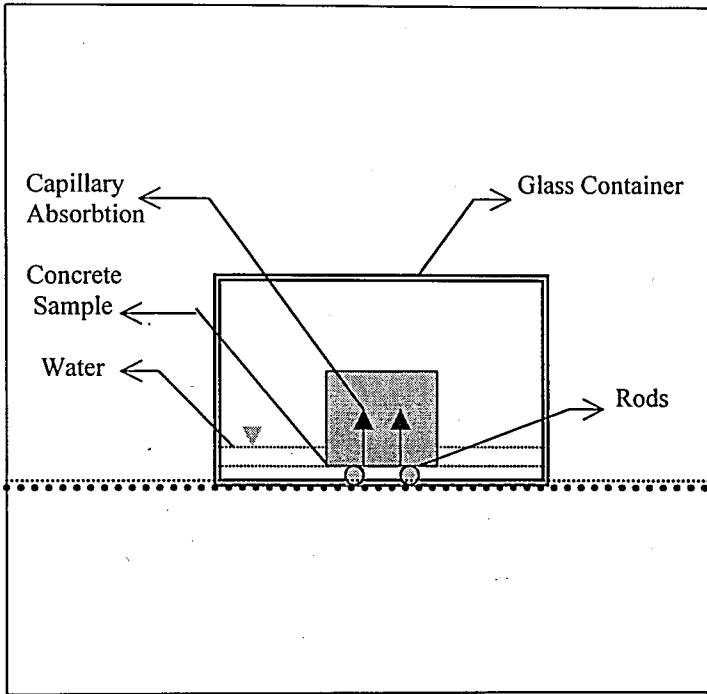


FIGURE 3.2. - Experimental setup for sorptivity test.

3.4.2.3. Chloride Ion Permeability. In this stage of the study, the chloride permeability of the concrete specimens was determined as a measure of concrete's durability performance. For this purpose the rapid chloride permeability method described by AASHTO T227 [44] or ASTM C 1202 [45] was conducted. The experimental setup for the rapid chloride permeability test is given in FIGURE 3.3.

The chloride ion permeability of the specimens was measured at the age of 28 and 56 days. For each batch of concrete, two specimens were tested at the same time for their rapid chloride permeability. There were a total of four cylindrical 100x200 mm specimens cast for each mixture and two of these were tested at 28 and the other two was tested at 56 days. Two 50 mm thick specimens were cut from the mid-section of each cylinder. The specimens were then allowed to surface dry in air. In order to prevent evaporation of water from the saturated specimen a rapid setting coating was applied onto the lateral surface of the specimens. Then the specimens are subjected to a vacuum-saturation procedure for 1 hr. As a final stage of conditioning before rapid chloride permeability test, the specimens were kept immersed in water in the curing room at 20 C and 50 % R.H for  $18 \pm 2$  hr.

Following these conditioning procedures, the specimens were transferred to a test cell, as shown in FIGURE 3.3. After the specimens were fixed in position the positive electrode side of the cell (+) was filled with 0.30 N (1.2 percent) NaOH solution, while the negative electrode side (-) was filled with 0.50 N (3 percent) NaCl solution. Then the electrical connections of voltage application and data readout system were made and lead wires were attached to the cell banana posts. Then, a direct voltage of  $60.0 \pm 0.1$  V was applied across the two electrodes. Due to this applied voltage the chloride ions in the NaCl solution, being negatively charged, were attracted by the opposite positive electrode (+) and they penetrate through the pores of saturated concrete. The data acquisition system was adjusted to record the current passing through the specimens with 10 sec intervals over a 6 hours period (21600 sec). This current passing is interpreted as the result of penetration of chloride ions through the concrete. After 6 hours the test was terminated and the result were plotted in a current (amperes) versus time (sec) graph. The area under this curve was integrated to give the ampere-seconds, or coulombs, of charge passed through each concrete specimen during the 6 hours of testing. The total charge passed is evaluated as a measure of electrical conductance of the concrete during the test.

A basic classification rating given by AASHTO T 277 (or ASTM 1202) were used in interpreting the chloride permeability of different concretes based on rapid chloride permeability test results. The rating consists of five classes from "High" to "Negligible" on the basis of total coulomb value as given in TABLE 3.6.

TABLE 3.6. - Interpretation of results obtained using RCPT T 277-89 [44]

Charge Passed (coulombs)	Chloride Permeability	Typical of -
>4,000	High	High w/c ratio ( $< 0.6$ ) conventional portland cement concrete
2,000 – 4,000	Moderate	Moderate w/c ratio ( $0.4 - 0.5$ ) conventional portland cement concrete
1,000 – 2,000	Low	Low w/c ratio ( $< 0.4$ ) conventional portland cement concrete
100 – 1,000	Very Low	Latex-modified concrete, Internally sealed concrete
< 100	Negligible	Polymer-impregnated concrete, Polymer concrete

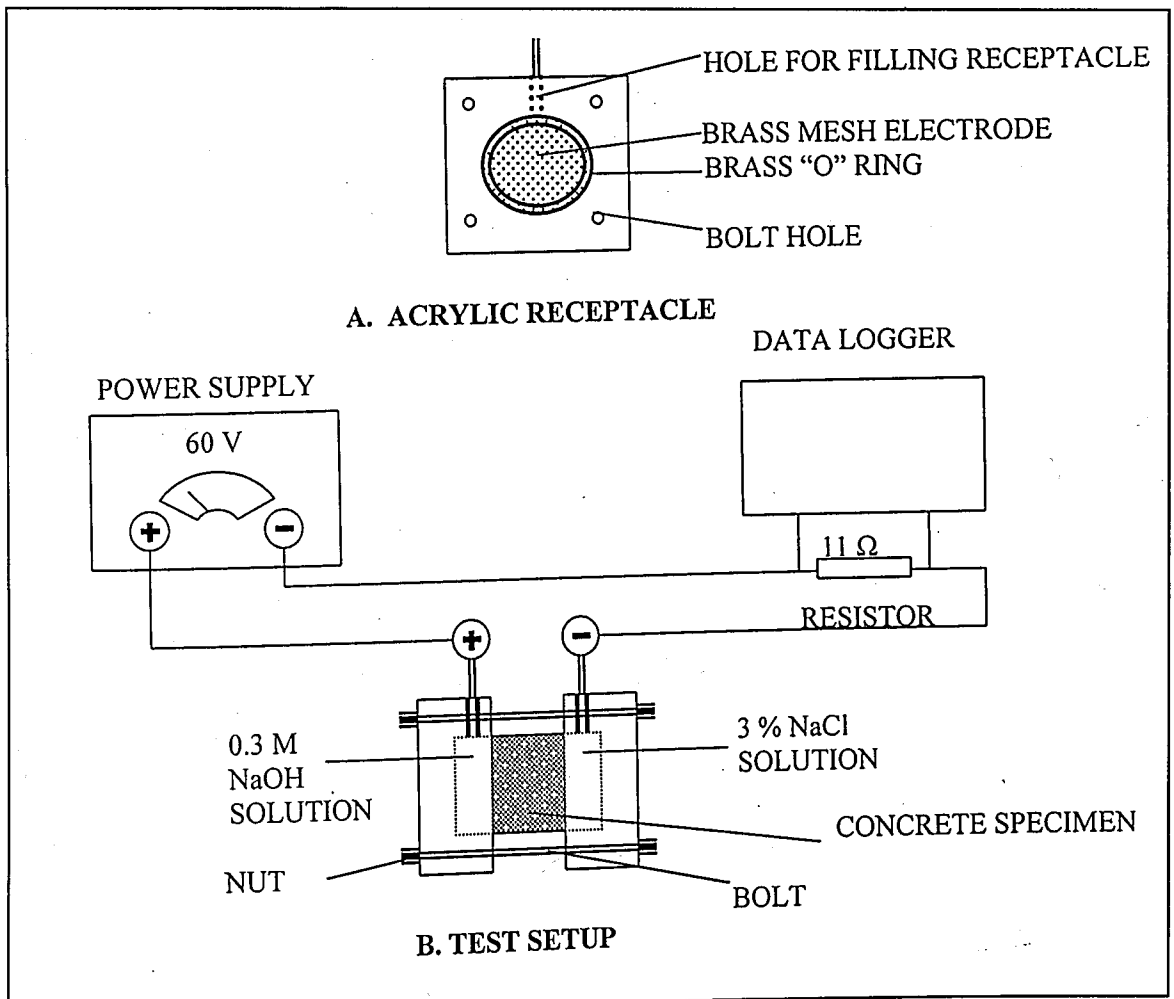


FIGURE 3.3.- Experimental set-up for Rapid Chloride Permeability Test

3.4.2.4. Deicing Salt Scaling Resistance. One of the other important aspects of concrete durability is scaling resistance. Although freezing and thawing tests done in accordance with ASTM Standard C 666 procedure A, do give indications of scaling resistance, the real test for scaling is that in the presence of deicer salts as suggested by ASTM Standard C 672. Scaling tests can be considered more practical because this problem is much more widespread and more common than the internal microcracking generated by rapid freeze-thaw tests [33]. For this reason, tests were conducted in accordance with ASTM C 672 (Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals).

The ASTM C 672 test was conducted on 100x100x500 mm prismatic specimens. A total of four specimens were casted for each concrete mixture. Two of these were exposed to the deicing salt scaling test at the ages of 28 and 56 days, while the remaining two were kept as reference specimens, one for each age. The standard suggests the placement of a dike about 25 mm wide and 20 mm high along the perimeter of the top surface of the specimen. This dike serves to maintain the brine pond (salt solution) on top of the specimen through out the period of the tests. The standard doesn't dictate the type of material to be used in forming the dike. It suggests mortar and epoxy mortar for this purpose, but due to practical reason which makes it necessary for the specimens to be stacked on top of each other in the limited freezing space, a more durable dike material has to be selected. So, fabricated galvanized frames of 50 mm depth were chosen as an appropriate dike material due to their durability and non-corrosiveness.

After surface finishing operation, the frames were inserted into the fresh concrete of the top surface of the specimens and the concrete was left to harden. After the relevant curing period (28 and 56 days), the flat surface of the specimen, surrounded by the galvanize frame was covered with approximately 6 mm of a solution of calcium chloride (CaCl) and water, having a concentration such that each 100 ml of solution contains 4 g of anhydrous calcium chloride (0.66 N). Before the placement of the salt solution, the inside perimeter of galvanized frame was coated with a thin line of silicone based paste to prevent the unwanted ingress of water through the interface between the frame and concrete. The drawing of the specimen with galvanized dike is shown in FIGURE 3.4. Then the specimens were placed in a freezing environment (at  $-18 \pm 3^{\circ}\text{C}$ ) for 16 to 18 hours. At the

end of this time the specimens were removed from the freezer and placed in curing room at  $23 \pm 1.7^{\circ} \text{C}$  and a relative humidity of 45 to 55% for 6 to 8 hours. Brine was added between each cycle as necessary to maintain the proper depth of solution. This cycle was repeated daily, flushing off the surface thoroughly at the end of each 5 cycles. The scaled off particles were collected and weighed after drying. After making a visual examination by taking photographs and notes, the solution was replaced and the test was continued. The specimens were kept frozen during the inevitable interruptions in the daily cycling.

The standard points out that generally 50 cycles are sufficient to evaluate a surface or surface treatment. However, where comparative tests are being made, it is recommended that the test be continued beyond the recommended minimum number of cycles if differences have not developed. In this study, 50 cycles have been found to be more than enough for making comparisons between normal and fly ash concrete, since all specimens apparently reached the final deterioration rating before the 50<sup>th</sup> cycle. The evaluation of the test results were made with the help of basic rating scale given by the ASTM C 672, shown in TABLE 3.7.

