EVALUATION OF AIR VOID PARAMETERS OF FLY ASH INCORPORATED SELF CONSOLIDATING CONCRETE BY IMAGE PROCESSING

A THESIS SUBMITTED TO
THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES
OF
MIDDLE EAST TECHNICAL UNIVERSITY

BY

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IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR
THE DEGREE OF DOCTOR OF PHILOSOPHY IN
CIVIL ENGINEERING

OCTOBER 2009
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Self consolidating concrete (SCC) is defined as an innovative concrete that does not require vibration for placing and compaction and it is able to flow under its own weight, completely filling formwork and achieving full compaction. Although significant amount of research has been carried out regarding the fresh properties, mix design, placing methods and strength of various SCC mixes, only a very limited amount of work has been done to assess the durability performance of SCC.

Concretes in cold climates are subjected to freeze-thaw cycles which are one of the major durability problems, and if the concrete is in a saturated or nearly saturated condition, those cycles lead to expansion of the water in the capillary pores of concrete causing great internal stresses. For a durable concrete subjected to freeze-thaw cycles, an adequate air void system is obtained by using air-entraining admixtures. The performance of the air void system is...
characterized by air void parameters that are determined using microscopical examination of the concrete microstructure. In this thesis a software tool, based on image analysis of concrete surface, is developed to evaluate the air void parameters of concrete using both American and European standards.

Later on, an experimental program is conducted to evaluate the effect of freezing-thawing on self consolidating concrete that contain different percentages of fly ash (FA) and air entraining agents. For this purpose, a total of ten self consolidating concrete mixtures that contain four different contents of fly ash, and three different levels of air entrainment were prepared. During the casting operation, the workability properties of SCCs were observed through slump flow time and diameter, air content, V-funnel flow time, L-box height ratio, and segregation ratio. Hardened properties were evaluated by compressive strength, permeability tests (water absorption, sorptivity and rapid chloride permeability test), freezing-thawing test, resonant frequency test, ultrasonic pulse velocity test. The developed tool was used to characterize and evaluate the effects of air void parameters of SCC on its resistance to freeze-thaw cycles.

At the end of this experimental investigation, it was concluded that the addition of air entraining agent increased the flowability and an increase in the fly ash content decreased the effect of air entraining agent. On the other hand, during image processing, it was observed that the surface preparation procedures have a crucial effect on processing quality. Moreover, spacing factor - which is the most important air void characteristic that is utilized for determination of the resistance to freezing-thawing - should not be restricted to 0.2 mm for SCC, since SCCs with spacing factors smaller than 0.4 mm could still exhibit good freeze-thaw resistance.

Keywords: Self Consolidating Concrete, Fly Ash, Freeze-Thaw Resistance, Air Void Parameters, Image Processing
ÖZ

ÇEVRE KÜL İÇEREN KENDİLİĞİNDEN YERLEŞEN BETONUN HAVA BOŞLUĞU
ÖZELLİKLERİİNİN GÖRÜNTÜ ANALİZİ İLE BELLİLENMESİ

Özerkan, Nesibe Gözde
Doktora, İnşaat Mühendisliği Bölümü
Tez Yöneticisi: Doç.Dr. İsmail Özgür Yaman

Ekim 2009, 131 sayfa

Kendiliğinden yerleşen beton (KYB), yerleştirme ve sıkıştırma sırasında vibrasyon gerektirmeyen, kendi ağırlığı altında tam sıkıştırma yaparak kalıbı tamamen doldurabilen beton teknolojisinin yeni bir ürünü olarak tanımlanmaktadır. Çeşitli KYB karışımının taze beton özellikleri, karışım dizaynı, yerleştirme metotları ve dayanım özellikleri ile ilgili literatürde çok sayıda çalışma olması rağmen dayanıklılık özellikleri ile ilgili çok sınırlı sayıda çalışma bulunmaktadır.

Soğuk havalarda beton en önemli dayanıklılık problemlerinden biri olan donma-
çoğunluk döngülerine maruz kalır ve doygun veya yaklaşık doygun durumda ise bu
döngüler büyük içsel gerilimlere sebep olarak betonun kapiler boşluklarından suyun genleşmesine sebep olurlar. Donma-çoğunluk döngülerine maruz kalan
dayanıklık betonlar için uygun hava boşluğu sistemi hava sürükleyici katkılar
kullanılarak elde edilir. Hava boşluğu sisteminin performansı, betonun mikro
yapısının mikroskopik incelenmesi ile elde edilen hava boşluğu parametreleri ile
Bu tezde Amerikan ve Avrupa standardlarını kullanarak betonun hava boşluğu parametrelerini belirlemek amacı ile beton yüzeyinin görüntü analizine dayandırılarak bir yazılım geliştirilmiştir.


Bu araştırmanın sonucunda, hava sürükleyici katkı ilave edilmesinin akışkanlığı artırdığı ve uçucu kül miktarı artışının hava sürükleyici etkisini azalttığı sonucuna ulaşılmıştır. Diğer taraftan, görüntü analizi sırasında, yüzey hazırlama prosedürünün görüntü analizinin kalitesin üzerinde çok önemli etkisi olduğu gözlemlenmiştir. Ayrıca, betonun donma-çözülmeye karşı dayanıklılığının değerlendirilmesinde kullanılan hava boşluğu özelliklerinin en önemlilerinden biri olan boşluk faktörü değerinin 0.4’ten küçük olması durumunda da beton hala donma-çözülmeye karşı dayanıklı olabileceğinden, boşluk faktörü değerinin KYB için 0.2 ile sınırlandırılması gerektiği sonucuna ulaşılmıştır.

**Anahtar Kelimeler:** Kendiliğinden Yerleşen Beton, Uçucu Kül, Donma-Çözülme Dayanımı, Hava Boşluğu Parametreleri, Görüntü Analizi
To My Husband, **M. Kemal Özerkan**
ACKNOWLEDGMENTS

First and foremost I offer my sincerest gratitude to my supervisor, Assoc.Prof.Dr. İ. Özdür Yaman, who has supported me throughout my thesis with his patience and knowledge. I attribute the level of my Ph.D. degree to his encouragement and effort and without him this thesis would not have been completed or written. I am indebted to him more than he knows.

Secondly, I would like to thank my godfather, Prof.Dr. Mustafa TOKAY not only for his valuable suggestions about this thesis but also for his support about any trouble I have faced since the beginning of my life at METU.

I also gratefully thank to my committee members for their constructive comments on this thesis. Moreover, I appreciate Assoc.Prof.Dr. Ahmet Türer and Assoc.Prof.Dr. Murat Güler for their valuable contributions.

Also I would like to thank my friends, Mustafa Şahmaran, Dilak Okuyucu, Evin Soysal, Bahadir Keskin, Özlem Kasap Keskin, Haydar Tokgöz and Emre Akın, and also to my students Peyman Jafari and Emre Esastürk for their technical and moral support. Additionally, words alone cannot express the thanks I owe to Burhan Alam, my spiritual brother, for his encouragement and assistance.

And of course, I am very grateful to Paksoy and Özerkan families for their encouragement and patience. Moreover, I am obliged to both Sevinç Paksoys and Yağmur Paksoy in my life to enable me taking breath.
Words fail me to express my appreciation to my husband M. Kemal Özerkan whose dedication, love and persistent confidence in me, has taken the load off my shoulders and whose patient love enabled me to complete this work.

I am thankful to Turkish Scientific and Tecnological Research Council, TÜBİTAK for their scholarship support during my Ph.D. study.

Thanks are also due to Şahismail Tekin, Cuma Yıldırım and Ali Sünbüle for their assistance with laboratory work.
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LIST OF ABBREVIATIONS

ACI : American Concrete Institute
AE  : Air Entraining
ASTM : American Society for Testing and Materials
DF  : Durability Factor
EFNARC : European Federation of National Trade Association
EN  : European Norms
FA  : Fly Ash
GGBS : Ground Granulated Blast Furnace Slag
PC  : Portland Cement
PFA : Pulverized-fuel ash
RCPT : Rapid Chloride Permeability Test
RGB : Red-Green-Blue
SCC : Self Consolidating Concrete
SP  : Superplasticizer
TS  : Turkish Standards
UPV : Ultrasonic Pulse Velocity
W/C : Water-Cement Ratio
W/CM : Water-Cementitious Material Ratio
LIST OF SYMBOLS

$A$ : Air Voids
$T_t$ : Total Traverse Length
$T_p$ : Traverse Length through Paste
$T_a$ : Traverse Length through Air
$N$ : Number of Voids
$n$ : Void Frequency
$\bar{l}$ : Average Chord Length
$r$ : Paste-Air Ratio
$\alpha$ : Specific Surface
$\bar{L}$ : Spacing Factor
$E$ : Dynamic Modulus of Elasticity
$\rho$ : Density
$V$ : Wave Velocity
CHAPTER 1

INTRODUCTION

1.1. General

High performance concrete is defined in American Concrete Institute (ACI) as concrete meeting special combinations of performance and uniformity requirements such as enhancements in long-term mechanical properties, early-age strength, toughness, volume stability, or service life in severe environments [ACI Committee Report 363]. As concrete technology advances many types of high performance concrete like air-entrained high performance concrete, foamed concrete, high strength concrete, lightweight concrete, heavyweight high performance concrete, self consolidating concrete, sprayed concrete, waterproof concrete, etc. are being developed [Aïtcin, 1998].

Considered as a special type of self consolidating concrete (SCC) can be placed and compacted under its own weight with little or no vibration effort. SCC was first developed in Japan in the mid-1980s, and it has been further improved in the last decade utilizing various materials such as pulverized-fuel ash (PFA), ground granulated blast furnace slag (GGBS) and fly ash (FA) [Okamura and Ouchi, 2003]. The main reasons for the employment of self consolidating concrete and the advantages of SCC are [Kodama, 1997; Lachemi et.al., 2000]:

- Improving the durability of concrete;
- Reducing the construction time and labor cost;
- Eliminating the need for vibration, especially in confined zones where vibrating compaction is difficult;
- Reducing the noise pollution;
- Improving the filling capacity of high congested structural members; and
- Facilitating constructability and ensuring good structural performance.

Although significant amount of research has been carried out regarding the fresh properties, mix design, placing methods and strength of various SCC mixes, only very limited amount of work has been done to assess the durability performance of SCC. Current studies on durability characteristics of SCC are mostly studied through characterization of its permeability namely, water absorption, initial surface absorption, water permeability, and chloride permeability [Şahmaran, 2006; Zhu and Bartos, 2002]. By observing the reduction in its permeability, those studies have postulated that SCC has better durability properties.

When concrete is subjected to the freezing-thawing cycles which are one of the most major durability problems, and if it is in saturated condition, those cycles lead to expansion of the water in the capillary pores of concrete and cause great internal stresses, causing a reduction in its durability. The vital role of air entrainment in preventing freeze-thaw damage in concrete is well known and by using an air entraining agent, an air void system is established and it has several parameters which are considered important indicators of freeze-thaw resistance. These parameters were first characterized by Powers by providing an approximate representation of the air void structure in the cement paste, and in the case of this factor, it is assumed that the spheres all have the same radius, and are uniformly distributed in space. According to Powers, for a spacing factor of less than 0.2 mm, concrete has a significantly better freeze-thaw durability than those with spacing factors above this threshold which was later on accepted by other researchers [Powers, 1949; Snyder, 1998; Scott, 1997; Mehta and Monterio, 2006].
1.2. Research Objectives

This thesis consists of four parts which have four main objectives. These are:

- To develop a tool to characterize the air void system of concrete specimens.
- To develop a tool to determine the resonant frequency of concrete specimens which is used to nondestructively evaluate the freeze-thaw durability of concrete.
- To evaluate the effectiveness of the content of the fly ash and the air entraining admixtures in the freeze-thaw durability of self-consolidating concrete.
- To evaluate the effect of curing conditions on freeze-thaw durability of self-consolidating concrete.