TABLE 3.7. - The rating scale for ASTM C 672 [46]

Rating	Condition of Surface
0	No scaling
1	Very slight scaling ( 3.2 mm depth, max, no coarse aggregate visible )
2	Slight to moderate scaling
3	Moderate scaling ( some coarse aggregate visible )
4	Moderate to severe scaling
5	Severe scaling ( coarse aggregate visible over entire surface )



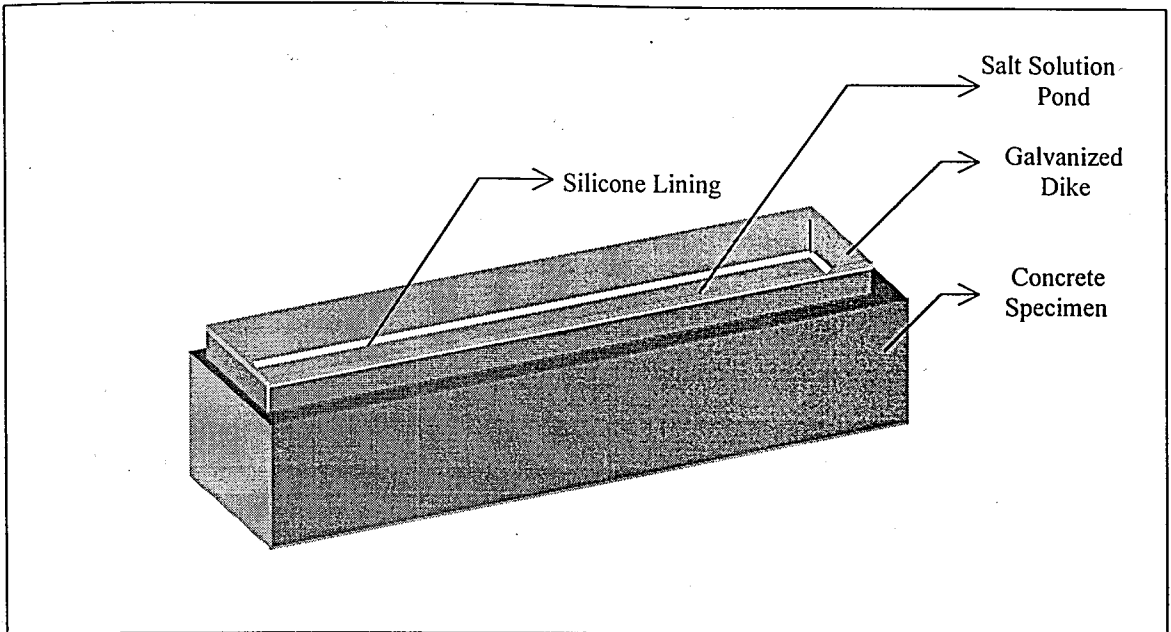


FIGURE 3.4. -The deicing salt scaling specimen with galvanize dike

**3.4.2.5. Freezing and Thawing Resistance.** In winter, concrete is exposed to temperature cycles where water freezes to ice and ice melts to water. This is known as freezing and thawing cycles. Concrete deteriorates due to this temperature cycling. In practice, concrete can be exposed simultaneously to freezing and thawing cycles and freeze salt. For example, deicing salts are used on roads and pavements. In marine environments, salt deposits are distributed by wind. In polluted areas, salts are formed when pollutant gases interact with the alkalis in concrete. Thus, it is not easy to divorce them from each other. Freeze-thaw deterioration occurs due to the expansion caused when water freezes to ice. When deicing salts are used, this deterioration rate becomes more severe. It is known as freeze-salt resistance. Resistance of concrete to freezing and thawing is determined by different test methods. Each test method yields different grades of degradation.

In a study comparing four of these different methods the following methods were used; [16];

- Freezing and thawing in 3 percent NaCl solution, similar to the Swedish standard SS137244, except that the samples were in contact with the salt solution from the bottom side, whereas in the Swedish standard, the salt solution is on top of the sample during testing.

- Freezing and thawing in 3 percent  $\text{CaCl}_2$  solution, similar to ASTM 672, except that the samples in this case were moist-cured for 5 days and then placed in a climate room

for 22 days at 55 percent RH and 20 C. Also, the samples were presaturated, whereas in ASTM they are moist-cured for 14 days and tested after 28 days without any presaturation.

- Freezing in saturated NaCl (30 percent) solution and thawing in water at room temperature.

- ASTM 666 B-84 (Procedure B), freezing at  $-17^{\circ}\text{C}$  and thawing in water at  $+4^{\circ}\text{C}$ .

And it was found that maximum deterioration occurred when 3 percent NaCl was used followed by 30 percent NaCl and 3 percent  $\text{CaCl}_2$ . Weight losses shown by ASTM 666 B-84 were almost negligible [16].

In this part of the study, the freezing and thawing resistance of fly ash concrete was also evaluated through freezing and thawing test in the presence of deicer salt (4 percent NaCl solution). Actually, deicer salt scaling and freezing and thawing tests were conducted simultaneously on the same specimens. It is worth noting that in this method the deicing salt affected only the top surfaces of the specimens. In the deicing salt scaling test 100x100x500 mm prismatic specimens were placed in a freezing environment (at  $-18 \pm 3^{\circ}\text{C}$ ) for 16 to 18 hours at the age of 28 and 56 days. At the end of this period the specimens were removed from the freezer and placed in the curing room at  $23 \pm 1.7^{\circ}\text{C}$  and a relative humidity of 45 to 55% for 6 to 8 hours. These freezing and thawing cycles were evaluated separately as a freezing and thawing test with the above cycle conditions.

The deicing salt scaling tests were terminated after the specimens have reached a certain deterioration rating due to surface scaling, so this coincided with the termination of the freezing and thawing test of the same specimen. Therefore, the total number of freezing and thawing cycles of each specimen was limited by the cycles of the deicing salt scaling test and never exceeded 50 cycles. Due to this relatively low number of cycles the effects of freezing and thawing were expected to be less significant. The freezing thawing resistance of the concrete specimens was evaluated by comparing the flexural and equivalent cube strengths of the specimens exposed to freezing and thawing test with the same batch reference specimens. Any visual deterioration on the lateral and bottom surface due to freezing and thawing cycles were also recorded.

## 4. TEST RESULTS AND EVALUATION

### 4.1. Fresh Concrete Properties

Fresh concrete properties such as slump and unit weight are given in TABLE 4.1. A slight decrease in slump value was observed as the fly ash replacement percent increased. The reference concretes (FA 00x) had an average slump value of approximately 220 mm while the fly ash concretes with 45% cement replacing fly ash had nearly 200 mm of slump. This slight decrease in slump may be considered immaterial; however, it was observed during the placement of fresh concrete into the moulds that fly ash concretes seemed to lose initial workability more quickly. This might be attributed to the initial water absorption by the finer fly ash particles. It is also noted however, that fly ash concretes have a distinguishable superiority in the finishability property, especially significant during the finishing operation of the 100x100x500 mm deicing salt scaling specimens.

As expected, the unit weights of the fly ash concretes were somewhat lower than those of normal concretes. The unit weights for reference concretes ranged from 2417 to 2450 kg/m<sup>3</sup> whereas the unit weight of the concretes with 45% fly ash replacement was low as 2350 kg/m<sup>3</sup> of unit weight. This is reasonable since the replacement of cement with fly ash implies the replacement of a denser material with a less dense material, which in turn decreases the overall density of the fresh mixture.

TABLE 4.1. - Properties of fresh concrete

Type of Concrete	W / ( C + FA )	Slump Value mm	Unit Weight kg/ m <sup>3</sup>	S. P. * Content %
FA001	0.5	220	2450	0.6
FA002	0.5	230	2417	0.6
FA003	0.5	210	2457	0.6
FA151	0.5	210	2416	0.6
FA152	0.5	220	2386	0.6
FA153	0.5	220	2417	0.6
FA301	0.5	200	2384	0.6
FA302	0.5	210	2403	0.6
FA303	0.5	210	2383	0.6
FA451	0.5	200	2351	0.6
FA452	0.5	200	2351	0.6
FA453	0.5	180	2354	0.6

\* S.P: superplastizer (Sikament FF-N)

## 4.2.Hardened Concrete Properties

### 4.2.1. Mechanical Properties

4.2.1.1. Splitting Tensile Strength. The results of the splitting tensile strength tests are given in TABLE 4.2. and shown in FIGURES 4.1.- 4.3. These values show the effect of curing condition and age on concrete specimens having different percentages of fly ash replacing cement and are obtained from two test specimens for each parameter. The general observations were as follows;

The splitting tensile strength values ranged from 2.33 to 1.70 MPa at 28 days and from 3.06 to 1.95 MPa at 56 days for specimens cured under Curing Condition 1. Likewise, the specimens subjected to Curing Condition 2 exhibited results ranging from 2.76 to 1.63 MPa at 28 days and from 3.45 to 2.66 MPa at 56 days. The 28-day and 56-

days splitting tensile strength values of the specimens cured under Curing Condition 3 ranged from 2.92 to 2.09 MPa and from 3.66 to 2.45 MPa respectively.

Considering the overall results, the splitting tensile strength values of those specimens having only portland cement are found to be higher than those incorporating fly ash for any curing condition. The curing conditions affected the splitting tensile strength and best results are observed for Curing Condition 3, followed by Curing Condition 2 and Curing Condition 1. Moreover, it was evident that the increase in the percentage of cement replacement by fly ash caused significant decreases in splitting tensile strength.

Generally, the splitting tensile strength showed an increase with age between 28 and 56 days under any given curing condition. This increase is somewhat more pronounced for Curing Condition 2 and less significant for Curing Condition 3 and 1. The results further indicate that the effect of curing condition was significant for high volume fly ash concrete. Generally, the splitting tensile strength showed a decrease with increased fly ash content and it seems that hot curing under Curing Condition 3, greatly decreases this negative effect of fly ash especially at 56 days. This improvement is clearly observed when Curing Condition 3 results are compared with the Curing Condition 2 and 1 results.

TABLE 4.2. – Splitting tensile strengths of concretes at 28 and 56 days and percent change in splitting tensile strength with respect to the reference and age

PC / FA	Curing Condition	$f_{s,28}$ MPa	Percent change with respect to the reference %	$f_{s,56}$ MPa	Percent change with respect to the reference %	Percent change with respect to the age %
100/0	1	2.33	0	3.06	0	31
85/15	1	2.23	-4	2.75	-10	23
70/30	1	1.73	-26	2.32	-24	34
55/45	1	1.63	-41	1.95	-36	19
100/0	2	2.76	0	3.45	0	25
85/15	2	2.18	-21	3.09	-11	42
70/30	2	2.18	-21	2.98	-14	37
55/45	2	1.70	-26	2.66	-23	56
100/0	3	2.92	0	3.66	0	25
85/15	3	2.43	-17	2.45	-33	1
70/30	3	2.08	-29	2.84	-23	37
55/45	3	2.09	-29	2.94	-20	41

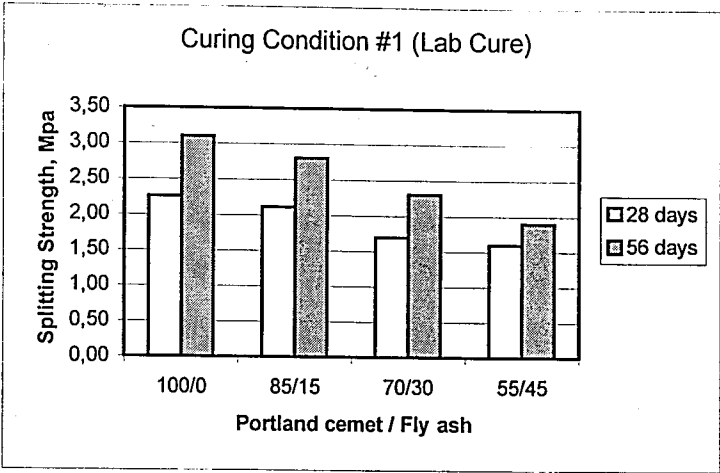


FIGURE 4.1. - 28 and 56 day splitting tensile strengths of concretes cured under Curing Condition 1 and having various percentages of fly ash and portland cement

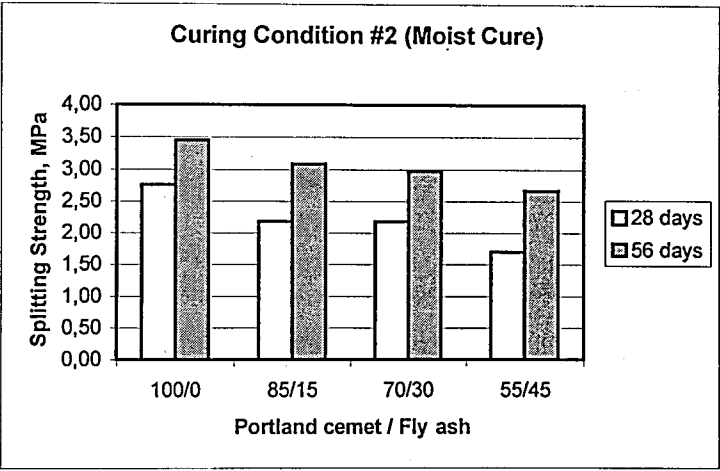


FIGURE 4.2. - 28 and 56 day splitting tensile strengths of concretes cured under Curing Condition 2 and having various percentages of fly ash and portland cement

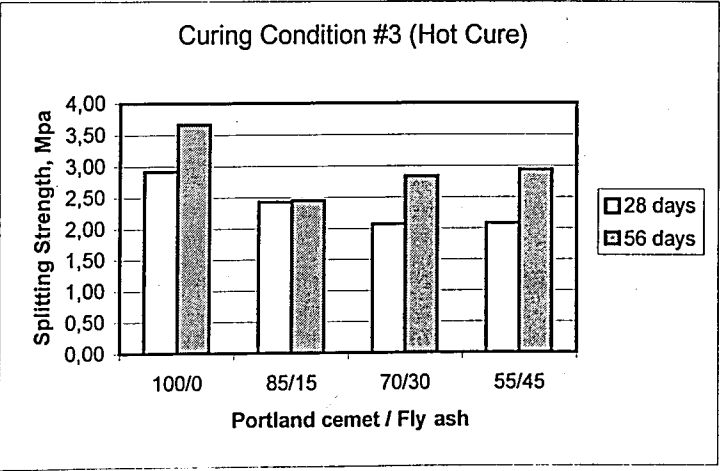


FIGURE 4.3. - 28 and 56 day splitting tensile strengths of concretes cured under Curing Condition 3 and having various percentages of fly ash and portland cement

4.2.1.2. Flexural Strength. Flexural strength determinations were performed on two sets of specimens at 28 and 56 days. First set of results belonged to the reference concretes, which were not exposed to freezing and thawing and deicing salt scaling tests. These reference specimens were tested when their counterpart, exposed to freezing and thawing and deicing salt scaling tests, has reached a predetermined deterioration rating given by ASTM C 672. The freezing and thawing specimens tested together with their same batch reference specimens after the termination of their tests provided the second set of results.