Intended for these objectives, a total of ten self-consolidating concrete mixtures including four different contents of fly ash as a replacement of cement (0%, 15%, 30% and 45% by weight) and three different air entraining agent contents (0%, 0.15% and 0.30%) were prepared. All concrete mixtures have same superplasticizer content as 1.3% of cementitious materials by weight. A total of 29 cylinder specimens were prepared from each SCC mix. The fresh properties of SCCs were observed through, slump flow time and diameter, V-funnel flow time, L-box height ratio, air content and segregation ratio tests. The hardened properties included the compressive strength, freezing-thawing test, ultrasonic pulse velocity test, resonant frequency test, permeability tests (water absorption, sorptivity and rapid chloride permeability tests). Image processing was applied with the developed tool to characterize the air void system and to determine the air void parameters of concrete specimens.
1.3. Scope

This thesis consists of six chapters. Chapter 2 presents a literature review and devotes general background on SCC and durability properties of concrete. Freezing-thawing effect on concrete and the factors that affect the resistance of freezing-thawing are also evaluated in this chapter. At the end of the chapter, nondestructive testing methods used to evaluate concrete properties are also presented. The developed image processing method that can be utilized for the determination of air void characteristics is presented in Chapter 3. In Chapter 4, details about the experimental program, material properties, and test methods on fresh and hardened properties of SCC are given. The results of the test program are presented and discussed in Chapter 5. In that Chapter, the relationship between durability factor and fly ash and air entraining agent content was evaluated, and air void characteristics are assessed. Chapter 6 presents a summary of thesis and lists the findings of this research. Suggestions for future studies are also included in that chapter.
CHAPTER 2

LITERATURE REVIEW AND BACKGROUND

2.1. High Performance Concrete

Since the first US patent covering the fabrication and utilization of water reducers based on polycondensates of naphthalene sulfonate was obtained in 1938, the concrete industry started to use superplasticizers in the 1960s in both Japan and Germany [Tucker, 1932; Hattori, 1981; Meyer, 1981]. On the other hand, in the 1970s, the concrete used in the columns of some high-rise buildings in Chicago had higher compressive strength value (about 60 MPa) than that of the usual concretes used in the construction, and they were called as “high-strength concretes”. They were used because of their higher strength, and were made using the same technology of usual concrete except that the materials used to make them were carefully selected and controlled [Freedman, 1971; Blick et al, 1974]. However, it was found that concretes with a very low water/cement or water/binder ratio also had other improved characteristics, such as higher modulus of elasticity, higher flexural strength, higher flowability, lower permeability, improved abrasion resistance and better durability. Thus the statement “high-strength concrete” no longer described the overall improvement in the properties of this new concrete [Malier, 1992]. Therefore the expression “high-performance concrete” was started to be used. The high performance concrete is defined in the American Concrete Institute (ACI) as concrete meeting special combinations of performance and uniformity requirements that cannot always be achieved routinely when using conventional constituents and normal mixing, placing and curing practices. The requirements may involve
enhancements in long-term mechanical properties, early-age strength, toughness, volume stability, or service life in severe environments [ACI Committee Report 363].

Although the development of high-performance concrete is quite recent, there are already high-performance concretes that have gained the qualification of special like air-entrained high-performance concrete, high workability concrete, self consolidating concrete (SCC), foamed concrete, high strength concrete, lightweight concrete, heavyweight high performance concrete, no-fines concrete, pumped concrete, sprayed concrete, waterproof concrete, autoclave aerated concrete and roller compacted concrete etc. [Aïtcin, 1998]. Among these applications, the air-entrained high performance concrete containing a stable air bubble system is used to protect the concrete from repeated freezing and thawing cycles. On site productivity can be greatly increased by utilizing the high workability concrete such as self-consolidating concrete. Self consolidating concrete is defined as an innovative concrete that does not require vibration for placing and compaction offering a rapid rate of concrete placement with faster construction times and ease of flow around congested reinforcement. They are especially suitable for the applications like inaccessible locations, large flat areas, underwater applications, and pumping concrete over long distances etc. [Aïtcin, 1998; EFNARC, 2005].

In this chapter, the properties of self-consolidating concretes and air-entrained high performance concrete will be provided that constitutes the object of this study.

2.1.1. Self Consolidating Concrete

The necessity of the concrete which can be compacted into every corner of a formwork, purely by means of its own weight and without the need for vibrating compaction was proposed by Okamura in 1986, and the studies to develop self-
consolidating concrete, including a fundamental study on the workability of concrete, were carried out by Ozawa and Maekawa at the University of Tokyo [Ozawa, 1989]. Since 1988 in which the development of the prototype of self-consolidating concrete was first completed, the use of self-consolidating concrete in actual structures has gradually increased [Kodama, 1997; Lachemi et al., 2000].

According to the European Federation for Specialist Construction Chemicals and Concrete Systems (EFNARC) self-consolidating concrete is defined as an innovative concrete that does not require vibration for placing and compaction and it is able to flow under its own weight, completely filling formwork and achieving full compaction, even in the presence of congested reinforcement [EFNARC, 2005]. In that guideline, the properties of SCC in its fresh and hardened state and also the test methods used to support this specification are described. The specific requirements in the guideline for SCC in the fresh state depend on the type of application, and especially on confinement conditions, placing equipment, placing methods and finally finishing method. The guideline states the key characteristics to cover these requirements as flowability, viscosity, passing ability and segregation resistance:

- **Flowability** is the ease of fresh SCC when unconfined by formwork and/or reinforcement
- **Viscosity** is the resistance of SCC once flow has started
- **Passing Ability** is the capacity of SCC to flow through congested sections such as spaces between steel reinforcing bars without segregation, under its own weight
- **Segregation Resistance** is the ability of SCC to remain homogeneous in composition during and after casting.

Since a single test method which can measure and quantify all the above mentioned requirements of SCC does not exist, a list of test methods is described in the guideline to evaluate the fresh state characteristics (Table 2.1).
Among those, slump-flow, L-Box, U-Box, V-funnel and GTM sieve stability tests are the most widely used tests [EFNARC, 2005; Okamura and Ouchi, 2003].

Table 2.1 Test Properties and Methods for Evaluating Fresh State Characteristics of SCC

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Test Method</th>
<th>Measured Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flowability/Filling Ability</td>
<td>Slump-flow</td>
<td>Total Spread</td>
</tr>
<tr>
<td></td>
<td>Kajima Box</td>
<td>Visual Filling</td>
</tr>
<tr>
<td>Viscosity/Flowability</td>
<td>$T_{500}$</td>
<td>Flow Time</td>
</tr>
<tr>
<td></td>
<td>V-Funnel</td>
<td></td>
</tr>
<tr>
<td></td>
<td>O-Funnel</td>
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<td></td>
<td>Orimet</td>
<td></td>
</tr>
<tr>
<td>Passing Ability</td>
<td>L-Box</td>
<td>Flow Time</td>
</tr>
<tr>
<td></td>
<td>U-Box</td>
<td>Passing Ratio</td>
</tr>
<tr>
<td></td>
<td>J-Ring</td>
<td>Height Difference</td>
</tr>
<tr>
<td></td>
<td>Kajima Box</td>
<td>Step Height, Total Flow</td>
</tr>
<tr>
<td>Segregation Resistance</td>
<td>Penetration</td>
<td>Depth</td>
</tr>
<tr>
<td></td>
<td>Sieve Segregation</td>
<td>Percent Laitance</td>
</tr>
<tr>
<td></td>
<td>Settlement Column</td>
<td>Segregation Ratio</td>
</tr>
</tbody>
</table>

Many other investigators dealt with the mixture design problems of SCC and different approaches have been used. One such method considers that the successful development of SCC requires a careful control of the rheological properties of matrix, content of supplementary cementitious materials (SCM), as well as the content and particle size distribution of coarse aggregates. Moreover, the use of fly ash and blast furnace slag in SCC reduces the dosage of superplasticizer needed to obtain similar slump flow compared to concrete made with Portland cement, only. Moreover, the use of fly ash improves rheological properties and reduces cracking of concrete due to the heat of hydration of the cement [Yahia et al., 1999; Lachemi et al., 2000].
Although significant amount of research has been carried out regarding the fresh properties, mix design, placing methods and strength of various SCC mixes, only very limited amount of work has been done to assess the durability performance of SCC. In fact, most of the work is about determining the preparation properties of SCC, mixes which is known to be directly related to durability. For example, Zhu and Bartos (2002) studied the permeation properties of SCC, which include permeability, absorption, diffusivity etc and which have been widely used to quantify durability characteristics of concrete. They concluded that the SCC mixes showed significantly lower values of coefficient of permeability and the SCC mix using no additional powder but a viscosity agent to maintain stability of the fresh mix had the highest permeability, sorptivity and chloride diffusivity. Another research studied by Şahmaran (2006) showed that the durability of SCC evaluated by its permeability as measured by RCPT, absorption and the sorptivity tests, addition of fly ash (especially low-lime fly ash) seemed to be beneficial leading to a more durable concrete.

As the object of this study is related to one of the durability of SCC, in the following sections the durability of concrete in general and freeze-thaw resistance in specific will be covered.

2.1.2. Durability of Concrete

The physical causes of concrete degradation are grouped into two categories by Mehta and Gerwick [Mehta and Monteiro, 2006]:

a. Loss of mass or surface wear due to abrasion, erosion, and cavitation;

b. Cracking due to normal temperature and humidity gradients, crystallization of salts in pores, structural loading, and exposure the extreme temperature such as freezing.
Similarly, they grouped the chemical causes of concrete degradation into three categories:

a. Hydrolysis of the cement paste components by soft water;
b. Cationic reactions between aggressive fluids and the cement paste;
c. Reactions leading to formation of expansive products, such as in the case of sulphate attack, alkali-aggregate reaction, and corrosion of reinforcing bars in the concrete.

A durable concrete should retain its original form, quality, and serviceability when exposed to both physical and chemical processes. Therefore, durability of portland cement concrete is defined by ACI Committee 201, as the ability of concrete to resist weathering action, chemical attack, abrasion or any other process of deterioration while maintaining its desired engineering properties [ACI Committee Report 201]. The use of wrong materials or false design, improper detailing, insufficient quality control, inadequate curing, etc. reduce the service life and durability of concrete [Ramachandran and Beaudoin, 2001; Mehta and Monterio, 2006; Erdogan, 2003; Basheer et al., 2001].

There are three requirements for a durable concrete [Ramachandran and Beaudoin, 2001; Mehta and Monterio, 2006; Basheer et al., 2001]:

1. **Mix Design:** Mix must be designed to ensure cohesion and prevent segregation and bleeding. Limiting the maximum water/cement ratio and/or minimum cement content may guarantee on adequate durability for the particular site conditions. If cement is reduced then at fixed water/cement ratio, the workability will be reduced leading to inadequate compaction. If water is added to improve workability, water/cement ratio increases resulting in a higher permeability which is the most important factor for durability. Moreover, when designing the mixture proportions, the exact environmental conditions to which the concrete will be subjected during its service life should be taken into consideration.
2. Construction Processes: For a durable concrete, it is also necessary to be sure that concrete is placed and cured in a correct manner. The concrete as a whole contain voids that can be caused by inadequate compaction, and these voids decrease the durability of concrete. The workability of the mix must match to compaction equipments, the type of formwork, and the density of steelwork. Furthermore, curing is very important to permit proper strength development aid for moisture retention and to ensure hydration process since premature drying of concrete surface can have a catastrophic effect on the durability of concrete.

3. Ingredient Properties: The properties of concrete ingredients such as cement, aggregates, admixtures and pozzolanic materials, and their interactions have also an important effect on concrete durability.

2.1.2.1. Freeze-Thaw Durability of Concrete

Concretes in cold climates are subjected to freeze-thaw (F-T) cycles, and if the concrete is in a saturated or nearly saturated condition, those cycles lead to expansion of the water in the capillary pores of concrete causing great internal stresses that take place upon freezing and thus expanding of the water in the capillary pores of concrete. Frost damage in concrete can take several forms like cracking, spalling, scaling and D-cracking. The deterioration of concrete under the action of freezing and thawing is a very complex phenomenon and a variety of explanations have been presented for the deterioration mechanism. The first important explanation was presented by Powers and he stated that the degradation due to freezing and thawing is caused by a combination of hydraulic and osmotic pressures that are induced by the increase in volume that water experiences when it changes from a liquid phase to a solid phase. According to the Hydraulic Pressure hypothesis based on the studies of Powers, the moisture contained within the saturated capillary voids would begin to freeze once the temperature of concrete dropped below 0°C, and the part that has already
frozen and expanded exerts a force on the unfrozen water in the pore, and drives the unfrozen water out of the pore creating hydraulic pressure. On the other hand, after 1950s, the studies by Powers and Helmuth showed strong evidence that the hydraulic pressure hypothesis was not consistent with the experimental results, and they discovered that freezing non-air entrained concrete paste first underwent shrinkage and then expansion during freezing. Moreover, they determined that the temperature at which water freezes depended upon the size of the void in which that water resided. They theorized that the water in the capillary and gel pores would be at thermodynamic equilibrium until the temperature of the paste dropped below the freezing point, and at that point there is an initial period of supercooling and then ice crystals start forming in the larger capillary voids while the water in the smaller gel pores remained bonded within the sheets of C-S-H. Since the water in cement paste is in the form of a weak alkali solution, the alkali content of the unfrozen portion of the solution in these capillaries increases creating an osmotic pressure which impels water in the nearby unfrozen pores to begin diffusing into the solution in the frozen cavities. These expansive pressures cause the failure of the paste and reduce the durability of concrete, and Figure 2.1 shows typical results of hydraulic and osmotic pressures when they are repeatedly induced in cement paste [Mehta and Monterio, 2006; Carlson, 2005; Erdogan, 1997; Scott, 1997].
Ice formation process is also studied most recently by other investigators. One of those studies was performed by Kaufmann (2004), who studied on a series of experiments, the freezing process, its initiation, continuation and the resulting damage. For this purpose, calorimetric, expansion and acoustic methods were applied to monitor heat release, mechanical deformation and damage during a series of frost cycles. At the end of his study, he separated the ice formation into two main phases: an instantaneous nucleation part and a progressive ice penetration part. He also demonstrated that big hysteresis effects between freezing and thawing can be partly avoided when ice nucleation is initiated through special ice formation germs or in a macroscopic liquid layer. Freezing of pore solution in concrete exposed to a freeze-thaw cycle was studied also by Cai and Liu (1998), by following the change of concrete’s electrical conductivity at freezing temperatures. In their study, concrete samples were subjected to freeze-thaw cycles with temperature varying between 0°C and -20 °C, and it was concluded that in the freezing process, more pore solution in concrete freezes above -10°C than below -10°C. Furthermore they concluded that to ordinary
concrete, although pore solution goes on freezing below -10°C, the consequent damage was so small that they can be neglected. However for high strength concrete, serious damage occurs by freezing below -10 °C.