In this section only the results of the control specimens are evaluated in the scope of hardened concrete properties and the results of 28 days and 56 days control specimens are presented on TABLE 4.3. As presented in TABLE 4.3, the flexural strength results for control specimens, not exposed to freezing and thawing cycles (after 28 days) and cured under Curing Condition 1, range from 6.01 to 4.99 MPa. The results for the same set cured under Curing Condition 2 ranged from 6.64 to 5.82 MPa, and Curing Condition 3 specimens exhibited results ranging from 7.45 to 5.07 MPa.

These results obtained for 28 days specimens have a common trend; the addition of fly ash had negative effect on the flexural strength of concrete. The lowest results were reached for 45 percent cement replacement by fly ash in every set of specimens. This was an expected effect also observed by other researchers [16]. The negative effect of fly ash is observed to be less for specimens cured under Curing Condition 2 compared to the Curing Condition 1 and 3 specimens.

Similarly, the flexural strength results for control specimens, not exposed to freezing and thawing cycles (after 56 days) implies same kind of behaviour. The specimens cured under Curing Condition 1 showed results ranging from 5.89 to 4.23 MPa. The results for the same set cured under Curing Condition 2 ranged from 7.01 to 5.92 MPa, and Curing Condition 3 specimens exhibited results ranging from 7.54 to 6.03 MPa.



TABLE 4.3. – Flexural strength of control specimens of 28 and 56 days freezing and thawing specimens

PC / FA	Curing Condition	Control Specimens of 28 days F-T specimens $f_c$ Mpa	Percent change with respect to the reference %	Control Specimens of 56 days F-T specimens $f_c$ MPa	Percent change with respect to the reference %
100/0	1	6.0	0	5.9	0
85/15	1	5.5	-8	5.0	-14
70/30	1	5.2	-13	5.5	-6
55/45	1	4.9	-17	4.2	-28
100/0	2	6.6	0	7.0	0
85/15	2	6.7	1	6.7	-4
70/30	2	6.4	-3	6.5	-8
55/45	2	5.8	-12	5.9	-18
100/0	3	7.4	0	7.5	0
85/15	3	5.7	-24	6.9	-10
70/30	3	5.6	-25	7.3	-3
55/45	3	5.9	-33	6.0	-25

The same trend obtained for 28 days specimens can also be observed for the 56 days specimens; the addition of fly ash has negative effect on the flexural strength of concrete. The lowest results were reached for 45 percent cement replacement by fly ash in every set of specimens. The negative effect of fly ash is again observed to be less for specimens cured under Curing Condition 2 compared to the Curing Condition 1 and 3 specimens.

**4.2.1.3. Equivalent Cube Strength.** Compressive strength of concrete was assessed by the equivalent cube strength tests which were performed right after the flexural strength tests since the specimens used for equivalent cube strength determination were the portions of beams broken in flexure. As for the equivalent cube strength determination, the same two sets of results are evaluated; first the control specimens not subjected to freezing and thawing cycles, second the freezing and thawing specimens tested at the end of 28 and 56 days as given in TABLE 4.4.

TABLE 4.4. – Equal cube strengths of beam sections of control specimens broken after freeze-thaw cycles started at 28 and 56 days

PC / FA	Curing Condition	Control Specimens of 28 days F-T specimens $f_c$ Mpa	Percent change with respect to the reference %	Control Specimens of 56 days F-T specimens $f_c$ MPa	Percent change with respect to the reference %
100/0	1	50.4	0	54.2	0
85/15	1	50.2	0	52.5	-3
70/30	1	48.8	-3	44.3	-20
55/45	1	34.2	-32	38.7	-31
100/0	2	58.8	0	59.0	0
85/15	2	57.0	-3	57.2	-4
70/30	2	53.7	-9	55.9	-6
55/45	2	47.1	-20	48.1	-22
100/0	3	62.6	0	63.6	0
85/15	3	59.6	-5	60.2	-7
70/30	3	54.5	-13	54.8	-17
55/45	3	50.2	-20	52.0	-23

It is clear that the above results show a similar trend as the flexural strength results; there is a significant decrease of strength with the addition of fly ash instead of cement. As expected this effect is more distinguished for specimens cured under uncontrolled conditions. There is also an overall improvement with age between 28 and 56 days.

The equivalent cube strength values ranged from 50.4 to 34.2 MPa at 28 days and from 54.2 to 38.6 MPa at 56 days for specimens cured under Curing Condition 1. Likewise, the specimens subjected to Curing Condition 2 exhibited results ranging from 58.8 to 47.1 MPa at 28 days and from 59.0 to 48.1 MPa at 56 days. The 28-day and 56-days equivalent cube strength values of the specimens cured under Curing Condition 3 ranged from 62.6 to 50.2 MPa and from 63.6 to 52.0 MPa, respectively.

Considering the overall results, the equivalent cube strength values of those specimens having only portland cement are found to be higher than those incorporating fly ash for any curing condition. The curing conditions affected the equivalent cube strength and best results are observed for Curing Condition 3, followed by Curing Condition 2 and Curing Condition 1. Moreover, it was evident that the increase in the percentage of cement replacement by fly ash caused significant decreases in equivalent cube strength. Generally, the equivalent cube strength showed an increase with age between 28 and 56 days under any given curing condition. This increase is somewhat more pronounced for Curing Condition 2 and less significant for Curing Condition 3 and 1.

### 4.3. Capillary Absorption

The capillary absorption results given as diffusion coefficients are presented in TABLE 4.5. and FIGURES 4.4. - 4.6. The 28 days results ranged from  $2.0 \times 10^{-10}$  to  $2.0 \times 10^{-9}$  m<sup>2</sup>/min and the 56 days diffusion results ranged from  $1.0 \times 10^{-10}$  to  $1.8 \times 10^{-9}$  m<sup>2</sup>/min. Generally, the results reveal that there is a decrease in capillary absorption as the fly ash content increases for specimens cured under Curing Condition 3 and 2, where as just the opposite is observed for uncontrolled curing specimens. Overall evaluation shows that all specimens show a decrease in sorptivity with increasing age.

TABLE 4.5. - The diffusion coefficient data and percent change of the diffusion coefficient of concretes with respect to the reference and age

PC / FA	Curing Condition	Diffusion Coefficients, $D_{28}$  $m^2/min$	Percent change with respect to the reference  %	Diffusion Coefficients, $D_{56}$  $m^2/min$	Percent change with respect to the reference  %	Percent change with respect to the age  %
100/0	1	9.0E-10	0	8.0E-10	0	-11
85/15	1	1.0E-09	11	1.0E-09	25	0
70/30	1	1.0E-09	11	1.0E-09	25	0
55/45	1	2.0E-09	122	1.8E-09	163	-13
100/0	2	5.0E-10	0	4.0E-10	0	-20
85/15	2	4.0E-10	-20	3.0E-10	-25	-25
70/30	2	3.0E-10	-40	2.0E-10	-50	-33
55/45	2	2.0E-10	-60	2.0E-10	-50	0
100/0	3	3.0E-10	0	3.0E-10	0	0
85/15	3	3.0E-10	0	2.0E-10	-33	-33
70/30	3	3.0E-10	0	1.0E-10	-67	-67
55/45	3	2.0E-10	-33	1.0E-10	-67	-50

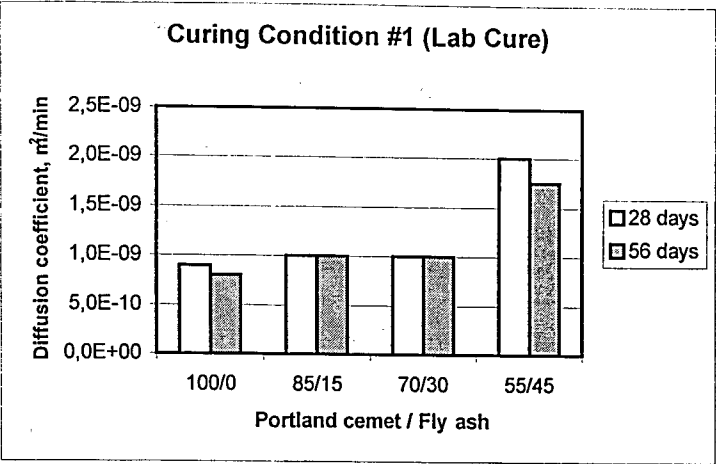


FIGURE 4.4. - 28 and 56 days diffusion coefficients of concrete specimens cured under Curing Condition 1 and having various percentages of fly ash and P.cement

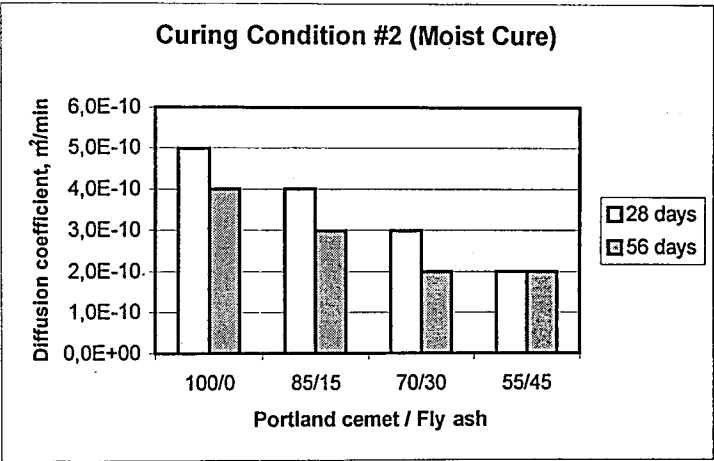


FIGURE 4.5. - 28 and 56 days diffusion coefficients of concrete specimens cured under Curing Condition 2 and having various percentages of fly ash and P. cement

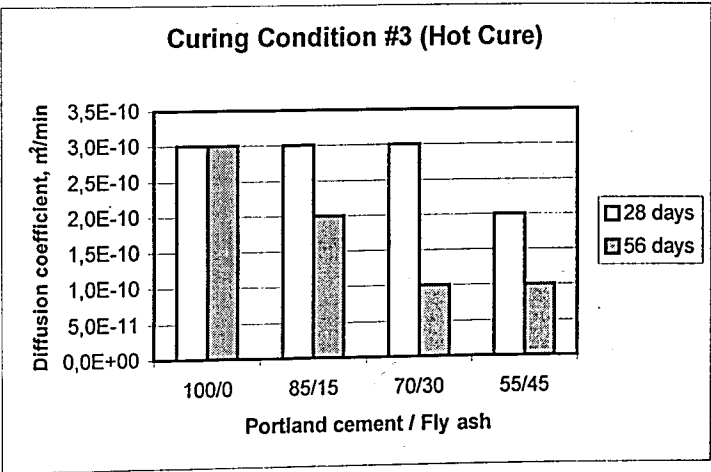


FIGURE 4.6. - 28 and 56 days diffusion coefficients of concrete specimens cured under Curing Condition 3 and having various percentages of fly ash and P. cement

#### 4.4. Chloride Ion Permeability

The results of the chloride ion permeability tests are presented in TABLE 4.6. and 4.7. and FIGURES 4.7. - 4.9. The results of these tests are evaluated according to the calculated total charge passed which is accepted as an indication of the chloride permeability of the concrete specimen. The total charge passed range from 2624 to 7431 coulombs for specimens tested at the age of 28 days. Whereas, the 56 days results ranged from 2430 to 6575 coulombs. The data obtained in these tests clearly show that the use of fly ash as replacement for portland cement has positive effects on concrete when cured under Curing Condition 2 and Curing Condition 3, the latter having better results. On the contrary, the increase in fly ash percentage negatively affected the chloride ion permeability for specimens cured under Curing Condition 1. The results further show that the permeability is reduced with age comparing the 28 and 56 days results.

As presented in TABLE 4.6 the results show that for Curing Condition 1 and 2, the chloride permeability values are classified as 'High' regardless of the fly ash content. Whereas, the specimens cured under hot curing, Curing Condition 3, exhibited 'Moderate' chloride permeability.

Evaluating the results according to different curing conditions the following conclusions are reached. The chloride ion permeability values indicated as total charge passed ranged from 7431 to 7795 coulombs as fly ash content increases from 15 % to 45 % at 28 days and from 6575 to 7781 coulombs at 56 days for specimens cured under Curing Condition 1. Likewise, the specimens subjected to Curing Condition 2 exhibited results ranging from 5443 to 4460 coulombs at 28 days and from 4580 to 3656 coulombs at 56 days. The 28-day and 56-days chloride ion permeability values of the specimens cured under Curing Condition 3 ranged from 4198 to 4460 coulombs and from 4080 to 2430 coulombs respectively.

The overall results of the rapid chloride permeability test are presented in the plot in FIGURE A1. as total charge passed in coulombs. This plot can be used as a summary for the entire rapid chloride permeability tests conducted in this study.

TABLE 4.6. - Chloride permeability of concrete specimens, determined by the total charge passed, at the ages of 28 and 56 days

PC/FA	Type of Curing	Age days	Total Charge Passed Coulombs	Chloride Perm. Rating	Age days	Total Charge Passed Coulombs	Chloride Perm. Rating
100/0	1	28	7431	High	56	6575	High
85/15	1	28	7510	High	56	6700	High
70/30	1	28	7736	High	56	7574	High
55/45	1	28	7795	High	56	7781	High
100/0	2	28	5443	High	56	4580	High
85/15	2	28	4686	High	56	4262	High
70/30	2	28	4604	High	56	4258	High
55/45	2	28	4460	High	56	3656	Moderate
100/0	3	28	4198	High	56	4080	High
85/15	3	28	3875	Moderate	56	3578	Moderate
70/30	3	28	2807	Moderate	56	2606	Moderate
55/45	3	28	2624	Moderate	56	2430	Moderate

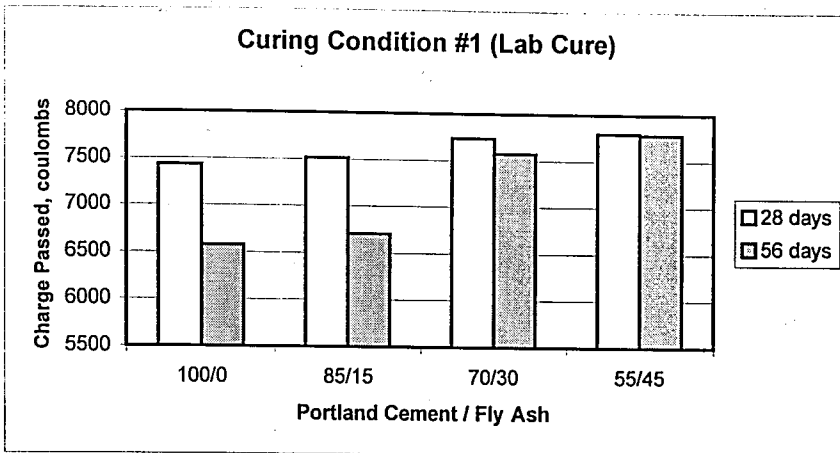


FIGURE 4.7. - 28 and 56 days chloride permeability of concrete specimens cured under Curing Condition 1 and having various percentages of fly ash and P. cement

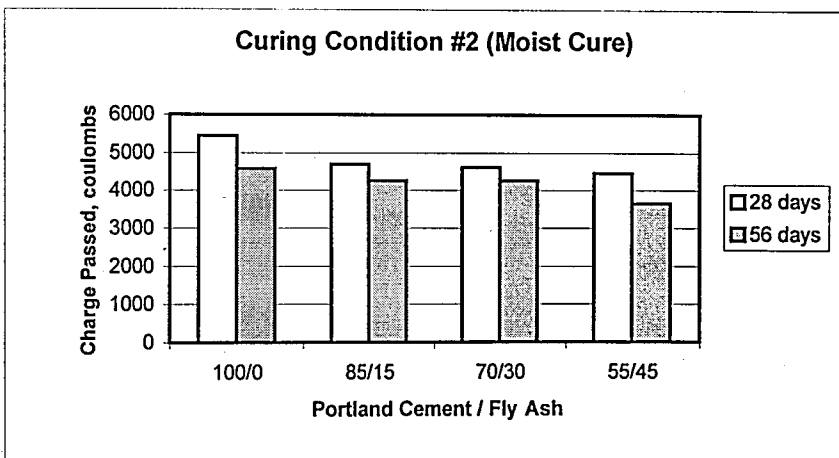


FIGURE 4.8. - 28 and 56 days chloride permeability of concrete specimens cured under Curing Condition 2 and having various percentages of fly ash and P. cement

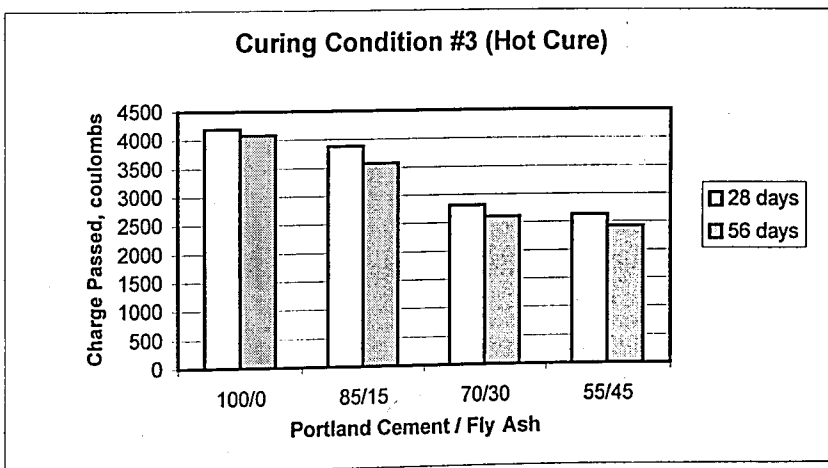


FIGURE 4.9. - 28 and 56 days chloride permeability of concrete specimens cured under Curing Condition 3 and having various percentages of fly ash and P. cement



TABLE 4.7. – Percent change of the chloride ion permeability, determined by the total charge passed, with respect to the reference and age

PC/FA	Type of Curing	Age	Change with respect to reference	Age	Change with respect to reference	Change with respect to age
		days	%	days	%	%
100/0	1	28	0	56	0	-12
85/15	1	28	1	56	2	-10
70/30	1	28	4	56	15	2
55/45	1	28	5	56	18	5
100/0	2	28	0	56	0	-16
85/15	2	28	-14	56	-7	-22
70/30	2	28	-15	56	-7	-22
55/45	2	28	-18	56	-20	-33
100/0	3	28	0	56	0	-3
85/15	3	28	-8	56	-12	-15
70/30	3	28	-33	56	-36	-38
55/45	3	28	-38	56	-40	-42

#### 4.5. Correlation between Initial Current and Total Charge Passed

During the rapid chloride permeability test, conducted to determine the chloride ion permeability of the concrete specimens, a basic observation is very evident; a specimen having a higher initial current reading at the start of the experiment usually has a higher total charge passed value at the end. The above correlation is high for all specimens ranging from 67.34 to 99.57 as presented in TABLE 4.9. This table also presents the results of the regression analysis of the data. The initial current data are presented in TABLE 4.8 and the plots of initial current versus charge passed are presented in FIGURES 4.10. and 4.11. These figures reveal that the data nearly fits straight lines showing high correlation.

Evaluating the initial current results according to different curing conditions the following conclusions are reached. The initial current values ranged from 216 to 228 miliamperes as fly ash content increases from 15 % to 45 % at 28 days and from 187 to 202 miliamperes at 56 days for specimens cured under Curing Condition 1. Likewise, the specimens subjected to Curing Condition 2 exhibited results ranging from 181 to 103 miliamperes at 28 days and from 145 to 94 miliamperes at 56 days. The 28-day and 56-days initial current values of the specimens cured under Curing Condition 3 ranged from 128 to 70 coulombs and from 134 to 63 coulombs respectively.

All these above data are in agreement with the total charge passed data and may be used for the prediction of the chloride permeability of concretes from the initial current values providing the advantage of not extending the test to full 6-hour duration. However, the effect of wider range of  $w/b$  and age as well as type and grading of aggregates, curing type, and different chemical admixtures should be investigated.

TABLE 4.8. – Initial current measurements at 28 and 56 days

PC / FA	Curing Condition	$I_{0,28}$	$I_{0,56}$
		Miliamperes	Miliamperes
100/0	1	216	187
85/15	1	218	194
70/30	1	223	197
55/45	1	228	202
100/0	2	181	145
85/15	2	154	129
70/30	2	134	100
55/45	2	103	94
100/0	3	128	134
85/15	3	122	114
70/30	3	87	76
55/45	3	70	63

TABLE 4.9. – Slope and correlation coefficients of the relation between initial current and total charge passed at 28 and 56 days

Curing Condition	Age	Slope	Correlation Coefficient
	Days	Coulomb/mA	$R^2$
1	28	33.1	0.9416
2	28	15.1	0.7850
3	28	28.3	0.9700
1	56	43.1	0.8151
2	56	19.6	0.6734
3	56	23.7	0.9957

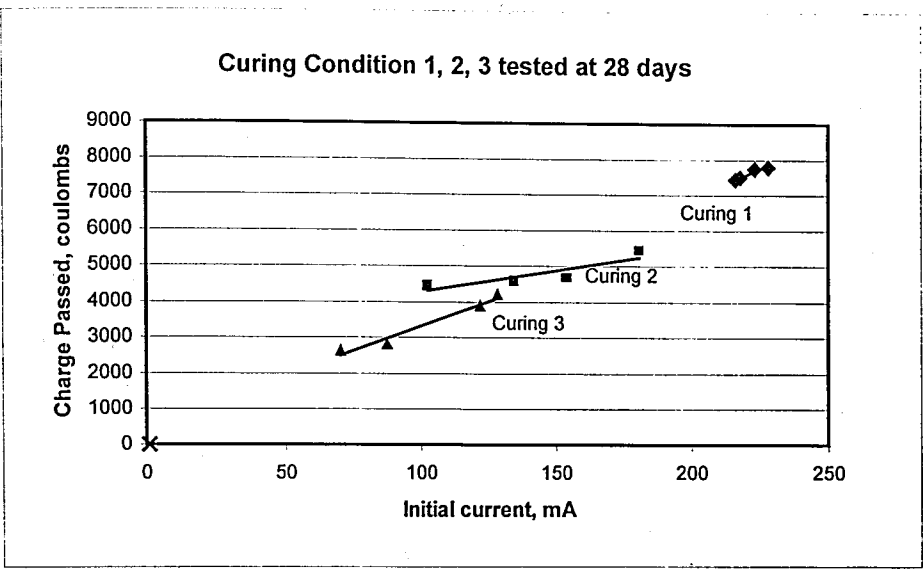


FIGURE 4.10. - Initial current versus charge passed in RCPT at 28 days for concrete having various percentages of fly ash and cured under different curing conditions.

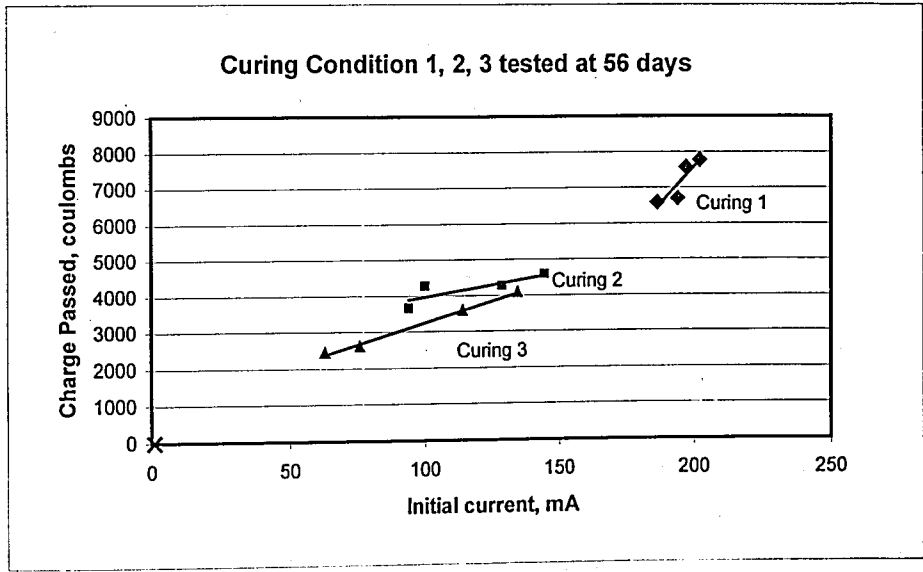


FIGURE 4.11. - Initial current versus charge passed in RCPT at 56 days for concrete having various percentages of fly ash and cured under different curing conditions.

#### 4.6. Resistance to Deicing Salt Scaling

The results for the resistance to deicing salt scaling tests are evaluated as number of cycles to reach 5<sup>th</sup> rating of deterioration described by ASTM 672 as 'Severe scaling; coarse aggregate visible over entire surface'. This classification is based mainly on visual inspection and the results of these inspections are presented in TABLE 4.10 and 4.11. The visual inspections made at the end of each 5<sup>th</sup> cycle were supplemented with additional photographs and notes. The notes taken at the end of each 5<sup>th</sup> cycle are used to construct TABLE 4.11. which presents the degree of deterioration in each stage of the experiment for all specimens. The photographs are presented in Appendix. The results show that in uncontrolled curing, Curing Condition 1, the durability of all the specimens are lower than Curing Conditions 3 and Curing Condition 2. It is also found that the incorporation of fly ash does not make difference between specimens having different percentages of fly ash in Curing Condition 1.

The specimens cured under Curing Condition 3 performed better than the Curing Condition 2 specimens. For both of these curing types the increase in fly ash content resulted in an increase in durability just on the contrary to Curing Condition 1. It took nearly 25 cycles for all Curing Condition 1 specimens to reach the final stage of deterioration, whereas the Curing Condition 2 specimens exhibited cycles ranging from 25 to 35 as fly ash content increases. The best performing Curing Condition 3 specimens on the other hand, showed a range of 25 to 40 cycles with increasing fly ash percentage. The durability performances for Curing Condition 2 and 3 specimens are positively affected by age. This is evident when the 28 days and 56 days results are compared.

In addition to the visual rating evaluation, the scaled off particles were collected and weighed at the end of each 5<sup>th</sup> cycle and their total at the end of each test were summed to obtain a value called total scaling residue. The values for total scaling residue ranged from 5.52 to 6.95 kg / m<sup>2</sup> at 28 days and ranged from 5.23 to 7.80 kg / m<sup>2</sup> at 56 days. These total scaling residue values don't exhibit a great diversity since the numbers of cycles to reach 5<sup>th</sup> rating of deterioration are not very much different for all types of specimens.