When air voids are entrained into the cement paste, and the average distance between these voids is not too great, the water can seep from the cement paste into the air void where the pressure is lower, and thus further ice-accretion and expansion are prevented. Entrained air voids are characteristically spherical in shape and mainly range in between 10 μm and 1 mm, and usually exhibit a sensibly uniform distribution throughout the concrete matrix, except near the concrete surface and for poorly mixed concrete. Air-entrainment in concrete is achieved by two ways: first method is the addition of air entraining agents to the Portland cement during the final grinding stage of cement production, and second and most commonly used method is the addition of a liquid chemical admixture to the mixture. Air entraining admixtures are subdivided into those that are naturally occurring and those that are synthetically produced. Naturally occurred ones are obtained as byproducts of industries and they can be chemically classified as salts of wood resins, salts of sulfonated lignin, salts of petroleum acids, salts of proteinaceous materials, fatty and resinous acids and their salts, and organic salts of sulfonated hydrocarbons, and the synthetic detergents are the commercially produced air entraining admixtures. Most of the air entraining agents are organic materials and are classified as surface active agents or surfactants. The surface active agents are composed of a hydrocarbon chain and these molecules have distinct characters on each end while one end of the molecule is hydrophilic (water-attracting) and the other end is hydrophobic (water-repelling). When air entraining agents are added to the concrete mixture, these agents align themselves with the charged hydrophilic portion as seen in Figure 2.2, which can be anionic (negatively charged), cationic (positively charged) or nonionic (neutral), in the water and the hydrophobic portion in the air [Carlson, 2005; Erdogan, 1997; John et al., 1998] Since air bubble entrainment and stability in fresh concrete is an extremely complex problem, Du and Folliard studied the mechanisms of air entrainment in concrete and they stated that the
air void system is affected by every component in the concrete mixture, including Portland cement, aggregates, water and admixtures, the batch quantity and the placing methods [Du and Folliard, 2004].

![Figure 2.2. Schematic Representation of Anionic Air Entraining Agent [Carlson, 2005]](image)

In Figure 2.3, an area called as the protected paste region which describes the portion of the cement paste that is close enough to an air void that it can provide pressure relief for the water in that paste can be seen [Scott, 1997].

![Figure 2.3. Schematic Description of Protected Paste Region [Scott, 1997]](image)
Many experimental studies have been conducted to characterize the spacing of air voids and to establish measurable geometric parameters that reflect freeze-thaw durability by first Powers, which forms the basis for the American Society for Testing and Materials (ASTM) C 457 spacing factor ($\bar{L}$). The Powers spacing factor provides an approximate representation of the air void structure in the cement paste, and in the case of this factor, it is assumed that the spheres all have the same radius, and are uniformly distributed in space. The Powers spacing factor is defined as the half of the maximum distance between the boundaries of adjacent spheres, and for a spacing factor of less than 0.2 mm, concrete has a significantly better freeze-thaw durability than those with spacing factors above this threshold. It was developed using two idealized systems [Snyder, 1998]:

- For small values of the paste/air ($p/A$) ratio, there is very little paste for each air void. Powers used the “frosting” approach of spreading all of the paste in a uniformly thick layer over each air void whose thickness is approximately equal to the ratio of the volume of paste to the total surface area of air voids.

$$\bar{L} = \frac{p}{4R^2} = \frac{p}{\alpha A} \quad \text{if} \quad p/A < 4.342 \tag{2.1}$$

where $R$ is the radii of sphere and $\alpha$ is the surface area of the air voids.

- For large values of the $p/A$ ratio, he used the cubic lattice approach and supposed that the spheres are placed at the vertices of a simple cubic array, the air voids are monosized, each with a specific surface area equal to the bulk value (Figure 2.4). The cubic lattice spacing was chosen such that the air content equals to the bulk value. The resulting spacing factor was defined as the distance from the center of a unit cell to the nearest air void surface.
where \( p \) is the paste content, \( A \) is the air content, \( R \) is the radius of the voids, and \( \propto \) is the specific surface defined as the surface area of the air voids divided by their volume. The \( p/A \) value of 4.342 is the point at which these two equations are equal.

\[
L = \frac{3}{\propto} \left[1.4 \left(\frac{P}{A} + 1\right)^{1/3} - 1\right] \quad \text{if} \quad \frac{p}{A} \geq 4.342
\]  

(2.2)

The spacing factor has been also proposed by Philleo, Attiogbe, and Pleau and Pigeon. Philleo extended the approach of Powers by attempting to quantify the volume fraction of paste within some distance of an air void system. He started with an idealized air void system composed of randomly distributed points using the paste-void proximity distribution for zero-radius points. For an air-paste system, the Philleo spacing factor for the volume fraction of paste within a distance \( s \) of an air void surface is defined as seen in Equation 2.3 [Snyder, 1998].
\[ F(s) = 1 - \exp[-4.19x^3 - 7.80x^2(\ln(1/p))^{1/3} - 4.84(\ln(1/p))^{2/3}] \] (2.3)

where \( x = sn^{1/3} \).

Recently, Attiogbe proposed a spacing equation which estimates the mean spacing of air voids in concrete [Snyder, 1998]:

\[
\bar{s} = 2F \frac{p^2}{\infty A}
\] (2.4)

where \( F \) is the factor that is equivalent to the probability factor defined in Philleo’s protected paste volume concept, and it is determined as in Eq. 2.5.

\[
F = \begin{cases} 
\frac{8}{p/A + 1} & p/A \geq 7 \\
1 & p/A < 7 
\end{cases}
\] (2.5)

Pleau and Pigeon have also recently proposed a spacing equation for the paste-void spacing distribution considering both the air void radii distribution and the distribution of distances between a random point in the paste and the nearest air void center. The fundamental equation of Pleau and Pigeon is given in Equation 2.6 [Snyder, 1998].

\[
k(s) = \int_0^\infty h(r+s) f(r) \theta(r+s) dr
\] (2.6)

where \( h(r+s) \) represents the probability density function of the distance between a random point in the system and the center of the nearest air void,
\( f(r) \) represent the probability density function of air void radii, the Heaviside function \( \theta(r + s) \) insures that the argument of the function \( h \) remains positive.

The spacing of voids in air-entrained concrete is characterized by each of these equations, the Attiogbe equation estimates the spacing among air voids, and other equations estimate the distance water must travel to reach the nearest air void. Two of these approaches, the Power spacing factor and the Philleo factor, have become widely accepted as excellent predictors of freeze-thaw durability [Snyder, 1998; Scott, 1997; Mehta and Monteiro, 2006; ASTM C 457].

2.1.2.2 Effect of Curing

The hydration of the cement is defined as the cement-water reaction which includes both chemical and physical processes. The rate and extent of hydration depend on the availability of water. Therefore, it can be said that the objectives of concrete curing are preventing the loss of moisture and controlling the temperature of concrete during cement hydration so that it can develop the properties that the mixture was designed to achieve. Since drying may remove the water needed for hydration and concrete may not achieve its potential properties in this situation, curing has a strong influence on the properties of hardened concrete such as durability, strength, permeability, and volume stability. Furthermore, inadequate or insufficient curing is one of the main factors contributing to weak, powdery surfaces with low abrasion resistance, and also if the concrete is allowed to dry out quickly, it may undergo considerable early age drying shrinkage [ACI Committee Report 308; Mehta and Monteiro, 2006].

In 1948, Powers studied the relation between the hydration of cement and curing of concrete, and pointed out that the development of both strength and durability in concrete not only depends on the degree to which the cement has hydrated but also the degree to which the pores between the cement particles
have been filled with hydration products. He also demonstrated that concrete mixtures with a water to cement (w/c) ratio less than 0.50 and sealed against loss of moisture cannot develop their full potential hydration due to lack of water, and such mixtures would therefore benefit from externally applied curing water [Powers, 1948]. High performance concrete has a very low water content and the developing capillary pores are consequently very small. Unless the loss of water from the surface of the concrete is prevented, this would lead to plastic shrinkage, and hence the need for wet curing from the earliest possible moment must be continued until the strength of the hydrating cement paste is high enough to resist internal microcracking. Neville and Aïtcin (1997) studied the distinct shrinkage behavior of high performance concrete and the reasons for an absolute necessity of wet curing, and they stated that high performance concrete that has not been very well cured will be of poor quality. On the other hand the effects of self-desiccation which refers to the process by which concrete dries itself from the inside should be taken into consideration in high performance concretes with low water to cement ratio. To prevent early-age self-desiccation, water that is consumed by hydration has to be replaced by the ingress of external moisture [Carino and Meeks, 2000].

Curing at early age is especially important for concretes having blended cements. Iyoda et al., in their research, investigated the relationship between curing and durability for the concrete using blast-furnace slag blended cement as compared with concrete using ordinary Portland cement. As a result of their study, they concluded that the concrete obtained by using the blended blast-furnace slag cement type was more sensitive to curing than the ordinary Portland cement concrete since the larger pores were remained at a curing period of 5 days for concretes having blended blast-furnace slag cements. This means that the blended cement concrete needed longer curing periods. In blended cements, the pozzolanic reaction between a pozzolan and calcium hydroxide is slow; the strength development will be accordingly slow [Iyoda et al., 2008; Mehta and Monteiro, 2006]. Another research was studied by Güneyisi et al. and they investigated the effects of cement type and curing condition on the chloride
permeability experimentally, and they concluded that the extension of curing period from 28 to 180 days and enhancement of the curing condition reduced the total charge passes through all concretes with the difference much more noticeable for blended cement concretes [Güneyisi et al., 2007].

2.1.2.3. Effect of Permeability

Since concrete is permeable to water to the extent, the increased permeability allows more water and chemical ions to get into the concrete and thus facilitates concrete deterioration and consequently adversely affects the overall concrete durability. It should be obvious that the size and volume of the interconnected capillary pores in the cement paste, and also the intensity of microcracks at the aggregate-cement paste interface as well as within the paste itself determine the permeability of concrete. Porosity and interconnectivity are controlled by the amount of cementitious materials, water content, aggregate grading, degree of compaction, degree of hydration and curing efficiency. On the other hand, intensity and location of interfacial microcracks are determined by the level of external or internal applied stress [Banthia, 2004; Lee, 2001; Şahmaran, 2006; Güneyisi et al., 2007; Mehta and Monteiro, 2006].

Since the water to binder ratio of usual concrete is high, the concrete is very porous and its capillaries being interconnected, the measurement of the water permeability of usual concrete is relatively easy depending on Darcy’s law. However, when such a water/binder ratio is lower than 0.40 like in high performance concrete, most permeameters are unable to register any significant water flow through the concrete sample, although a high pressure is applied on water. Therefore, high-performance concrete permeability is measured by another way such as chloride permeability and there is a strong correlation between the water/binder ratio and the chloride ion permeability of high performance concrete [Aïtcin, 1998]. Although the high performance concrete offers better durability properties and the permeability is one of the most
important parameter of measuring the durability of concrete, there are very limited research on the permeability of high-performance concrete. One such research was reported by Poon et.al. and Janotka and Bagel and they showed that permeability of all high strength concrete specimens increased drastically with the increase in the temperature, and concrete specimens showed a very low permeability at lower temperature degrees [Janotka and Bagel, 2003; Poon et al., 2001]. Zhu and Bartos (2002) also studied on the permeation properties of self-consolidating concrete and they concluded that SCC mixes showed significantly lower values of coefficient of permeability and sorptivity of water absorption compared to the traditional concrete.

Furthermore it is known that the use of pozzolanic materials such as blast furnace slag, fly ash, silica fume, etc. in concrete considerably reduces its permeability and the rate of diffusion of moisture. Several studies indicated that low-lime fly ash concretes at ages up to 28 days was more permeable than no-fly-ash concretes, but at 6 months, fly ash concretes became more impermeable than no-fly-ash concretes [Davis, 1954; Kanitakis, 1981]. However, the good resistance to the penetration of chloride ions was achieved at relatively early ages by addition of slag or silica fume as a supplemental cementitious material with a low water to cementitious materials ratio by Özyıldırım and Halstead. Fly ash can improve the permeability of concrete due to its capability of transforming large pores of concrete into small pores and reducing microcracking in the transition zone [Mehta and Monteiro, 1997]. The use of fly ash and slag also tends to reduce the resistance to scaling since it decreases the permeability while the total amount of freezable water remains more or less constant [Özyıldırım and Halstead, 1988].

2.2. Nondestructive Testing of Concrete Properties

Nondestructive test methods allow the materials to be inspected and measured without damaging them, and these test methods do not affect the future
usefulness of the material. There are two general classes of nondestructive testing methods for concrete: the first class includes those methods that are used to estimate strength, conversely the second class includes those methods that measure other characteristics of concrete such as moisture content, density, thickness, resistivity and permeability. According to this classification, the surface hardness, penetration resistance, pullout, break-off, rebound and maturity techniques belong to the first category, and dynamic or vibration techniques, radioactive and nuclear methods, magnetic and electrical methods and acoustic emission methods belong to the second category [Malhotra and Carino, 2004]. In this thesis, the resonant frequency test, ultrasonic pulse velocity test and image processing method were used to determine the dynamic modulus of elasticity, and air void parameters of concrete, respectively.

2.2.1. Resonant Frequency

Resonance is the tendency of a system to oscillate with high amplitude when excited by energy at a certain frequency which is known as the system’s natural frequency of vibration or resonant frequency. Resonant systems can be used to generate vibrations of a frequency or pick out specific frequencies from a complex vibration containing many frequencies. Resonant phenomena occur with all types of vibrations or waves such as mechanical resonance, acoustic resonance, electromagnetic resonance, electrical resonance, optical resonance, orbital resonance etc. The resonant frequency method was first developed by Powers in 1938 by matching the musical tone created by concrete specimens when tapped by a hammer with the tone created by one of a set of orchestra bells calibrated according to frequency. Other investigations on the development of this method included those by Hornibrook in 1939, by Thomson in 1940, by Obert and Duvall in 1941, and in all the tests the specimens were excited by a vibrating force [Ramachandran and Beaudoin, 2001].
The resonant frequency of vibration of a beam can be used to determine its dynamic modulus of elasticity; because in solid mechanics it is defined as a dynamic property of an elastic system and so it is primarily related to the dynamic modulus of elasticity and density in the case of a vibrating beam. The relationship between them is valid for homogeneous solid media which are isotropic and perfectly elastic, but they may be applied to heterogeneous systems such as concrete when the size of a specimen is large in relation to the size of its constituent materials. For flexural vibrations of a long-thin rod, the dynamic modulus of elasticity is calculated by using the following equation [Malhotra and Carino, 2004; Ramachandran and Beaudoin, 2001; Lamond and Pielert, 2006]:

\[ E = \frac{4\pi^2 L^4 N^2 d}{m^4 k^2} \]  

(2.7)

where \( E \) is the dynamic modulus of elasticity, \( d \) is the density of the material, \( L \) is the length of the specimen, \( N \) is the fundamental flexural frequency, \( k \) is the radius of gyration of the section about an axis perpendicular to the plane of bending (=\( t/12 \) for rectangular cross section where \( t \)=thickness), \( m \) is a constant which is 4.73 for the fundamental mode of vibration.

The dynamic modulus of elasticity can also be computed from the fundamental longitudinal frequency of vibration of a specimen as seen in Equation 2.8:

\[ E = 4L^2 dN^2 \]  

(2.8)

Equations 2.7 and 2.8 were obtained by solving the respective differential equations for the motion of a vibrating bar, in flexure in the free-free mode and in the longitudinal mode. Therefore, the resonant frequency of vibration of a concrete specimen is directly related to its dynamic modulus of elasticity [Malhotra and Carino, 2004].
ASTM C 666, entitled as “Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing”, requires the calculation of the relative dynamic modulus of elasticity and durability factor, and these calculations are based on the resonant frequency methods for studying the deterioration of concrete specimens subjected to repeated cycles of freezing and thawing. The relationship between the relative dynamic modulus of elasticity and the resonant frequency is defined in the ASTM C 215, entitled as “Standard Test Method for Fundamental Transverse, Longitudinal and Torsional Resonant Frequencies of Concrete Specimens”, by the following equation:

\[ E = DL^2 \rho N^2 \]  

(2.9)

where \( E \) is the dynamic modulus of elasticity, \( D \) is equal to 5.093*(\( L/d^2 \)) for a cylinder specimen and equal to 4*(\( L/bt \)) for a prism specimen, \( \rho \) is the density of the material, \( L \) is the length of the specimen, \( N \) is the fundamental longitudinal frequency, \( d \) is the diameter, \( b \) is the width and \( t \) is the thickness [Malhotra and Carino, 2004; ASTM C 666; Ramachandran and Beaudoin, 2001; ASTM C 215].

According to ASTM C 215, the resonant frequency is measured using one of two alternative procedures: (a) the forced resonance method, and (b) the impact resonance method. In the forced resonance method, the test system in which the vibration is produced by a small oscillating driver in contact with one end of the beam, and the response of the beam is picked up by a lightweight accelerometer at the other end. The amplitude of vibration varies along the beam, and the frequency of the driver is altered until maximum amplitude of vibration is detected by the accelerometer. On the other hand, in the impact resonance method, a supported specimen is struck with a small impactor and the specimen response is measured by an accelerometer on the specimen. After recording the output of the accelerometer, the fundamental frequency of vibration is determined by using digital signal processing methods. The
fundamental frequencies for the three different modes of vibration (longitudinal, transverse and torsional) are obtained by proper location of the impact point and the accelerometer [IIlston and Domone, 2001; Popovics, 1998; ASTM C 215]. In this thesis, the impact resonance method was used to determine the longitudinal resonant frequency of the concrete specimens.

2.2.2. Ultrasonic Pulse Velocity

During the 1914-18 war, Langevin made the first important use of ultrasonics for underwater soundings, and investigated the use of quartz transducers for transmitting and receiving ultrasonic waves. The use of ultrasonic waves in detecting metal objects was first practiced by Sokolov in 1929 and 1935. Mulhauser used two transducers to detect flaws in solids and obtained a patent for using ultrasonic waves in 1931, and Firestone in 1940 and Simons in 1945 used a pulse-echo technique and developed pulsed ultrasonic testing. After the 1939-45 war, the discovery of radar triggered the development of the pulse technique and it was used for the nondestructive testing of materials and for medical diagnosis. After 1950, the ultrasound was used to detect gallstones, breast masses, and tumors in the international medical community of United States and Europe. In the 1960s, microwave propagation was developed, and new materials and techniques were discovered, and also ultra-high frequencies, up to 100 GHz, were used to generate ultrasonic waves [Blitz 1971; Blitz and Inst 1963].

The measurement of ultrasonic velocities depends on propagating a dynamic pressure wave (pulse) through a material of known thickness and measuring the transit time of the emerging acoustic pressure wave [Brown 1997]. After measuring the transit time of wave, ultrasonic pulse velocity (UPV) is calculated by dividing the thickness of material to the transit time. In solids, sound waves are created in four principle modes when the surface of a solid elastic medium is disturbed by a dynamic or vibratory load: (1) Compressional waves (also called
longitudinal or P-waves), (2) Shear Waves (also called transverse or S-waves), (3) Surface waves (also called Rayleigh waves), (4) and Plate waves (also called Lamb waves) in thin materials. Among these waves, the most widely used modes of propagation used in ultrasonic testing are longitudinal and transverse waves. These waves travel through a uniform material at a constant velocity provided that the deformations produced by the action of the waves are purely elastic, and this velocity depends on the density and the modulus of elasticity for longitudinal waves as shown by the following equation [Krautkrämer, 1983; Malhotra and Carino, 2004]:

\[ V = \sqrt{\frac{E}{\rho}} \quad (2.10) \]

where \( V \) is the longitudinal wave velocity, \( E \) is the dynamic modulus of elasticity, and \( \rho \) is the density. As seen from the equation, sound waves travel at different speeds in different materials since the mass of the atomic particles related to the density of the material, and the elastic constants of the material are different [Blitz, 1971; Brown, 1997].

The wave velocity of ultrasonic waves can be determined by two modes of measurements: through transmission and pulse echo. In through transmission, two transducers are used; one is the transmitter and the other one is the receiver (Figure 2.5a). On the other hand, in the pulse echo procedure, only one transducer is used which delivers the pulse and also receives the reflected signal (Figure 2.5b). Through transmission is preferred in concrete because of its highly attenuative nature. Attenuation is the reduction in the energy of the wave as it passes through the medium [Yaman et.al. 1998].
The typical ultrasonic pulse velocity (UPV) testing system consists of several functional units which are pulser/receiver, transducer and display devices as schematically described in Figure 2.6.
UPV was first used for concrete by Leslie and Cheesman in Canada 1949. Leslie and Cheesman presented a new apparatus named Soniscope for field laboratory testing of concrete and they reported that using this apparatus, the presence of internal cracks in a block of mass concrete, the depth of surface cracks and the dynamic modulus of concrete in any part of a structure regardless of the shape of the structure were obtained [Jones, 1962]. The determination of the dynamic modulus of elasticity using UPV was also studied by other researchers. One of those studies was made by Qixian and Bungey in 1999 using the relationship between the dynamic modulus of elasticity, Poisson’s ratio and the velocity of surface and longitudinal waves. In their research, concrete specimens were made to permit measurement of dynamic modulus of elasticity and Poisson’s ratio using the proposed method and results were compared with values obtained by the conventional resonance method, and the experimental results showed that the proposed new method was reliable [Qixian and Bungey, 1999]. In 1998, the detection of freezing and thawing damage in concrete studied by Akhras using signal energy and ultrasonic wave velocity, and at the end of his study he showed that the ultrasonic wave velocity was not sensitive to early
damage in concrete caused by freezing and thawing; however it was sensitive to
damage caused at a later stage [Akhras, 1998].

2.2.3. Image Processing

Images play the single most important role in human perception, since vision is
the most advanced of our senses. Imaging machines cover almost the entire
electromagnetic spectrum ranging from gamma to radio waves, and can operate
also on images generated by sources that humans are not accustomed to
associating with images including ultrasound, electron microscopy and computer-
generated images. Therefore, digital image processing encompasses a wide and
varied field of applications like computer vision, face detection, feature
detection, lane departure warning system, medical image processing,
microscope image processing, morphological image processing, remote sensing,
and automated sieving procedures [Gonzalez etc., 2004].

An image may be defined as a two-dimensional function, \( f(x, y) \), where \( x \) and \( y \)
are “spatial coordinates”, and the amplitude of \( f \) at these coordinates is called
“the intensity or gray level” of the image at that point. If spatial coordinates and
the intensity are all finite, this image is called as a digital image which is
composed of a finite number of elements, each of which has a particular location
and value. Pixel is the term most widely used to denote these elements of a
digital image, and also they are known as picture elements, image elements and
pels. The field of digital image processing refers to processing digital images by
means of a digital computer. Although the first usage of the digital image
processing may be defined as the picture transmission using the submarine
cable between London and New York in the early 1920s, it is only in the late
1980s that the potential impact in civil engineering has been recognized
[Gonzalez et.al., 2004].
The processing of the digital images begins with transferring the images to the computer memory and this operation is called as the acquisition process. The acquisition can be performed by various types of the sensor formations, and the most encountered acquisition devices for engineering purposes are:

- **Scanners**: functional and cost effective devices, and can be used if the scanned objects are in two-dimensional (2D) form.
- **CCD (Charge-Coupled Device) Cameras**: an image sensor, consisting of an integrated circuit containing an array of linked, or coupled, light-sensitive sensors.
- **Electron Microscopes**: can be used if there is a need for higher optical magnification compared to the conventional microscopes, and their spatial resolutions may reach 1 nm for some instruments, but in general 4 nm is a common magnification level.
- **Magnetic Resonance Imaging (MRI) and X-Ray Computed Tomography (X-CT)**: can generate multiple two dimensional cross sections of the object, and by processing of the two dimensional sections, a detailed three dimensional reconstruction can be generated.