TABLE 4.10. – Results of the deicing salt scaling tests on concrete specimens tested at the ages of 28 and 56 days

PC/FA	Type of Curing	Age days	Number of cycles to reach 5 <sup>th</sup> rating of deterioration	Total Scaling Residue kg / m <sup>2</sup>	Age Days	Number of cycles to reach 5 <sup>th</sup> rating of deterioration	Total Scaling Residue kg / m <sup>2</sup>
100/0	1	28	25	5.52	56	25	5.23
85/15	1	28	25	5.53	56	25	5.47
70/30	1	28	20	5.79	56	25	6.46
55/45	1	28	25	6.17	56	25	5.80
100/0	2	28	25	5.86	56	30	6.21
85/15	2	28	25	6.57	56	30	7.65
70/30	2	28	30	6.36	56	35	7.80
55/45	2	28	35	5.87	56	35	7.66
100/0	3	28	25	6.95	56	30	5.89
85/15	3	28	35	6.47	56	35	6.50
70/30	3	28	40	6.45	56	40	6.52
55/45	3	28	40	6.12	56	40	6.56

TABLE 4.11. – 28 and 56 days results of the deicing salt scaling tests on concrete specimens

	Visual rating results after every 5 cycles until 5 <sup>th</sup> rating of deterioration is reached								
TYPE	5	10	15	20	25	30	35	40	45
fa00128	0-1	2	3	4-5	5				
fa15128	0-1	2-3	3-4	4-5	5				
fa30128	1	2-3	4	5					
fa45128	1	2	3-4	4-5	5				
fa00228	1-2	2-3	3-4	4-5	5				
fa15228	1	3	3-4	4-5	5				
fa30228	0-1	2	3-4	4	4-5	5			
fa45228	1-2	2-3	3	3-4	4	4-5	5		
fa00328	1-2	2-3	4-5	4-5	5				
fa15328	1	2	2-3	4	4	4-5	5		
fa30328	1	2	3	3-4	4	4	4-5	5	
fa45328	0-1	1-2	3	3-4	3-4	4	4-5	5	
fa00156	0-1	2-3	3	4	5				
fa15156	1	2	3	4-5	5				
fa30156	1	2	3-4	4-5	5				
fa45156	1	2	3-4	4-5	5				
fa00256	1	2	3-4	4	4-5	5			
fa15256	1	1-2	3-4	4	4-5	5			
fa30256	1	1-2	2-3	3	3-4	4	5		
fa45256	0	1-2	2-3	3-4	4	4-5	5		
fa00356	1	3	3-4	4	4-5	5			
fa15356	0-1	2-3	3	3-4	4	4-5	5		
fa30356	0-1	1-2	2	2-3	3	4	4-5	5	
fa45356	0	1	1-2	2	3	4	4-5	5	

## 4.7. Resistance to Freezing and Thawing Cycles

It was mentioned before that, in this study the freezing and thawing tests were done together with the deicing salt scaling tests on the same specimens. It was assumed that the effect of deicing salt during the freezing and thawing cycles of the ASTM 672 were limited only to the top surface of the specimens and the other effect of freezing and thawing on the concrete specimens could be assessed separately on the same specimens.

In this scope, the effects of freezing and thawing cycles on the specimens are evaluated as the changes in flexural and equivalent cube strengths of 100\*100\*500 mm beams with respected to the reference specimens of the same mix proportions not subjected to freezing and thawing cycles. It should be mentioned that the flexural and equivalent cube strengths of these reference concretes are evaluated separately among each other as a measure of hardened concrete mechanical properties earlier in sections 4.2.1.2. and 4.2.1.3.

### 4.7.1. Flexural Strength of Beams Subjected to freezing and thawing Cycles

Flexural strength determinations were performed on two sets of specimens tested at 28 and 56 days. First set of results belonged to the reference concretes, which were not exposed to freezing and thawing and deicing salt scaling tests. These reference specimens were tested when their counterpart, exposed to freezing and thawing and deicing salt scaling tests, has reached a predetermined deterioration rating given by ASTM C 672. The second set of results is the results of the freezing and thawing specimens after the termination of their tests. The results are presented on TABLES 4.12. and 4.13. FIGURES 4.12 - 4.14 show the 28 days whereas FIGURES 4.15. - 4.17. show the 56 days results.

As presented in TABLE 4.12, the flexural strength results for specimens exposed to freezing and thawing showed lower flexural strength values than the control as expected. The range of results for specimens tested after 28 days and cured under Curing Condition 1 was between 4.75 and 3.94 MPa. The Curing Condition 2 specimens were between 5.07 and 4.70 MPa whereas, the flexural strength results for Curing Condition 3 specimens ranged from 5.76 to 4.52 MPa. On the other hand, the control specimens not exposed to freezing and thawing cycles (after 28 days) and cured under Curing Condition 1, range



from 6.01 to 4.99 MPa. The results for the same set cured under Curing Condition 2 ranged from 6.64 to 5.82 MPa, and Curing Condition 3 specimens exhibited results ranging from 7.45 to 5.07 MPa.

These results obtained for 28 days specimens have a common trend; the addition of fly ash has negative effect on the flexural strength of concrete. The lowest results were reached for 45 percent cement replacement by fly ash in every set of specimens. This was an expected effect also observed by other researchers [16]. The negative effect of fly ash is observed to be less for specimens cured under Curing Condition 2 compared to the Curing Condition 1 and 3 specimens. The percent change in flexural strength of freezing and thawing specimens with respect to the control specimens exhibited similar values (~20 percent decrease) for all curing cases.

Similarly, TABLE 4.13 presents the flexural strength results for control specimens, not exposed to freezing and thawing cycles and test specimens tested after 56 days. The test specimens exposed to freezing and thawing again showed relatively lower flexural strength values than control. The range of results for specimens tested after 56 days and cured under Curing Condition 1 was between 5.01 and 4.04 MPa. The Curing Condition 2 specimens were between 5.33 and 4.88 MPa whereas, the flexural strength results for Curing Condition 3 specimens ranged from 6.16 to 4.94 MPa. When we look at the control specimens the general results are; the control specimens cured under Curing Condition 1 showed results ranging from 5.89 to 4.23 MPa. The results for the same set cured under Curing Condition 2 ranged from 7.01 to 5.92 MPa, and Curing Condition 3 specimens exhibited results ranging from 7.54 to 6.03 MPa.

TABLE 4.12. – Flexural strength of beams tested after freeze-thaw cycles started at 28 days.

PC / FA	Curing Condition	Control $f_c$ MPa	Percent change with respect to the reference %	Number of F-T cycles before testing	F-T cycles started at 28 days $f_c$ MPa	Percent change with respect to the reference %	Percent change with respect to the control %
100/0	1	6.01	0	25	4.75	0	-21
85/15	1	5.51	-8	25	4.68	-1	-15
70/30	1	5.23	-13	20	4.10	-14	-22
55/45	1	4.99	-17	25	3.94	-17	-21
100/0	2	6.64	0	25	5.07	0	-24
85/15	2	6.68	1	25	5.04	-1	-24
70/30	2	6.45	-3	30	4.87	-4	-25
55/45	2	5.82	-12	35	4.70	-7	-19
100/0	3	7.45	0	25	5.76	0	-23
85/15	3	5.69	-24	35	5.01	-13	-12
70/30	3	5.56	-25	40	5.11	-11	-8
55/45	3	5.07	-32	40	4.52	-22	-11

The same trend obtained for 28 days specimens can also be observed for the 56 days specimens; the addition of fly ash has negative effect on the flexural strength of concrete. The lowest results were reached for 45 percent cement replacement by fly ash in every set of specimens. The negative effect of fly ash is again observed to be less for specimens cured under Curing Condition 2 compared to the Curing Condition 1 and 3 specimens. The percent change in flexural strength of freezing and thawing specimens with

respect to the control specimens exhibited similar values (~20 percent decrease) for all curing cases.

TABLE 4.13. – Flexural strength of beams tested after freeze-thaw cycles started at 56 days.

PC / FA	Curing Condition	Control  $f_c$  MPa	Percent change with respect to the reference  %	Number of F-T cycles before testing	F-T cycles started at 56 days  $f_c$  MPa	Percent change with respect to the reference  %	Percent change with respect to the control  %
100/0	1	5.89	0	25	5.01	0	-15
85/15	1	5.02	-14	25	4.91	-2	-2
70/30	1	5.55	-6	25	4.35	-14	-22
55/45	1	4.23	-28	25	4.04	-20	-4
100/0	2	7.01	0	30	5.33	0	-24
85/15	2	6.74	-4	30	5.31	0	-21
70/30	2	6.51	-8	35	5.22	-2	-20
55/45	2	5.92	-18	35	4.88	-9	-18
100/0	3	7.54	0	30	6.16	0	-18
85/15	3	6.92	-10	35	5.99	-4	-13
70/30	3	7.35	-3	40	5.49	-14	-25
55/45	3	6.03	-25	40	4.94	-26	-18

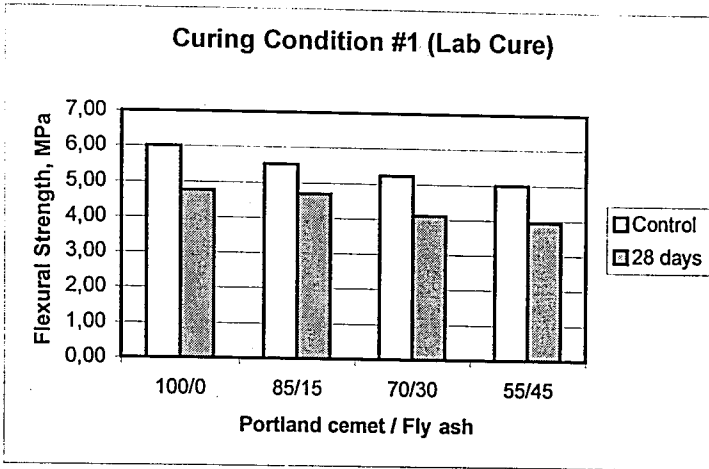


FIGURE 4.12.- 28 days flexural strengths of F-T and Control specimens cured under Curing Condition 1 and having various percentages of fly ash and portland cement

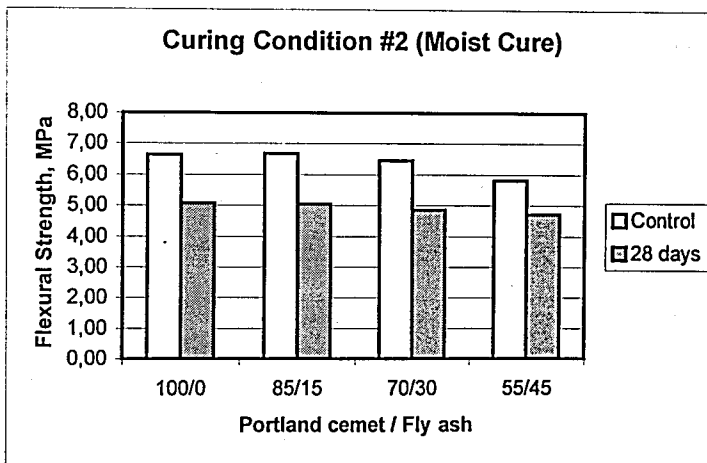


FIGURE 4.13.- 28 days flexural strengths of F-T and Control specimens cured under Curing Condition 2 and having various percentages of fly ash and portland cement

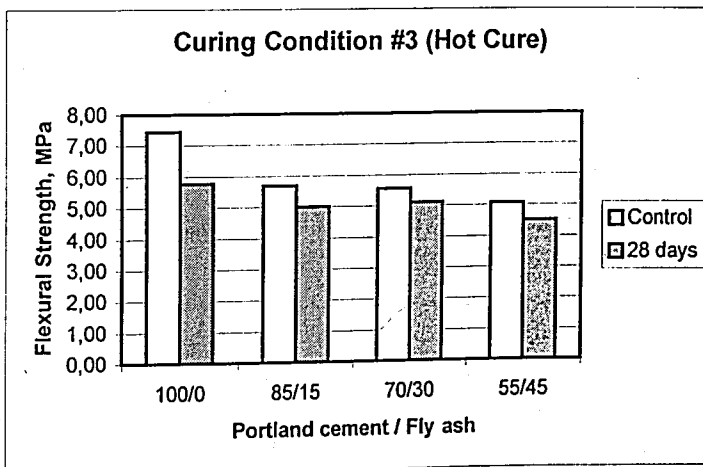


FIGURE 4.14.- 28 days flexural strengths of F-T and Control specimens cured under Curing Condition 3 and having various percentages of fly ash and portland cement

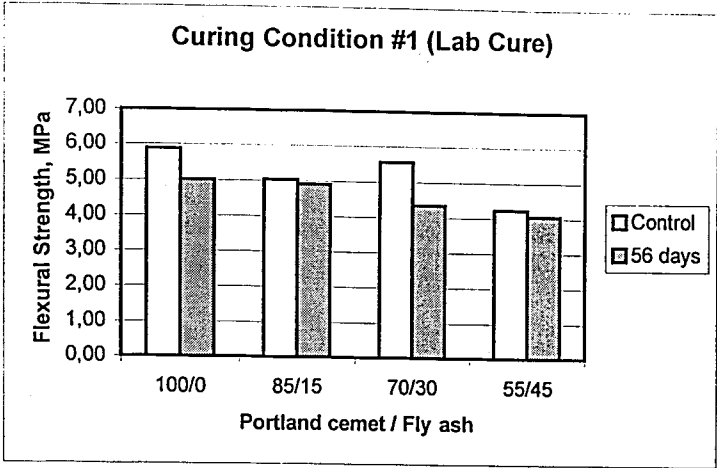


FIGURE 4.15. - 56 days flexural strengths of F-T and Control specimens cured under Curing Condition 1 and having various percentages of fly ash and portland cement

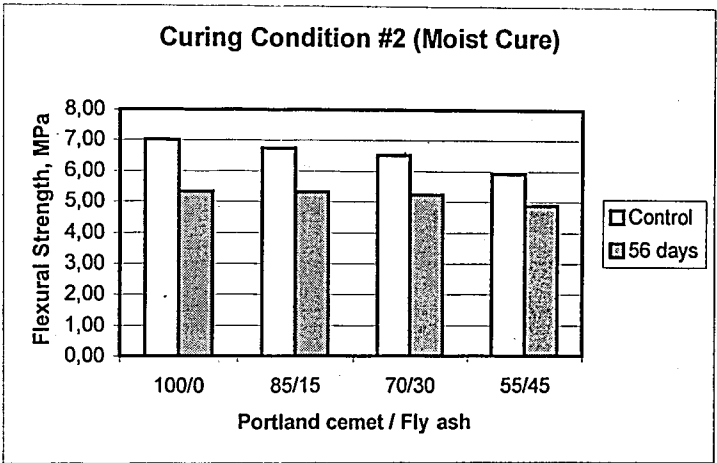


FIGURE 4.16.- 56 days flexural strengths of F-T and Control specimens cured under Curing Condition 2 and having various percentages of fly ash and portland cement

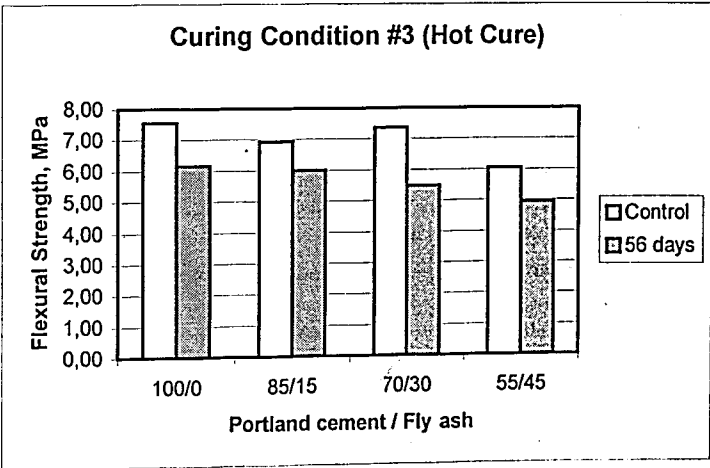


FIGURE 4.17.- 56 days flexural strengths of F-T and Control specimens cured under Curing Condition 3 and having various percentages of fly ash and portland cement

#### 4.7.2. Equivalent Compressive Strength of Specimens after Freezing and Thawing

The equivalent cube strength tests were performed right after the flexural strength tests since the specimens used for equivalent cube strength determination were the portions of beams broken in flexure. As for the equivalent cube strength determination, the same two sets of results are evaluated; first the control specimens not subjected to freezing and thawing cycles, second the freezing and thawing specimens tested at the end of 28 and 56 days. The results are as given in TABLE 4.13 and 4.14.

TABLE 4.14. – Equivalent cube strengths of beam sections broken after freezing and thawing cycles started at 28 days.

PC / FA	Curing Condition	Control  $f_c$  MPa	Percent change with respect to the reference  %	Number of F-T cycles before testing	F-T cycles started at 28 days  $f_c$  MPa	Percent change with respect to the reference  %	Percent change with respect to the control  %
100/0	1	50.4	0	25	46.2	0	-8
85/15	1	50.2	0	25	40.8	-12	-19
70/30	1	48.8	-3	20	38.1	-18	-22
55/45	1	34.2	-32	25	27.9	-40	-19
100/0	2	58.8	0	25	57.6	0	-2
85/15	2	57.0	-3	25	52.5	-9	-8
70/30	2	53.7	-9	30	51.8	-10	-4
55/45	2	47.1	-20	35	45.8	-20	-3
100/0	3	62.6	0	25	61.5	0	-2
85/15	3	59.6	-5	35	59.0	-4	-1
70/30	3	54.5	-13	40	52.8	-14	-3
55/45	3	50.2	-20	40	48.4	-21	-4

TABLE 4.15. – Equivalent cube strengths of beam sections broken after freeze-thaw cycles started at 56 days.

PC / FA	Curing Condition	Control  $f_c$  MPa	Percent change with respect to the reference  %	Number of F-T cycles before testing	F-T cycles started at 56 days  $f_c$  MPa	Percent change with respect to the reference  %	Percent change with respect to the control  %
100/0	1	54.24	0	25	47.02	0	-13
85/15	1	52.53	-3	25	44.61	-5	-15
70/30	1	44.3	-20	25	41.03	-12	-7
55/45	1	38.65	-31	25	34.85	-24	-10
100/0	2	59.05	0	30	57.83	0	-2
85/15	2	57.23	-4	30	53.77	-8	-6
70/30	2	55.88	-6	35	49.49	-17	-11
55/45	2	48.06	-22	35	47.35	-21	-1
100/0	3	63.6	0	30	63.51	0	0
85/15	3	60.17	-7	35	60.54	-6	1
70/30	3	54.85	-17	40	53.83	-19	-2
55/45	3	52.02	-23	40	52.27	-22	0

It is clear that the above results show a similar trend as the flexural strength results; there is a significant decrease of strength with the addition of fly ash instead of cement. As expected this effect is more distinguished for specimens cured under uncontrolled conditions. There is also an overall improvement with age between 28 and 56 days. The general observations are;

The equivalent cube strength values ranged from 46.20 to 27.90 MPa at 28 days and from 47.02 to 34.35 MPa at 56 days for specimens cured under Curing Condition 1.

These corresponded to a strength loss of up to 40 percent compared to same mix control specimens not exposed to freezing and thawing cycles. Likewise, the specimens subjected to Curing Condition 2 exhibited results ranging from 57.60 to 45.80 MPa at 28 days and from 57.83 to 47.35 MPa at 56 days. The 28-day and 56-days equivalent cube strength values of the specimens cured under Curing Condition 3 ranged from 61.50 to 48.40 MPa and from 63.51 to 52.27 MPa respectively. For Curing Condition 2 and 3 specimens the strength loss, relative to control specimens, is limited to nearly 20 percent, greater for specimens having higher percentages of fly ash.

Considering the overall results, the equivalent cube strength values of those specimens having only portland cement are found to be higher than those incorporating fly ash for any curing condition. It is also found that the curing conditions effected the equivalent cube strength and best results are observed for Curing Condition 3, then Curing Condition 2 and Curing Condition 1. Moreover, it is evident that the increase in the percentage of cement replacement by fly ash made significant decreases in equivalent cube strength. Generally, the equivalent cube strength showed an increase with age between 28 and 56 days under any given curing condition. This increase is somewhat more pronounced for Curing Condition 2 and less significant for Curing Condition 3 and 1. These results are also shown in graphical form in FIGURES 4.18 - 4.23.



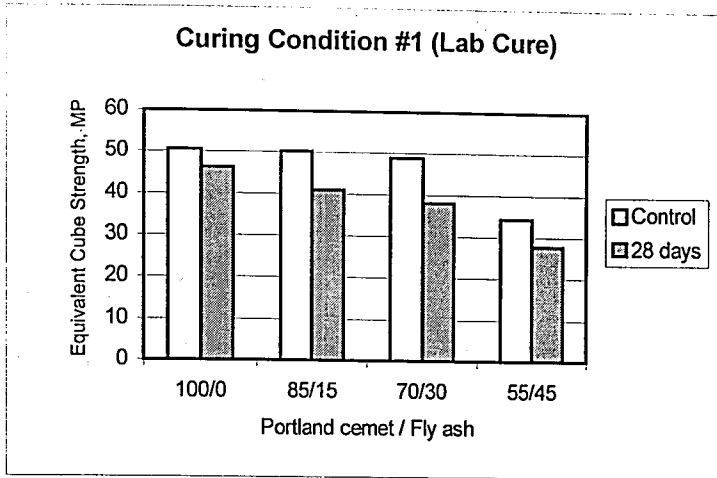


FIGURE 4.18.- 28 days equivalent cube strengths of F-T and Control specimens cured under Curing Condition 1 and having various percentages of fly ash and P.cement

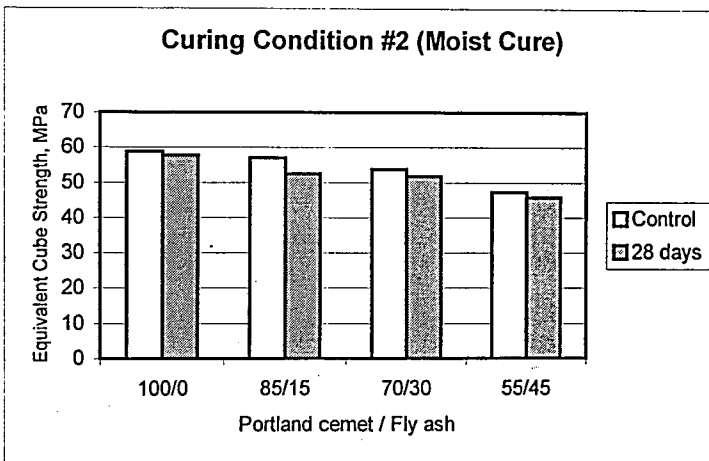


FIGURE 4.19.- 28 days equivalent cube strengths of F-T and Control specimens cured under Curing Condition 2 and having various percentages of fly ash and P.cement

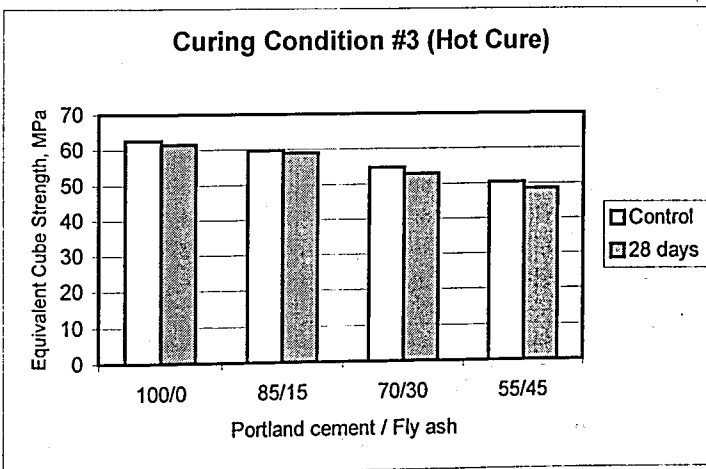


FIGURE 4.20.- 28 days equivalent cube strengths of F-T and Control specimens cured under Curing Condition 3 and having various percentages of fly ash and P.cement

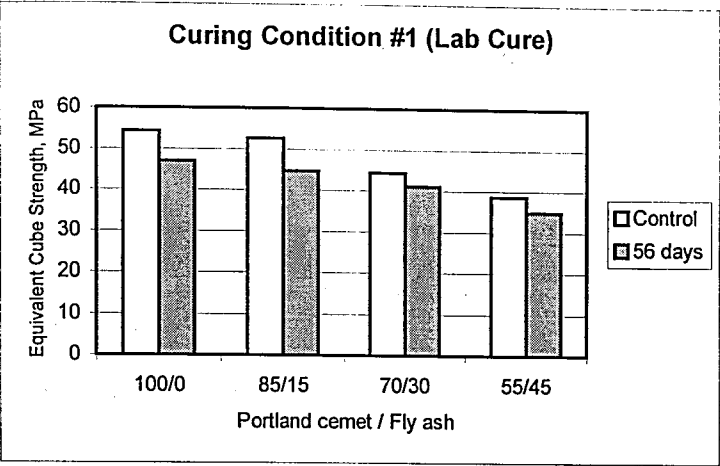


FIGURE 4.21.- 56 days equivalent cube strengths of F-T and Control specimens cured under Curing Condition 1 and having various percentages of fly ash and P. cement

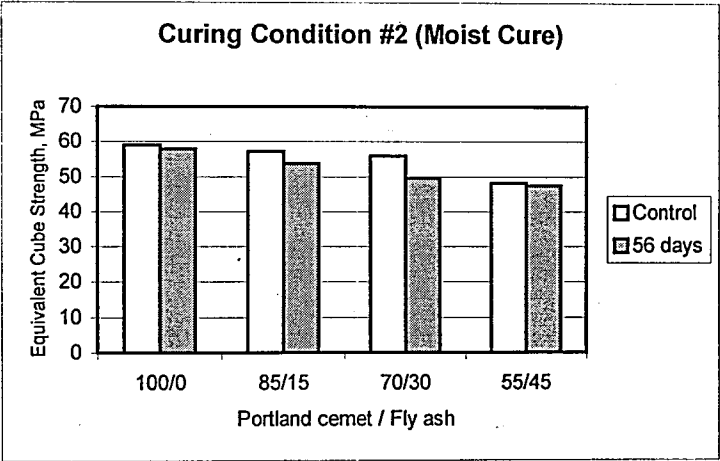


FIGURE 4.22.- 56 days equivalent cube strengths of F-T and Control specimens cured under Curing Condition 2 and having various percentages of fly ash and P. cement

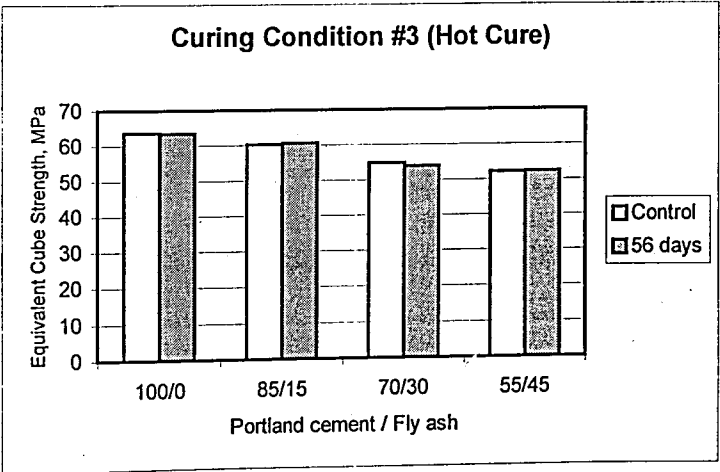


FIGURE 4.23.- 56 days equivalent cube strengths of F-T and Control specimens cured under Curing Condition 3 and having various percentages of fly ash and P. cement

## 5. CONCLUSION

1. The splitting tensile strength tests at 28 and 56 days showed that the replacement of cement by fly ash caused a decrease in splitting tensile strengths of the test specimens. This loss in strength increased as the percentage of fly ash increased up to 45%. The significance of this strength loss was greatly reduced by hot curing, Curing Condition 3, followed by standard curing (Curing Condition 2) and lowest results were observed for high volume fly ash concrete cured under uncontrolled curing, Curing Condition 1.