Regardless of how the image \( f(x, y) \) has been captured, it is arranged in such a manner that the resulting digital image has M rows and N columns, and in order to store this captured image in the computer memory, image coding techniques are used. In the case of a grayscale image, the brightness of each pixel is represented by a numeric integer value, and commonly stored with 8 bits per sampled pixel, which allows 256 different intensities, i.e. shades of gray, to be recorded ranging from 0 to 255. Binary representations assume that 0 is black and the maximum value is white not depending on what pixel depth is used. On the other hand a color image can be represented by 2D arrays of Red, Green and Blue layers, where 0 indicates that none of that primary color is present in that pixel and the maximum value indicates a maximum amount of that primary color. In the technical uses, more levels are required such as 10 to 12 bits per sampled
pixel to make full use of the sensor accuracy and to guard against roundoff errors in computations.

The data transferred through the image acquisition device to the storage media are controlled by the spatial and gray level resolutions (L) which are the most important features of any image. Spatial resolution is the minimum distance between two adjacent pixels or the minimum size of a pixel, and when it decreases, the image shows less detail as seen in Figure 2.7. Otherwise gray level resolution refers to the number of shades of gray in the image while the spatial resolution is kept constant (Figure 2.8) [Gonzalez etc., 2004; Önal, 2008].

![Figure 2.7. The Visual Appearance of Decrease in the Spatial Resolution [Önal, 2008]](image)
There are three main categories to group the image processing software packages. The first group is used for commercial general purposes, and it can be applied easily without any in depth of knowledge of the mathematical computations by selecting desired function in the software menu. The second group is the software that is bundled with the image acquisition system. The third group is the computer coded software via one of the several programming languages. There are several advantages of using a computing language for image analysis like that it has the ability to have direct access to any portion of the information available in the computing language in terms of ready-to-call image processing functions [Gonzalez etc., 2004]. In this thesis, the image processing and analysis operations have been coded in MATLAB Technical Computing Language.

2.2.3.1. Use of Image Analysis in Cement and Concrete Technology

In image analysis, the highly sufficient image contrast is so important, however in the case of concrete there are many problems in providing the necessary contrast. When using the polished sections of cement clinker, this problem is
now being overcome and the image analysis is being used as a semi-routine technique. Moreover, some aspects of ground surfaces of concrete such as air voids or cracks are imaged more successfully as contrast enhancement is easier. Some researchers like Chatterji and Gudmundsson (1977), Roberts and Scali (1984), Laurencot et al. (1992), Buckingham and Spaw (1988) and Challi et al. (1994) have proposed methods that the air voids are filled with contrast material and other methods such as filling the voids with fluorescent paint are now available [John et al., 1998; Marten et al., 1994]. In 2005, Carslon studied the determination of the air void system parameters of hardened concrete using images collected via a high-resolution flatbed scanner, and the developed approach provided a complete analysis including aggregate and paste fractions based upon image analysis techniques. In his study, the concrete slabs were cut and polished by a solution of acetone and fingernail hardener to stabilize the voids with the goal of obtaining the sharpest void edges possible. After obtaining polishing surface, thick hologram stickers were applied to the perimeter of the polished surface to protect the glass surface of the flat-bed scanners, and the image of the specimen surfaces were obtained by scanning at a pixel resolution of 8x8 microns. At the end of their study, they concluded that identifying the sample preparation as the key factor in obtaining quality results [Carlson, 2005]. In 2007, another research was performed to identify viable new methods for assessing air void systems in concrete by Sutter and Van Dam. In their study, the concrete surfaces were scanned in a total of three times by staining the surface with phenolphthalein to color the cement paste ink between the first and second scan, and by painting the surface black and pressing the white powder into depressions left by air voids between the second and third scan. The images collected from the three scans were aligned and input into a classification scheme to yield an output image in which each pixel is categorized as either air void, cement paste or aggregate. By digitally applying a grid of points and a series of lines to the output image, a modified point count method defined in the ASTM C 457 was performed, and a comparison was made between results obtained by an automatic analysis of the digital output image and results obtained by a manual analysis of the surface with an optical microscope. At the
end of the study they stated that the flatbed scanner method will allow engineers to more effectively monitor the quality of air void systems in concrete, leading to more durable pavement in challenging freeze-thaw environment [Sutter et al., 2007].
3.1. Introduction

This chapter targets the characterization of the air void system of concrete specimens by image analysis. Since it was aimed that developed algorithms would provide better characterization of air voids using digital image analysis techniques, a software tool was developed and coded using image processing toolbox of MATLAB Technical Computing Language. In order to calculate the spacing factor, which reflects the freeze-thaw durability, according to ASTM C 457 “Standard Test Method for Microscopical Determination of Parameters of the Air Void System in Hardened Concrete” the volume fraction of hardened cement paste is needed. The volume fraction of hardened cement paste was calculated by linear traverse method defined in that standard. The results of concrete specimens were compared with the results of traditional method obtained from laboratory in General Directorate of State Hydraulic Works of Turkey, which is accredited by TÜRKAK and has proven to have authority to perform this test.

3.2. Characterization of Air Voids by Image Processing

The first step in the characterization of air void parameters of concrete specimens is surface preparation. ASTM C 457 requires that the surface of the concrete specimen to be examined must be ground and polished to obtain an
acceptably smooth, plane surface for microscopical observation. However, that standard does not specify any particular preparation method or equipment; therefore, the preparation methods are often unique to individual laboratories.

On the other hand, in the method defined in TS EN 480-11, the surface preparation is specified and according to this specification, after curing of the concrete specimens at least 7 days, a sample with 100 mm wide, 150 mm in height and 20 mm thickness is cut in the middle of each sample using a diamond saw. One of the largest surfaces of each sample is first wet polished until to obtain a flat surface. After the initial polishing the surface, it should be fine polished and cleaned using water, compressed air or a suitable thin brush. The polishing and cleaning steps are followed by applying ink to the surface, and the samples are put into an oven at 50°C for 4h. The samples are removed from the oven after 4 h, and zinc oxide is spread over the surface to see the air voids clearly.

3.2.1. Analysis via ASTM C 457 Standard

In this analysis, the surfaces of the specimens were prepared by the method described in TS EN 480-11. The polished and inked surface was placed on a flatbed scanner and scanned in RGB color at a pixel resolution of 600 dpi and saved in a tagged image file format (TIFF). The RGB image was introduced to the MATLAB technical computer program, and cropped (Figure 3.1). According to the Image Processing Toolbox of MATLAB, four basic types of images are defined. **Binary Image**, also known as a bilevel image, is the logical array containing only 0s and 1s, interpreted as black and white, respectively. **Indexed Image**, also known as a pseudocolor image, consists of an array whose pixels are direct indices into a colormap. **Grayscale Image**, also known as an intensity, gray scale or gray level image, is a data matrix whose values represent intensities within some range. **Truecolor Image**, is also known as RGB Image, is an image in which each pixel is specified by three values-one each for the red, blue and green

Image segmentation subdivides an image into its constituent regions or objects, and the aim of segmentation is to simplify and/or change the representation of an image into something that is more meaningful and easier to analyze. There are few types of image segmentation like point, line and edge detection; thresholding; grayscaling; watershed, and color segmentation [Linda and George, 2001; Gonzalez et al., 2004]. In this thesis, grayscaling and thresholding techniques were used as the image segmentation.
3.2.1.1. Grayscale Segmentation for Air Void Determination

Cropped images in RGB colors were processed as multidimensional arrays. A transformation into grayscale images was made eliminating the hue and saturation information while retaining the luminance, reducing the image into an 8-bit intensity one with element values ranging 0 to 255 (Figure 3.2.). The algorithm for grayscale segmentation in this study can be seen in Appendix A-1. In order to make grain shapes more visible, the grayscale image was converted to a binary image following a contrast stretching transformation step. The transformation process consists of a piecewise linear function, which increases the dynamic range of the gray levels.

Figure 3.2. Grayscale Image

After grayscaling, the image was converted to the indexed image to obtain the entrapped and entrained air voids in the concrete (Figure 3.3). In this image,
entrained and entrapped air voids are seen as white, and the color of aggregate and paste is between the gray and black.

![Figure 3.3. Entrapped and Entrained Air Voids](image)

3.2.1.2. Thresholding for Aggregate/Paste Determination

Thresholding is the most widely used image segmentation methodology and resulted in a binary image, whose object pixels have one gray level and all background pixels have another. In this segmentation, the Otsu’s methodology, which chooses the threshold to minimize the interclass variance of the black and white, is used. This method is a histogram based thresholding method and the algorithm assumes that the image to be thresholded contains two classes of pixels then calculates the optimum threshold separating those two classes so that their combined spread is minimal [Gonzalez et al., 2004, MATLAB User’s Guide, 1993]. The process is summarized as
\[ g(x, y) = \begin{cases} 1 & \text{if } f(x, y) > T \\ 0 & \text{if } f(x, y) \leq T \end{cases} \] (3.1)

where \( g(x, y) \) is the value of the segmented image, \( f(x, y) \) is the gray level of the pixel \((x, y)\) and \( T \) is the threshold value. In this research, the threshold value was established in an automated manner according to the methodology suggested by Otsu where a threshold value to minimize the interclass variance of the black and white pixels is chosen.

In this study, both air content image and cropped image were thresholded to obtain the binary image of air content using the algorithm as seen in Appendix A-2 (Figure 3.4) and that of paste content. In Figure 3.4, air voids are seen as black, and the paste and aggregate are seen as white color. In Figure 3.5, the binary image of the original cropped image can be seen. Binary image of paste content was obtained by subtracting the air content image from the original cropped image as seen in Figure 3.6 in which black color shows the paste content.
Figure 3.4. Binary Image of Air Content

Figure 3.5. Thresholding of Original Cropped Image
3.2.1.3. Morphological Image Processing

During the scanning operation, the white spots, appearing within the coarse voids, probably result from non-uniform illumination or noise of various sources, and they were eliminated using a morphological operation. Morphology is a tool for extracting image components that are useful in the representation and description of region shape such as boundaries, skeletons, and the convex hull. Morphological operations apply a structuring element to an input image, creating an output image of the same size. In a morphological operation, the value of each pixel in the output image is based on a comparison of the corresponding pixel in the input image with its neighbors. In this thesis, two fundamental morphological operations, dilation and erosion, in terms of the union of an image with a translated shape were used as seen in Appendix A-II. Dilation adds pixels to the boundaries of objects in an image and grows or thickens objects in a binary image, while erosion removes pixels on object boundaries and shrinks or thins objects in a binary image. In both cases, the specific manner, and extent of
thickening or those of shrinking are controlled by a structuring element. Figure 3.7 illustrates how dilation and erosion work in a binary image.

![Figure 3.7. Illustration of Dilation and Erosion](Sun Microsystems web site, 2009)

**3.2.1.4. Image Information for Determining the Entrained Air Voids**

The image segmentation and morphological operation were followed by the characterization of the regions. Image analysis is extracting quantitative information from images and the image processing operations usually result in another image whereas the results of the image analysis algorithms are quantitative information. The sectional area, the centroid and the perimeter for
each void were defined for the problem of interest. In order to access the information obtained from binary images, a region map, where labels are assigned to pixels has to be generated and quantitative information about the objects could then be obtained examining their regional properties using various algorithms in the region map (Figure 3.8) [Gonzalez et al., 2004, MATLAB User’s Guide, 1993, Önal, 2008]. MATLAB image processing toolbox offers the measurement of a set of properties for each labeled region in the labeled matrix, and the available properties for each object in the labeled matrix are given in Table 3.1. In this thesis, the area, equivalent diameter and the perimeter of the voids were measured to distinguish the entrained and entrapped air voids as seen in Appendix A-III, and the voids whose equivalent diameters are smaller than 1 mm and which are close to a circle in shape, which is determined according to their equivalent perimeter and the voids whose equivalent perimeter is in the range of 1 to 1.2, were accepted as entrained air voids and Figure 3.9 shows the entrained air voids in the image. Furthermore, the diameters of the air voids, their locations and other geometric properties were determined.

![Figure 3.8. Binary Image, Binary Matrix, Label Matrix, and Labeled Matrix [Önal, 2008]](image-url)
| **Table 3.1. The Available Region Properties of the MATLAB’s Image Processing Toolbox** |
|---|---|---|
| **Area** | **Scalar** | The actual number of pixels in the region |
| **Bounding Box** | **Vector** | The smallest rectangle containing the region |
| **Centroid** | **Vector** | The center of mass of the region |
| **Convex Area** | **Matrix** | The number of pixels in convex image |
| **Convex Hull** | **Matrix** | The smallest convex polygon that can contain the region |
| **Convex Image** | **Binary Image** | The convex hull, with all pixels within the hull filled in |
| **Eccentricity** | **Scalar** | The eccentricity of the ellipse that has the same second-moments as the region |
| **Equivalent Diameter** | **Scalar** | The diameter of a circle with the same area as the region |
| **Euler Number** | **Scalar** | Equal to the number of objects in the region minus the number of holes in those objects |
| **Extent** | **Scalar** | The proportion of the pixels in the bounding box that are also in the region |
| **Extrema** | **Matrix** | The extrema point in the region |
| **Filled Area** | **Scalar** | The number of on pixels in filled image |
| **Filled Image** | **Binary Image** | Binary Image of the same size as the bounding box of the region |
| **Image** | **Binary Image** | Binary image of the same size as the bounding box of the region |
| **Major Axis Length** | **Scalar** | The length (in pixels) of the major axis of the ellipse that has the same normalized second central moments as the region |
| **Minor Axis Length** | **Scalar** | The length (in pixels) of the minor axis of the ellipse that has the same normalized second central moments as the region |
| **Orientation** | **Angle** | The angle (in degrees) between the x-axis and the major axis of the ellipse that has the same second-moments as the region |
| **Perimeter** | **Scalar** | The scalar containing the total number of pixels around the boundary of the region |
| **Pixel Idx List** | **Vector** | Vector containing the linear indices of the pixels in the region |
| **Pixel List** | **Matrix** | The actual pixels in the region |
| **Solidity** | **Scalar** | The proportion of the pixels in the convex hull that are also in the region |
3.2.1.5. Determination of Air Void Parameters

In ASTM C 457, two basic procedures are outlined to determine air void parameters of concrete as the linear traverse method and the point count method. Since the point count method is not practical for adapting to an image analysis procedure, in this study, the linear traverse method was chosen to determine the air void parameters. This method involves the examination of a cut and polished section of concrete which exposes the interior structure of the material. The linear traverse method is defined in the standard as the determination of the volumetric composition of the concrete by summing the distances traversed across a given component along a series of regularly spaced lines in one or more planes intersecting the sample. The data gathered are the total number of voids intersected (N), total length of traverse (T_t), traverse length through air (T_a) and traverse length through paste (T_p). After obtaining these data, air void characteristic parameters can be calculated. These parameters are [ASTM C 457]:

![Figure 3.9. Entrained Air Voids](image)
- **Air Content, A**: The proportion of the total volume of concrete that is air voids, 
\[
A = \frac{T_a \cdot 100}{T_t}
\]  
(3.2)

- **Void Frequency, n**: Voids per unit length of traverse
\[
\frac{n}{T_t} = \frac{N}{T_t}
\]  
(3.3)

- **Average Chord Length, \(\bar{l}\)**: The average length of the chords formed by the transaction of the voids by the line of traverse
\[
\bar{l} = \frac{T_a}{N}
\]  
(3.4)

- **Paste-Air Ratio, r**: The ratio of the volume of hardened cement paste to the volume of the air voids in the concrete
\[
r = \frac{p}{A} = \frac{T_p}{T_t}
\]  
(3.5)

- **Paste Content, p**: The proportion of the total volume of the concrete that is hardened cement paste
\[
p = \frac{T_p}{T_t}
\]  
(3.6)

- **Specific Surface, \(\propto\)**: The surface area of the air divided by their volume
\[
\propto = \frac{4N}{T_a}
\]  
(3.7)
• **Spacing Factor, \( \bar{L} \):** A parameter related to the maximum distance in the cement paste from the periphery of an air void.

\[
\bar{L} = \frac{T_p}{4N} \quad \text{when } p/A \leq 4.342
\]

\[
\bar{L} = \frac{3}{\alpha} \left[ 1.4 \left( 1 + \frac{p}{A} \right)^{1/3} - 1 \right] \quad \text{when } p/A > 4.342
\]  

(3.8)

Although the linear traverse method is based on summing the distances traversed across a given component along a series of regularly spaced lines in one or more planes intersecting the sample, in this thesis the air void parameters were calculated for the whole concrete surface area.

### 3.2.1.6. Analysis of Air Void Characteristics of Reference Concrete Material

The developed algorithm was controlled through six air entrained and two non air entrained specimens whose air void characteristics are known. These specimens were obtained from General Directorate of State Hydraulic Works in Turkey and air void characteristics were calculated utilizing optical microscope in that institution according to TS EN 480-11. Table 3.2 presents the air void characteristics analysis results of those specimens utilizing the developed algorithm developed according to the method defined in ASTM. In Figures 3.10 through 3.17, the aggregate, paste, total air voids and entrained air voids content for those specimens are given.
<table>
<thead>
<tr>
<th>Air Void Parameters</th>
<th>Specimen Number</th>
<th>with Air Entrainment (AE)</th>
<th>without AE</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td># 1</td>
<td># 2</td>
<td># 3</td>
</tr>
<tr>
<td>Aggregate Content (%)</td>
<td>57.09</td>
<td>49.71</td>
<td>49.44</td>
</tr>
<tr>
<td>Paste Content (%)</td>
<td>35.29</td>
<td>43.41</td>
<td>46.27</td>
</tr>
<tr>
<td>Air Content (%)</td>
<td>7.62</td>
<td>6.87</td>
<td>4.29</td>
</tr>
<tr>
<td>Number of Air Voids</td>
<td>5340</td>
<td>4775</td>
<td>7305</td>
</tr>
<tr>
<td>Void Frequency (mm⁻¹)</td>
<td>0.381</td>
<td>0.403</td>
<td>0.334</td>
</tr>
<tr>
<td>Specific Surface (mm⁻¹)</td>
<td>30.089</td>
<td>33.99</td>
<td>25.360</td>
</tr>
<tr>
<td>Paste/Air Ratio</td>
<td>3.94</td>
<td>4.06</td>
<td>3.6</td>
</tr>
<tr>
<td>Spacing Factor (mm)</td>
<td>0.131</td>
<td>0.119</td>
<td>0.141</td>
</tr>
</tbody>
</table>
Figure 3.10. Image Analysis of Specimen # 1 with Air Entrainment
Figure 3.11. Image Analysis of Specimen # 2 with Air Entrainment
Figure 3.12. Image Analysis of Specimen # 3 with Air Entrainment
Figure 3.13. Image Analysis of Specimen #4 with Air Entrainment
Figure 3.14. Image Analysis of Specimen # 5 with Air Entrainment
Figure 3.15. Image Analysis of Specimen # 6 with Air Entrainment
Figure 3.16. Image Analysis of Specimen #7 without Air Entrainment
Figure 3.17. Image Analysis of Specimen #8 without Air Entrainment
3.2.2. Analysis via TS EN 480 Standard

In this thesis, another code was developed according to TS EN 480 method. The algorithm of this code was similar to the previous code which is based on ASTM C 457 except for the determination of aggregate/paste ratio step. In this method, aggregate/paste ratio was obtained from the mix design as suggested by the standard and this ratio was introduced to the code. After introducing the aggregate/paste ratio, the aggregate content and paste content was computed from that ratio. The remaining parameters were computed using the same procedure in the ASTM method. The air void characteristics of the same six air entrained and two non air entrained specimens were also evaluated through this code and results are presented in Table 3.3.
Table 3.3. Analysis Results via TS EN 480-11

<table>
<thead>
<tr>
<th>Air Void Parameters</th>
<th>Specimen Number</th>
<th>Specimen Number</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>with Air Entrainment (AE)</td>
<td>without AE</td>
</tr>
<tr>
<td></td>
<td># 1</td>
<td># 2</td>
</tr>
<tr>
<td>Aggregate Content (%)</td>
<td>58.09</td>
<td>54.65</td>
</tr>
<tr>
<td>Paste Content (%)</td>
<td>37.20</td>
<td>40.34</td>
</tr>
<tr>
<td>Air Content (%)</td>
<td>4.71</td>
<td>5.01</td>
</tr>
<tr>
<td>Number of Air Voids</td>
<td>3317</td>
<td>4346</td>
</tr>
<tr>
<td>Void Frequency (mm⁻¹)</td>
<td>0.150</td>
<td>0.202</td>
</tr>
<tr>
<td>Specific Surface (mm⁻¹)</td>
<td>26.18</td>
<td>35.95</td>
</tr>
<tr>
<td>Paste/Air Ratio</td>
<td>7.83</td>
<td>8.05</td>
</tr>
<tr>
<td>Spacing Factor (mm)</td>
<td>0.217</td>
<td>0.160</td>
</tr>
</tbody>
</table>
3.2.3. Analysis via Traditional Microscopy

In the traditional method based on TS EN 480-11, the polished surface of the specimen is examined with a stereo microscope through 100±10x magnification. The method for microscopic examination in this standard is similar to the linear traverse method defined in ASTM C 457. In the examination, at least 4 lines are drawn to the surface and their length should be at least 2400 mm. The air voids are characterized on those lines. The data gathered are the total number of voids intersected (N), total length of traverse (Tt), traverse length through air (Ta) and traverse length through paste (Tp) same as ASTM C 457. However, in this method, paste content is determined from the mix design, and it is the total of volumetric ratios of cementitious material, water and admixtures. After obtaining these data, air void parameters such as air content, void frequency, average chord length, paste-air ratio, paste content, specific surface and spacing factor can be calculated. The microscopic determination results were obtained from the laboratory in the General Directorate of State Hydraulic Works of Turkey and those results are presented in Table 3.4.
| Air Void Parameters | Specimen Number | | | | | | | | | | with Air Entrainment (AE) | without AE | | | | | | | | # 1 | # 2 | # 3 | # 4 | # 5 | # 6 | # 7 | # 8 |
| Total Traverse Length (mm) | 2400 | 2400 | 1200 | 1200 | 1200 | 1200 | 2400 | 1200 |
| Traverse Length Through Paste (mm) | 2199.80 | 2234.70 | 1125.5 | 1116.7 | 1120.8 | 1135.9 | 2336.20 | 1178.60 |
| Traverse Length Through Air (mm) | 200.20 | 165.30 | 74.5 | 83.4 | 79.2 | 64.1 | 63.80 | 21.40 |
| Number of Air Voids | 1460 | 1589 | 638 | 697 | 657 | 534 | 212 | 64 |
| Average Chord Length (mm) | 0.137 | 0.104 | 0.117 | 0.12 | 0.121 | 0.120 | 0.301 | 0.334 |
| Void Frequency (mm⁻¹) | 0.608 | 0.662 | 0.532 | 0.581 | 0.548 | 0.445 | 0.088 | 0.053 |
| Specific Surface (mm⁻²) | 29.166 | 38.450 | 34.238 | 33.449 | 33.166 | 33.340 | 13.290 | 11.960 |
| Paste/Air Ratio | 3.4 | 4.20 | 4.8 | 4.3 | 4.5 | 5.6 | 16.80 | 16.10 |
| Spacing Factor (mm) | 0.115 | 0.109 | 0.133 | 0.128 | 0.133 | 0.146 | 0.494 | 0.655 |
3.3. Comparison of Results

The results from the developed codes based on ASTM C 457 and TS EN 480-11 were compared in Figure 3.18. In this figure, the comparison of the two most widely used air void parameters for freeze-thaw resistance - spacing factor and specific surface - are presented. As seen from those figures, if the mix includes the air entraining agent, the spacing factor is smaller than 0.2 mm, otherwise it is greater than 0.2 mm, and it can be concluded from the figure that the results of both analysis are assumed to be consistent.