2. A similar trend, as the splitting strength, is observed for the flexural strength development of the control specimens. The addition of fly ash resulted in a general reduction of flexural strength, which is found to be less negatively effective in hot curing. The highest strength values for high volume fly ash concrete specimens are found to be Curing Condition 3 specimens. The Curing Condition 2 specimens performed worse than the Curing Condition 3 ones. Whereas, Curing Condition 1 specimens formed the lowest strength group.

3. The equivalent cube strength tests conducted on the reference specimens of the freezing and thawing tests revealed that the same criteria of strength loss in concrete incorporating fly ash is also valid for equivalent cube strength. Once again, this adverse effect of fly ash replacement for cement, is inhibited to a certain extent by hot curing followed by standard curing. Lowest results are obtained for high volume fly ash concrete specimens cured under uncontrolled conditions.

4. In the sorptivity studies, it was observed that under uncontrolled curing the capillary absorption of the specimens increases by an increase in the percentage of fly ash. On the other hand the Curing Condition 2 and 3 specimens showed a reasonable decrease in capillary absorption as the fly ash content in the mixture increased up to 45%.

5. The rapid chloride permeability tests revealed that fly ash replacement for cement has negative effects for concrete specimens cured under uncontrolled curing conditions. Under such conditions, the highest permeability results, classified as 'High', are obtained for 45% fly ash content at 28 and 56 days. On the contrary, under Curing

Condition 2 and especially Curing Condition 3 the concrete specimens exhibited much lower chloride permeability values that can even be classified as 'Moderate'. The lowest permeability was obtained by hot curing and 45% fly ash replacement for cement. The permeability values are observed to be reduced with age from 28 days to 56 days. Generally, the chloride permeability values of all the specimens are high which might be attributed to the relatively high w/c of 0.5.

6. The relation between initial current reading and the final total charge passed after 6 hours in the rapid chloride permeability test was found to be quite linear, indicating a high correlation. Although the number of tests conducted are limited, this correlation seems to be independent of fly ash content, curing condition, and age. In an effort to better investigate this correlation, it is evident that some further research and testing with more specimens are necessary.

7. The resistance to deicing salt scaling is found to be unaffected for specimens containing different percentages of fly ash and cured under uncontrolled condition. The situation changes for moist and hot curing. Under these curing conditions the concrete specimens are found to have attained better deicing salt resistance. The highest resistance was observed for the concrete specimens incorporating 45% fly ash cured under Curing Condition 3. Curing Condition 2 specimens had the second highest results. The durability performance of all of the specimens are found to be little affected by age from 28 days to 56 days.

8. In the case of durability performance against freezing and thawing, the determining criteria was chosen as the changes which occur in mechanical properties of the freezing and thawing specimens relative to the control specimens. The flexural strength data obtained from freezing and thawing specimens show a similar trend with the control data. Among the three curing types, Curing Condition 1 specimens exhibit the lowest flexural strength values and higher percentages of fly ash reduced strength in all curing cases. When the freezing and thawing specimens are compared with control specimens, the overall reduction in strength is approximately 20 percent being independent of fly ash content, curing condition, and age. The evaluation of the equivalent cube strength tests conducted on the same specimens reveals results not very different from the flexural

strength tests. The fly ash percentage has negative effects whereas hot curing has positive effects on strength. The standard cured (Curing Condition 2) specimens performed considerable better than Curing Condition 1 specimens. The highest strength values are again attained by Curing Condition 3 with no fly ash content.

9. As a general remark, it is worth noting that the scope of this study was limited considering the age of the specimens, being 28 and 56 days. These ages might be considered insufficient for entire effects of fly ash reactions in the concrete paste to be fully assessed. Further research including 90 days and 1 year or even higher ages are recommended to support the findings of this study.

**APPENDIX**

# Chloride Ion Permeability

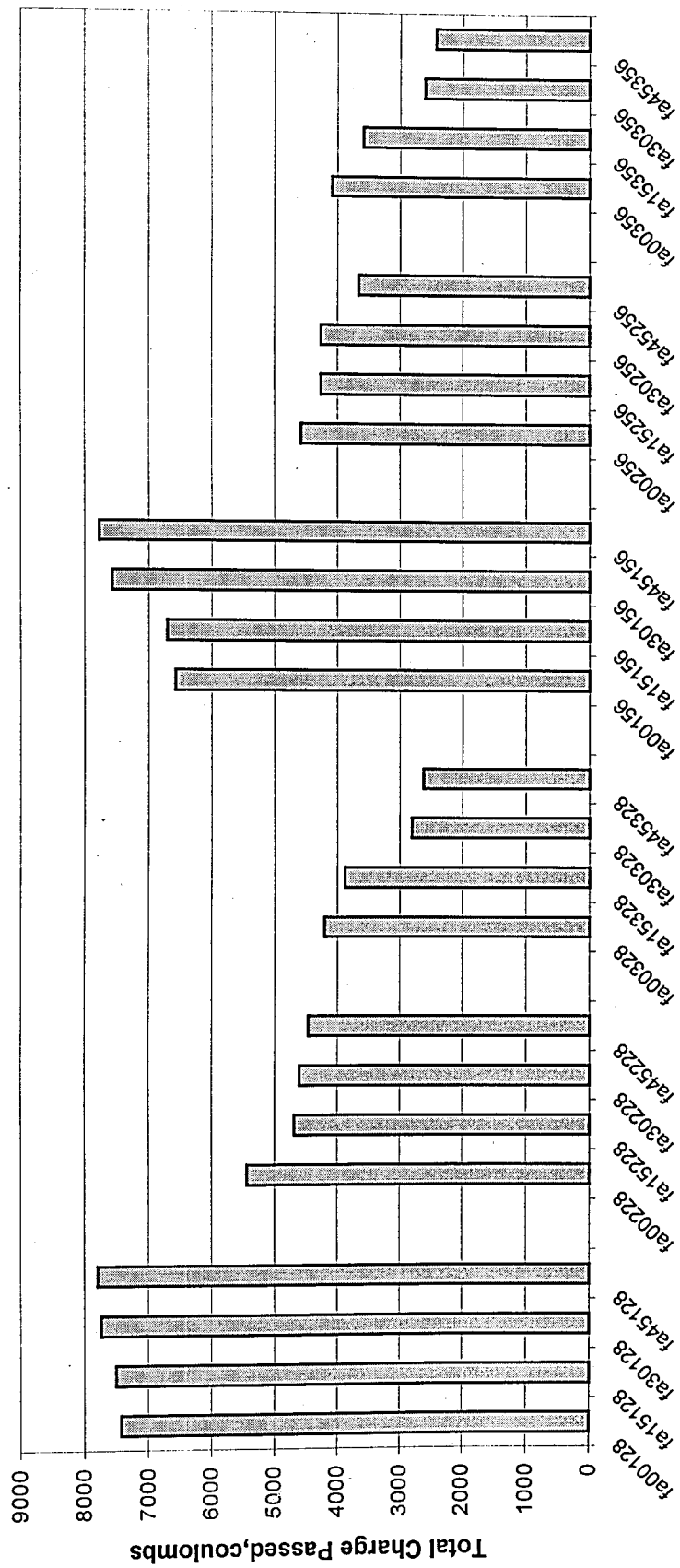


FIGURE A 1. - Overall test results of 28 and 56 days chloride permeability of concrete specimens cured under 3 different curing conditions and having various percentages of fly ash and portland cement tested at 56 days



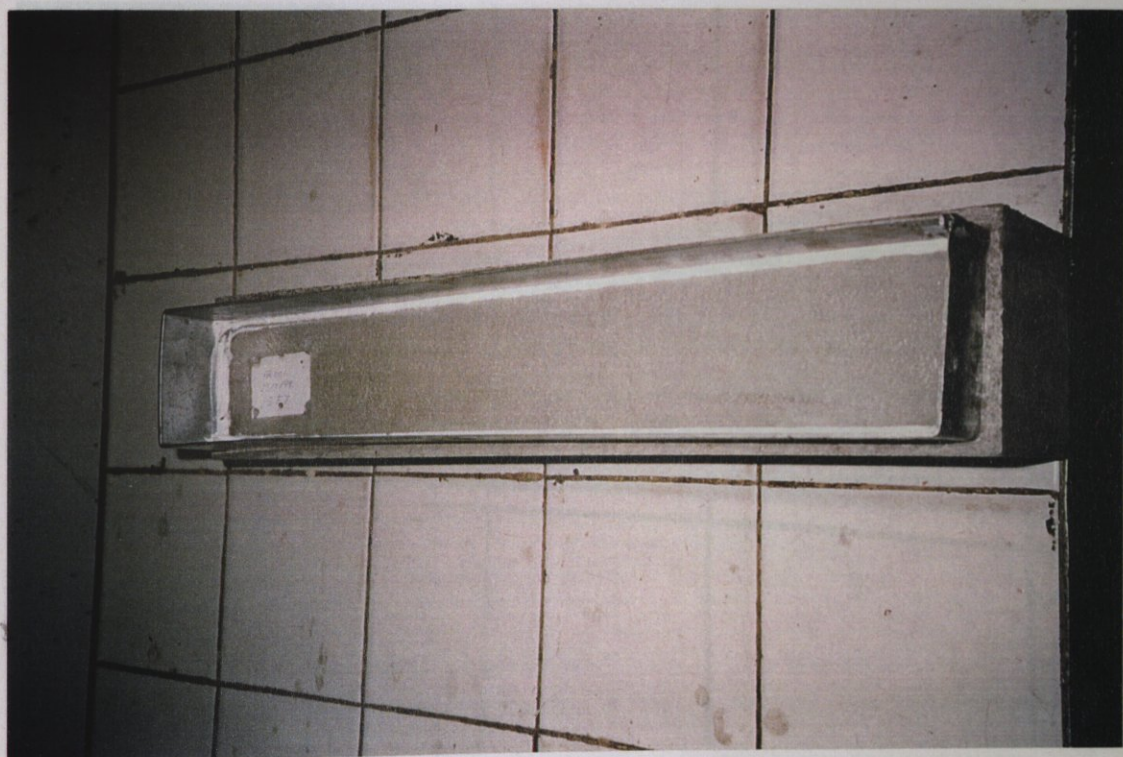


FIGURE A.2. - Test specimen at the beginning of the deicing salt scaling test.

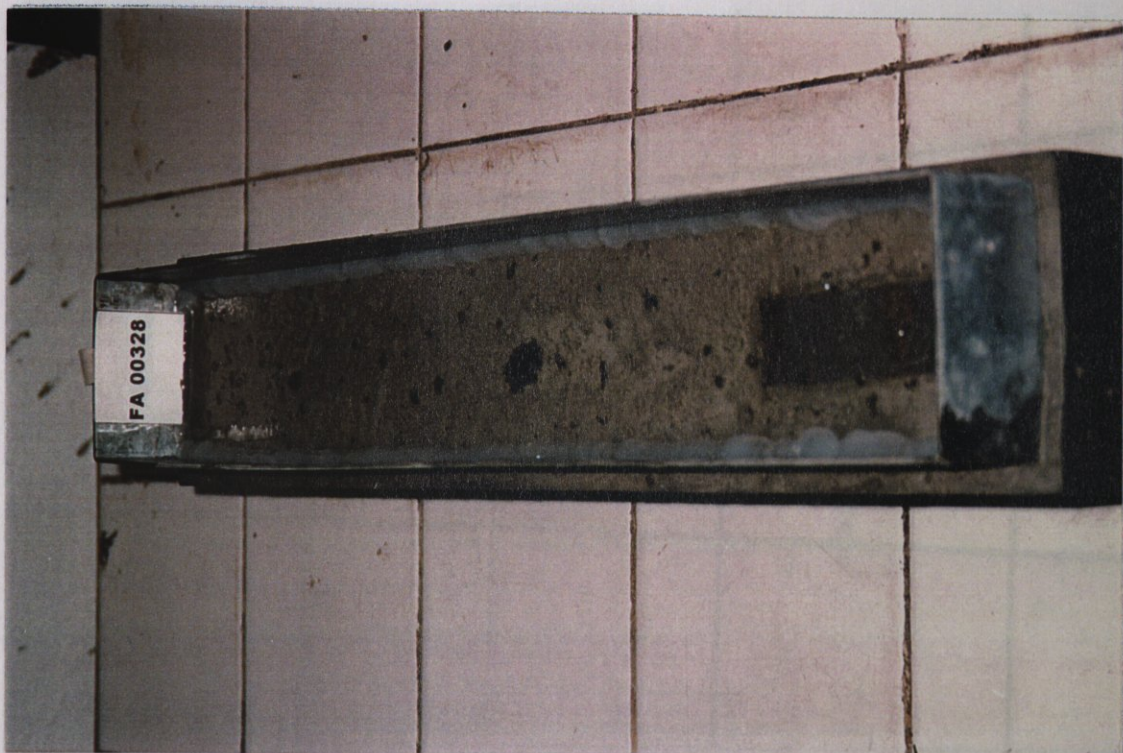


FIGURE A.3. - FA003 after the 5<sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days).