On the other hand, the results from the traditional method obtained from microscopic examination were compared with the air void characteristics results of developed algorithm which is based on ASTM C 457. Comparing the results of the two most widely used air void parameters for freeze-thaw resistance, spacing factor and specific surface, in Figure 3.19, it can be concluded that even though there is more variability for those mixes that do not contain any air-entrainment, the results of both analysis are assumed to be consistent. It should not be forgotten that the analysis of the traditional method is only over a line of 2400 mm length, whereas the image analysis results are over the whole cross-section. Therefore, it is quite possible that for those mixes with no air entrainment the probability of crossing an air void for a single line is low.
Figure 3.18. Comparison of ASTM and TS EN Methods (by Image Analysis)
Figure 3.19. Comparison of ASTM and Traditional Methods

(a) Specific Surface

(b) Spacing Factor
CHAPTER 4

EXPERIMENTAL STUDY

4.1. Introduction

In order to evaluate the effectiveness of fly ash and air entraining admixtures on the freeze-thaw durability of self consolidating concrete, an experimental program was conducted. A total of ten self consolidating concrete mixtures with four different fly ash content (replaced by 0%, 15%, 30% and 45% of cement content by weight), and with three different air entraining agent content (0%, 0.15% and 0.3%) were prepared for that purpose. All concrete mixtures had the same superplasticizer content as 1.3% of cementitious materials by weight. During the casting operation, the workability properties of SCCs were observed through, slump flow time and diameter, air content, V-funnel flow time, L-box height ratio, and segregation ratio tests. Hardened properties were evaluated by compressive strength, permeability tests (water absorption, sorptivity and rapid chloride permeability test), freezing-thawing test, resonant frequency test, ultrasonic pulse velocity test, and image processing. Moreover, to determine the effect of curing conditions on the durability properties of self consolidating concrete, some specimens were cured in air and the others in water. Moist-cured (M-C) specimens were kept in water for 14 days at a temperature of 23±2°C before they were subjected to freeze-thaw cycles. Air-cured (A-C) specimens were however left in ambient laboratory conditions and were saturated in water for a day before they were subjected to the same freeze-thaw cycles.
4.2. Material Properties

This section will provide information on the chemical and physical properties of all ingredients used in this study. For determining the properties of materials, if available, ASTM (American Society for Testing and Materials) procedures were followed.

4.2.1. Portland Cement

An ordinary Portland cement CEM I 42.5R (PC) which corresponds to ASTM Type I cement was used throughout the tests. The chemical composition and physical properties of the cement are presented in Table 4.1. Moreover, the particle size distributions of PC, obtained by a laser scattering technique, is given in Figure 4.1.

4.2.2. Fly Ash

Throughout the study, a Class F type fly ash (FA) which is obtained from the Seyitömer power plant was used as pozzolanic material. The chemical composition and physical properties of FA are given in Table 4.1. and the particle size distributions of FA is presented in Figure 4.1.
Table 4.1. Chemical Composition and Physical Properties of Portland Cement and Fly Ash

<table>
<thead>
<tr>
<th>Chemical Composition</th>
<th>PC</th>
<th>FA</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO (%)</td>
<td>64.55</td>
<td>5.40</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>19.56</td>
<td>51.55</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>5.00</td>
<td>18.12</td>
</tr>
<tr>
<td>Fe₂O₃ (%)</td>
<td>3.68</td>
<td>11.85</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>1.57</td>
<td>4.63</td>
</tr>
<tr>
<td>SO₃ (%)</td>
<td>2.41</td>
<td>0.11</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>0.66</td>
<td>1.88</td>
</tr>
<tr>
<td>Na₂O (%)</td>
<td>0.77</td>
<td>5.65</td>
</tr>
<tr>
<td>Loss on Ignition (%)</td>
<td>2.89</td>
<td>1.34</td>
</tr>
</tbody>
</table>

Physical Properties

<table>
<thead>
<tr>
<th></th>
<th>PC</th>
<th>FA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific Gravity</td>
<td>3.09</td>
<td>2.18</td>
</tr>
<tr>
<td>Blaine Fineness (cm²/g)</td>
<td>3527</td>
<td>3138</td>
</tr>
</tbody>
</table>

Mechanical Properties

<table>
<thead>
<tr>
<th></th>
<th>PC</th>
<th>FA</th>
</tr>
</thead>
<tbody>
<tr>
<td>f_c', 2 days (kgf/cm²)</td>
<td>248</td>
<td>-</td>
</tr>
<tr>
<td>f_c', 7 days (kgf/cm²)</td>
<td>343</td>
<td>-</td>
</tr>
<tr>
<td>f_c', 28 days (kgf/cm²)</td>
<td>504</td>
<td>-</td>
</tr>
</tbody>
</table>

Figure 4.1. Particle Size Distributions of Portland Cement and Fly Ash
4.2.3. Aggregates

Crushed limestone was used as for the fine and coarse aggregate in this study. Two types of coarse aggregate named as Coarse 1 and Coarse 2 with a nominal size of 12 mm had specific gravity of 2.68 and water absorption of 0.42% and 0.52%, respectively. The fine aggregate had a specific gravity of 2.67 and a water absorption of 0.75%. Table 4.2. presents the grading of fine and coarse aggregates determined according to the ASTM C 136.

<table>
<thead>
<tr>
<th>Sieve Size (mm)</th>
<th>% Passing</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fine</td>
</tr>
<tr>
<td>19.1</td>
<td>100</td>
</tr>
<tr>
<td>12.7</td>
<td>100</td>
</tr>
<tr>
<td>9.50</td>
<td>100</td>
</tr>
<tr>
<td>4.75</td>
<td>99.8</td>
</tr>
<tr>
<td>2.36</td>
<td>73.8</td>
</tr>
<tr>
<td>1.18</td>
<td>46.1</td>
</tr>
<tr>
<td>0.60</td>
<td>29.5</td>
</tr>
<tr>
<td>0.30</td>
<td>19.7</td>
</tr>
<tr>
<td>0.15</td>
<td>14.6</td>
</tr>
</tbody>
</table>

4.2.4. Chemical Admixtures

In all concrete mixtures, a polycarboxylic-ether type superplasticizer with a specific gravity of 1.09, pH of 6.6 was used. In addition to the superplasticizer, an air entraining admixture, BASF Micro Air 200 based on fatty alcohol and ammonium salt with a specific gravity of 1.02, and pH of 10 was also used.
4.3. Experimental Procedures

4.3.1. Mixture Proportions

The concrete mixture proportions of the mixes are given in Table 4.3. As seen from the table, within the scope of the experimental program ten concrete mixtures were prepared. The control mixture included only the Portland cement as a binder, and named as AE0-FAO which means that there is no air entraining agent and fly ash. The remaining mixtures had different fly ash contents of 15%, 30% and 45% by weight of PC and named as FA15, FA30 and FA45, respectively. For all the mixtures, the total amount of cementitious material (PC+FA) and superplasticizer (SP) content (1.3% of cementitious materials by weight) were kept constant. However, the air entraining agent (AE) changed from 0.08% to 0.30%, and named as AE0.08, AE0.15 and AE0.30 in the mix design label, respectively. As the FA and air entraining admixture interaction affected the amount of air entrainment, three levels of air entrainment were considered (none, moderate and max) and the amount of air entraining admixture was determined accordingly. Since the SCC characteristics such as slump flow diameter, V-funnel time were expected to be obtained, water was gradually added to the mixtures and therefore the water to cementitious ratio (w/cm) was not kept constant and changed from 0.27 to 0.33. The mixes were classified according to their air entraining content as seen in Table 4.4 and also according to the fly ash content as seen in Table 4.5, and comparisons were performed according to these classification.
<table>
<thead>
<tr>
<th>Mix ID</th>
<th>Mix Design Label</th>
<th>W/CM</th>
<th>Water kg/m³</th>
<th>PC kg/m³</th>
<th>FA kg/m³</th>
<th>Aggregate</th>
<th>SP kg/m³</th>
<th>AE kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Fine kg/m³</td>
<td>Coarse 1 kg/m³</td>
<td>Coarse 2 kg/m³</td>
</tr>
<tr>
<td>1</td>
<td>AE0-FA0</td>
<td>0.33</td>
<td>163</td>
<td>500</td>
<td>0</td>
<td>1000</td>
<td>269</td>
<td>539</td>
</tr>
<tr>
<td>2</td>
<td>AE0-FA15</td>
<td>0.33</td>
<td>166</td>
<td>425</td>
<td>75</td>
<td>980</td>
<td>264</td>
<td>529</td>
</tr>
<tr>
<td>3</td>
<td>AE0-FA30</td>
<td>0.33</td>
<td>166</td>
<td>350</td>
<td>150</td>
<td>964</td>
<td>260</td>
<td>521</td>
</tr>
<tr>
<td>4</td>
<td>AE0-FA45</td>
<td>0.31</td>
<td>155</td>
<td>275</td>
<td>225</td>
<td>978</td>
<td>260</td>
<td>521</td>
</tr>
<tr>
<td>5</td>
<td>AE0.15-FA15</td>
<td>0.32</td>
<td>158</td>
<td>425</td>
<td>75</td>
<td>997</td>
<td>267</td>
<td>535</td>
</tr>
<tr>
<td>6</td>
<td>AE0.15-FA30</td>
<td>0.32</td>
<td>159</td>
<td>350</td>
<td>150</td>
<td>981</td>
<td>263</td>
<td>526</td>
</tr>
<tr>
<td>7</td>
<td>AE0.15-FA45</td>
<td>0.27</td>
<td>135</td>
<td>275</td>
<td>225</td>
<td>1014</td>
<td>268</td>
<td>548</td>
</tr>
<tr>
<td>8</td>
<td>AE0.3-FA45</td>
<td>0.27</td>
<td>135</td>
<td>275</td>
<td>225</td>
<td>1013</td>
<td>268</td>
<td>547</td>
</tr>
<tr>
<td>9</td>
<td>AE0.08-FA15</td>
<td>0.27</td>
<td>136</td>
<td>425</td>
<td>75</td>
<td>1046</td>
<td>276</td>
<td>560</td>
</tr>
<tr>
<td>10</td>
<td>AE0.3-FA30</td>
<td>0.27</td>
<td>137</td>
<td>350</td>
<td>150</td>
<td>1028</td>
<td>271</td>
<td>550</td>
</tr>
</tbody>
</table>
4.3.2. Specimen Preparation

Concrete mixtures were prepared using an electrically driven mechanical mixer with a 200 kg capacity. The preparation procedure was the same for all mixtures except for the elapsed time of the mixing, since the concrete including the air
entraining agents needs more mixing time. For all the concrete mixture, first of all, the fine and coarse aggregates were mixed with 1/3 of mixing water for 1 minute, and then, the cementitious materials (cement or cement and fly ash) were added and mixed for 1 more minute. The 1/3 of mixing water mixed with the superplasticizer or superplasticizer and air entraining agent, and added to the mixer. Eventually, remaining water was gradually added and sometimes not all of it was used.

After completing the mixing procedure, the air content, slump flow time and diameter, V-funnel flow time, L-box height ratio, and segregation ratio were determined for the fresh concrete properties. From each concrete mixture, twenty nine 100x200 mm cylinder specimens and one 15x15x15 cube specimen were cast without any compaction to determine the hardened properties like compressive strength, permeability tests, freezing-thawing test and ultrasonic pulse velocity test. After 24 hours, the specimens were removed from the molds, and sixteen cylinder specimens were cured in water at a temperature of 20±2°C and other thirteen specimens were cured in air until the date of testing.

4.4. Tests on Fresh Concrete

In this study, the air content and setting time tests of fresh concrete were determined according to the related ASTM standard test methods. Furthermore, workability properties of SCC mixtures were evaluated through the measurement of slump flow time ($T_{50}$) to reach a concrete 50 cm spread circle, slump flow diameter, V-funnel flow time, L-box height ratio and GTM sieve stability according to the methods standardized by Specification and Guidelines for SCC prepared by EFNARC (2005).
4.4.1. Slump Flow

Slump flow test provides information on filling ability (flowability) and passing ability for a stable mix. The test is performed similar to the conventional slump test defined in ASTM C 143 using the Abrams cone. However, instead of measuring the slumping distance vertically, the mean spread of the resulting fresh concrete is measured horizontally. In the test, the slump cone is completely filled with fresh concrete without consolidation, the cone is lifted, and the diameter of the spread concrete (D) is measured (Figure 4.2). According to EFNARC (2005), a slump flow diameter ranging from 650-800 mm can be accepted for SCC. During the slump flow test, the viscosity of the SCC mixture can be estimated by measuring the time ($T_{50}$) taken for the concrete to reach a spread diameter or 500 mm from the moment the slump cone is lifted up. $T_{50}$ measurement typically varies between 2 and 6 seconds for SCC according to EFNARC.

![Figure 4.2. Slump Flow Test](image)

4.4.2. V-funnel

The test is performed to assess the viscosity and filling ability of SCC. In the test, the V shaped funnel (Figure 4.3) is filled completely with a representative sample
of SCC without applying any compaction or rodding. The period between the time when the bottom gate is opened and the time that light can be seen through the orifice is recorded as V-funnel time ($t_v$). According to EFNARC (2005), $t_v$ ranging from 6 to 12 s is considered adequate for a SCC.