FIGURE A.4. - FA003 after the 15<sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days).



FIGURE A.5. - FA003 after the 20<sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days).



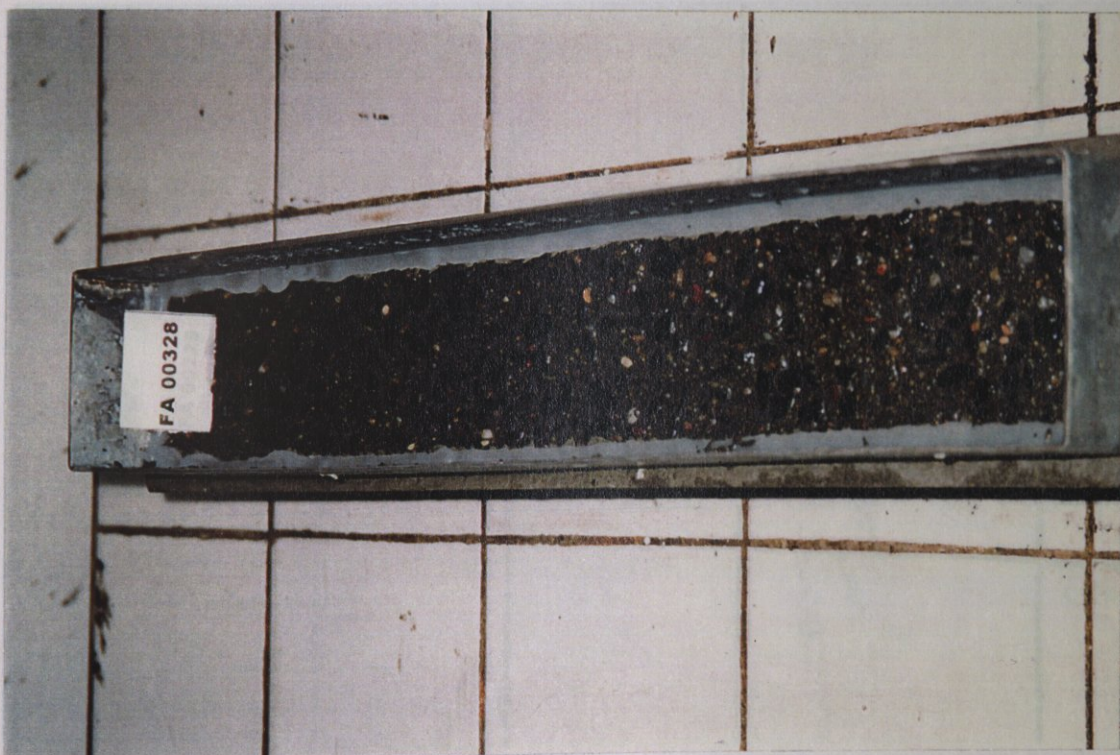


FIGURE A.6.- FA003 after the 25<sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)



FIGURE A.7.- FA153 after the 35<sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)





FIGURE A.8.- FA002 after the 25<sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)



FIGURE A.9.- FA152 after the 25<sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)





FIGURE A.10. - FA302 after the 30<sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)



FIGURE A.11. - FA452 after the 35<sup>th</sup> cycle of the deicing salt scaling test (tested at 28 days). (test terminated)



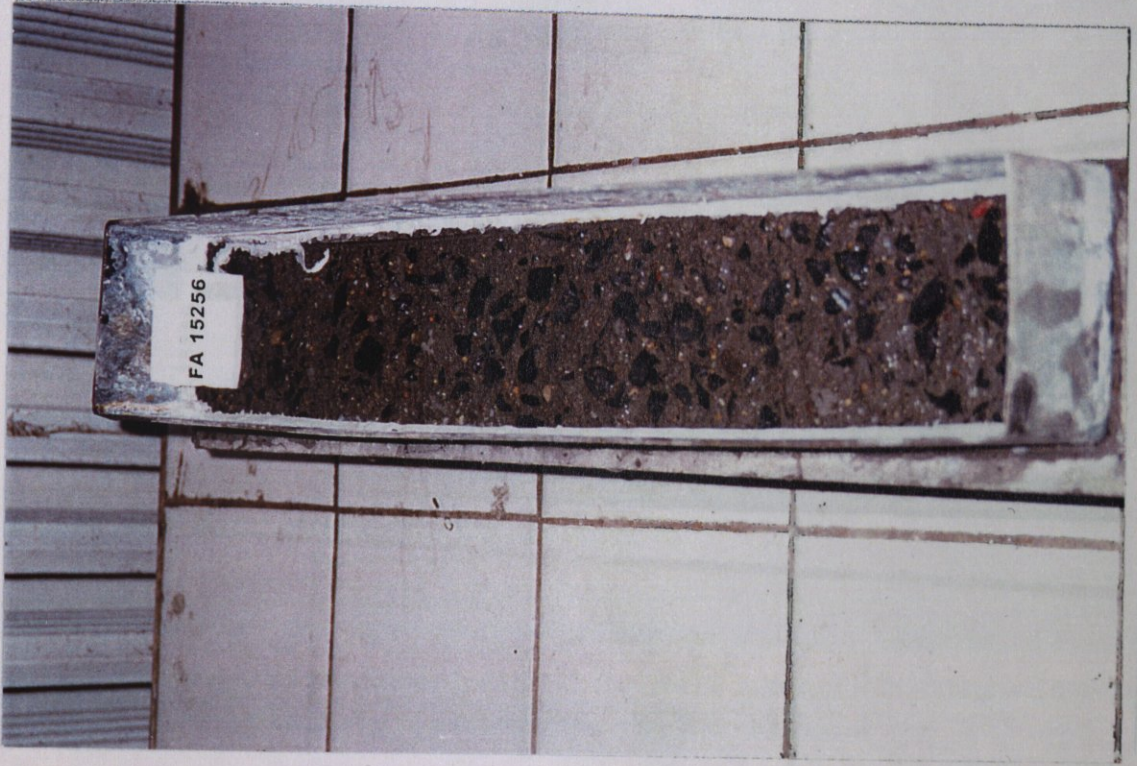


FIGURE A.13. - FA152 after the 30<sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days). (test terminated)



FIGURE A.12. - FA002 after the 30<sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days). (test terminated)



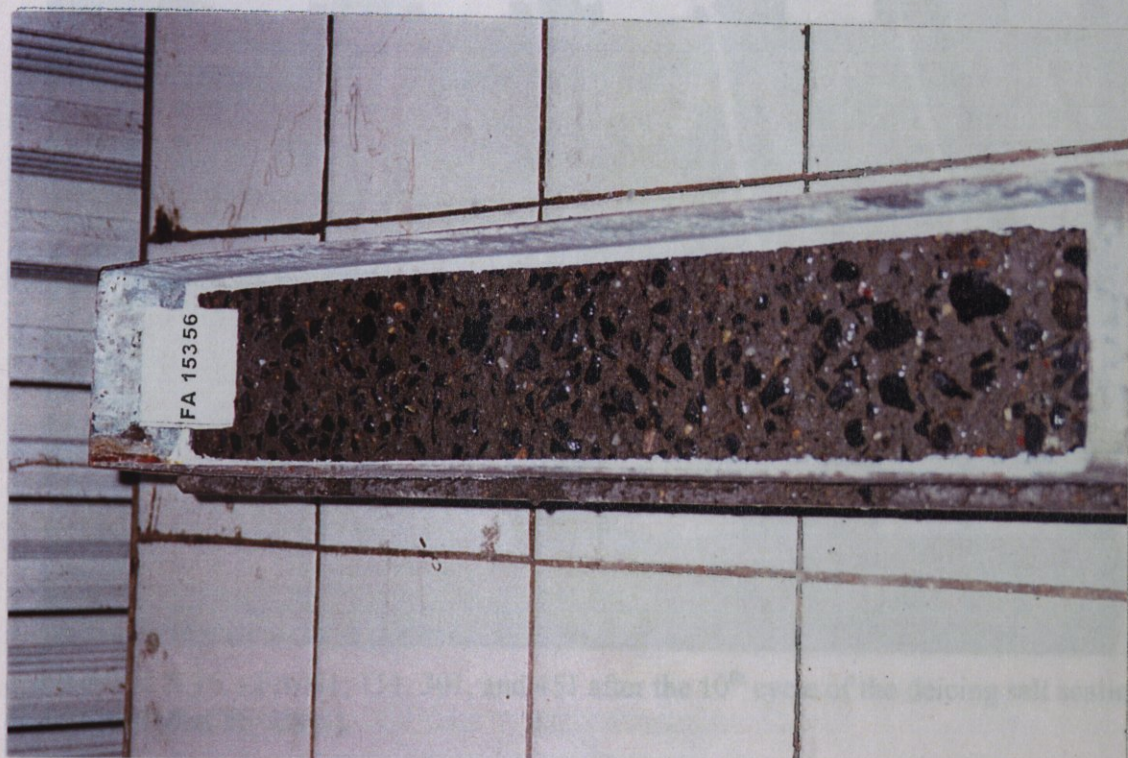


FIGURE A.15. - FA153 after the 35<sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days). (test terminated)



FIGURE A.14. - FA003 after the 30<sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days). (test terminated)



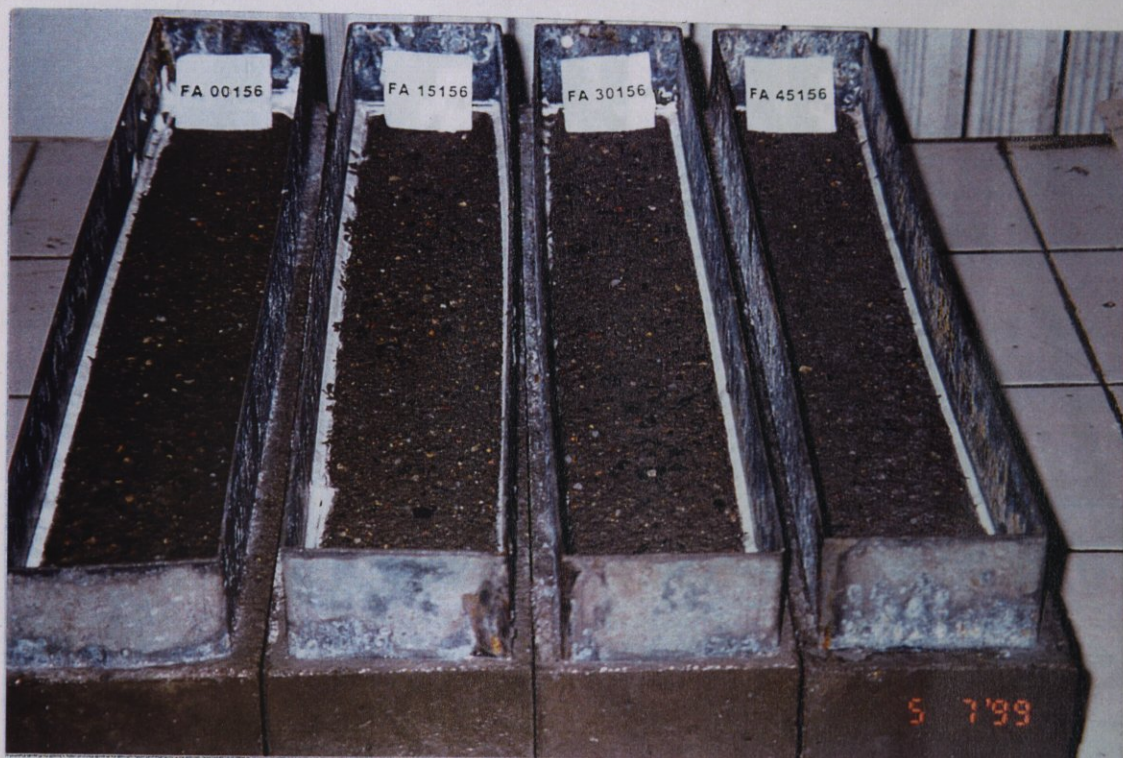


FIGURE A.16. - FA001, 151, 301, and 451 after the 10<sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days).



FIGURE A.17. - FA002, 152, 302, and 452 after the 20<sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days).





FIGURE A.18. - FA003, 153, 303, and 453 after the 15<sup>th</sup> cycle of the deicing salt scaling test (tested at 56 days).



FIGURE A.19. - Rapid Chloride Permeability Test Setup.



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