![V-funnel Apparatus](image)

**Figure 4.3. V-funnel Apparatus**

### 4.4.3. L-Box

This test method is developed to assess the passing ability and blocking resistance of SCC. It consists of vertical and horizontal section in the shape of “L” (Figure 4.4). After closing the vertical column of the L-box, concrete is filled in one lift without rodding. When the gate is lifted, SCC flows through the rebar obstructions and into the horizontal portion of the L-box. To assess the passing ability, the ratio between the concrete height at the end of the horizontal section, and the height of the remaining concrete in the vertical section has been proposed ($H_2/H_1$). EFNARC (2005) accepts this value in the range of 0.8 to 1.0.
4.4.4. GTM Sieve Stability

This test is a very effective way of assessing the stability of SCC. In this study, this test was performed by pouring approximately 6 liters of fresh concrete on to a 4.75 mm sieve, and the mass of mortar which passes through the sieve was weighed (Figure 4.5). The segregation ratio was taken as the ratio of mortar passing through the sieve to that weight of original sample on the sieve.

Figure 4.5. GTM Sieve Stability Test
4.4.5. Air Content

The air content of freshly mixed concrete was determined by pressure method described in ASTM C 231. In this study, the Type B meter of which operational principle consists of equalizing a known volume of air at a known pressure in a sealed air chamber with the unknown volume of air in the concrete sample was used.

4.5. Tests on Hardened Concrete

The hardened concrete properties determined at 14 days of age were compressive strength, absorption, sorptivity, rapid chloride permeability test, ultrasonic pulse velocity and resonant frequency. Later, the specimens were subjected to the freezing-thawing cycles and the resonant frequency was continuously monitored at every 30 cycles. Finally, by using the tool developed for image processing, the air void characteristics of concrete were determined.

4.5.1. Compressive Strength

The compressive strength test was performed according to the procedure defined in ASTM C 39 using a universal testing machine. A total of twenty one 100x150 mm concrete cylinders were tested for each mixture. The compressive strength was determined at 14 days, 28 days, after 150 cycles freezing-thawing and after the final cycle for both air curing and water curing specimens by using three cylinders at each age. In this test, the total maximum load indicated by the testing machine was recorded and the compressive strength values were calculated by dividing the total maximum load to the cross-sectional area.
4.5.2. Water Absorption

In this test, the absorption of the concrete expressed as the percentage of the absorbed water over the dry mass of concrete was determined following the ASTM C 642. In this test, two moist cured and two air cured (for 14 days) Ø100x50 mm disc specimens from each mixture were dried in an oven at 105±5°C to a constant weight. After removing each specimen from the oven, the specimens were immersed in water and weighed every 24 hours to check the increase in mass, until an increase in mass was less than 0.5% of the larger value of mass. At the end of this test, the total volume of permeable pores was determined, because water absorption can only take place in pores which were emptied during drying and filled with water during the immersion period.

4.5.3. Sorptivity

This test method is based on the ASTM C 1585 and is used to determine the rate of absorption of water by hydraulic cement concrete by measuring the increase in the mass of a disc specimen at given intervals of time (1, 5, 10, 20, 30, 60, 180, 240, 300 and 360 min) when only one surface of the specimen is exposed to water, with the depth of water between 3 to 5 mm. Two moist cured and two air cured (for 14 days) Ø100x50 mm disc specimens were first dried according to the procedure described in the absorption test and one side of each specimen was sealed by using a silicone coating. After measuring the increase in the mass of discs at given times, the rate of absorption (i in mm), defined as the change in mass (g) divided by the cross sectional area of the test specimen (mm²) and the density of water (g/mm³), was plotted against square root of time (t^{1/2} in min^{1/2}) as seen in Figure 4.6. The slope of the obtained line defines the sorptivity of the specimen during the initial six hours of testing.
4.5.4. Rapid Chloride Permeability (RCPT)

This test method is based on the ASTM C 1202 and was performed on two water cured and two air cured (for 14 days) Ø100x50 mm disc specimens from each mixture. In this test, the side of the specimens was coated with silicone coating, and after the silicone coating was dried, the specimens were vacuum saturated for 3 hours and allowed to soak for 18 hours. They were then placed in the test device. The left hand side (−) of the test cell was filled with a 3% NaCl solution and right hand side (+) of that was filled with 0.3N NaOH solution. The system was then connected and the specimens were subjected to a 60V applied DC voltage for 6 hours. The test equipment was equipped with automatic printer output at every 30 minutes and programmed to terminate the test automatically after 6 hours. At the end of 6h, the samples were removed from the cell and the total amount of coulombs passed through the specimen was calculated using the following equation.

\[
Q = 900(I_0 + 2I_{30} + 2I_{60} + \cdots + 2I_{300} + 2I_{330} + I_{360})
\]

(4.1)
where \( Q \) is charge passed (coulombs), \( I_0 \) is the current (Amperes) immediately after voltage is applied, and \( I_t \) is the current (Amperes) at \( t \) minutes after voltage is applied.

The total charge passed, in coulombs, is related to the ability of concrete to resist chloride ion penetration, and a high value for total charge passed indicates that the concrete is highly penetrable. In Table 4.6, typical raw data output from the rapid chloride test equipment, and in Table 4.7, the table based on ASTM C 1202 used to evaluate test results obtained from the equipment can be seen.

Table 4.6. Example of Raw Data Output from the RCPT Equipment

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Current (mA)</th>
<th>Charge (Coulombs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell #</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>94</td>
<td>99</td>
</tr>
<tr>
<td>30</td>
<td>101</td>
<td>108</td>
</tr>
<tr>
<td>60</td>
<td>104</td>
<td>109</td>
</tr>
<tr>
<td>90</td>
<td>111</td>
<td>114</td>
</tr>
<tr>
<td>120</td>
<td>116</td>
<td>118</td>
</tr>
<tr>
<td>150</td>
<td>123</td>
<td>123</td>
</tr>
<tr>
<td>180</td>
<td>128</td>
<td>127</td>
</tr>
<tr>
<td>210</td>
<td>132</td>
<td>131</td>
</tr>
<tr>
<td>240</td>
<td>136</td>
<td>134</td>
</tr>
<tr>
<td>270</td>
<td>140</td>
<td>137</td>
</tr>
<tr>
<td>300</td>
<td>143</td>
<td>138</td>
</tr>
<tr>
<td>330</td>
<td>145</td>
<td>140</td>
</tr>
<tr>
<td>360</td>
<td>147</td>
<td>141</td>
</tr>
</tbody>
</table>

Table 4.7. Chloride Ion Penetrability Based on Charge Passed

<table>
<thead>
<tr>
<th>Charge Passed (coulombs)</th>
<th>Chloride Ion Penetrability</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt; 4000</td>
<td>High</td>
</tr>
<tr>
<td>2000-4000</td>
<td>Moderate</td>
</tr>
<tr>
<td>1000-2000</td>
<td>Low</td>
</tr>
<tr>
<td>100-1000</td>
<td>Very Low</td>
</tr>
<tr>
<td>&lt; 100</td>
<td>Negligible</td>
</tr>
</tbody>
</table>
4.5.5. Freeze-Thaw Cycles

In this study, the SCC cylinder specimens were freezed and thawed in air and the temperature of the specimens was lowered from 4 to -18 °C and raised from -18 to 4 °C in 3 hours by using the climate test cabinet (Figure 4.7). Six water cured specimens and three air cured specimens from each mixture were used through the testing. Temperature variation inside the specimen was monitored by installing a thermocouple into the concrete (Figure 4.8). The time-temperature graph drawn for both climate data and thermocouple data is presented in Figure 4.9. As seen from that figure, the interior of concrete specimen reached freezing temperatures of -18°C in about 90 minutes and thawing temperature of 4°C in about 75 minutes. The concrete specimens were removed from the cabinet at each 30 cycle to determine the relative dynamic modulus of elasticity by using the resonant frequency method which is described in the next section. After determining the resonant frequency, the specimens were wetted by water and then replaced into the cabinet. After 150 cycles, three of the water cured specimens were removed from the cabinet and their compressive strength values were determined to see the loss of strength. The test was continued until the relative dynamic modulus of elasticity values of the specimens reached 60% of the initial modulus or 700 cycles whichever was reached first.
Figure 4.7. The Freezing-Thawing Test Cabinet

Figure 4.8. Monitoring the Temperature Variation Inside the Specimen
4.5.6. Resonant Frequency

In this study, to determine the resonant frequency of concrete specimens, a tool was developed following ASTM C 215. This test apparatus was developed by using the PTB 352A60 accelerometer with a sensitivity of 10mV/g and a frequency range of 5 to 60 kHz, and a NI USB 9233 data acquisition system with 24-Bit resolution and four simultaneously sampled analog inputs at up to 50 kS/s. In the test, the accelerometer was installed to the cylinder concrete specimen with silicone coating and attached to the data acquisition system as depicted in Figure 4.10. A small diameter steel ball was used as the impact source in the test. The ball was hit onto the top surface of a cylindrical specimen, and the accelerometer measured the vertical motion. The data were obtained by the LABVIEW computer program. According to this setup, the data were recorded from the data acquisition system and both the amplitude-time and amplitude-frequency graphs were obtained. From the amplitude-frequency graph, the peak value which shows the resonant frequency value of the concrete specimen was obtained (Figure 4.10).
Figure 4.10. Schematic Description of Resonant Frequency Test System
4.5.7. Ultrasonic Pulse Velocity

The UPV testing described in ASTM C 597 was performed on each freezing-thawing specimen before they were put into the climate test cabinet and at the end of the freezing-thawing test to see the variation in the modulus of elasticity. The testing system consisted of a pulser-receiver unit with a built-in data acquisition system and two transducers with 82 kHz central frequency. UPV measurements were conducted with the transducers firmly coupled to the opposite ends of the specimens using that is petroleum jelly as the couplant between the transducer and the specimen (Figure 4.11). UPV test gives the acquisition of the pulse arrival time which describes the elapsed time between the time of pulse application and arrival on the opposite face of the specimen. UPV was calculated by dividing the path length to the pulse arrival time.

Figure 4.11. Typical View of UPV Testing
5.1. Fresh Concrete Properties

In this thesis, the air content, unit weight and workability properties were evaluated through the fresh concrete. The workability properties of self consolidating concrete (SCC) were determined by slump flow time and diameter, V-funnel flow time, L-box height ratio and GTM sieve stability. The fresh concrete test results are given in Table 5.1.

The slump flow diameters of all mixtures were in the range of 66.5-72.5 cm, slump flow times were less 10s, the L-box height ratios were between the range of 0.22-1.00, and the segregation ratio as measured by the GTM sieve stability test was higher than 18%. Although all results were not in the range established by EFNARC except the slump flow time and diameter, all concrete mixtures filled the molds by its own weight without the need for vibration and the concrete mixtures were accepted as self-consolidating concrete. During the mixing procedure it was observed that the addition of air entraining agent increased the flowability of SCC mixes and so decreased V-funnel time, and also increasing fly ash content decreased the effect of air entraining agent. There are primarily two reasons for increasing flowability with increased air entraining agent. The first is that the entrained air increases the paste volume, which results in a more flowable concrete, and the second is that the additive itself influences the consistency of the cement paste.
Table 5.1. Fresh Concrete Test Results

<table>
<thead>
<tr>
<th>Mix #</th>
<th>Mix Design</th>
<th>V Funnel (s)</th>
<th>Slump Flow</th>
<th>Air Content (%)</th>
<th>L Box (h2/h1)</th>
<th>Unit Weight (g/cm³)</th>
<th>Segregation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>AE0-FA0</td>
<td>62</td>
<td>70.0</td>
<td>0.30</td>
<td>0.22</td>
<td>2.43</td>
<td>18.00</td>
</tr>
<tr>
<td>2</td>
<td>AE0-FA15</td>
<td>75</td>
<td>72.5</td>
<td>0.50</td>
<td>0.85</td>
<td>2.40</td>
<td>24.50</td>
</tr>
<tr>
<td>3</td>
<td>AE0-FA30</td>
<td>85</td>
<td>70.5</td>
<td>0.90</td>
<td>0.33</td>
<td>2.36</td>
<td>29.50</td>
</tr>
<tr>
<td>4</td>
<td>AE0-FA45</td>
<td>28</td>
<td>75.0</td>
<td>1.30</td>
<td>0.94</td>
<td>2.33</td>
<td>45.20</td>
</tr>
<tr>
<td>5</td>
<td>AE0.15-FA15</td>
<td>13</td>
<td>71.5</td>
<td>7.50</td>
<td>1.00</td>
<td>2.15</td>
<td>37.93</td>
</tr>
<tr>
<td>6</td>
<td>AE0.15-FA30</td>
<td>100</td>
<td>70.0</td>
<td>3.00</td>
<td>0.94</td>
<td>2.32</td>
<td>37.17</td>
</tr>
<tr>
<td>7</td>
<td>AE0.15-FA45</td>
<td>55</td>
<td>68.5</td>
<td>3.90</td>
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<td>2.32</td>
<td>30.24</td>
</tr>
<tr>
<td>8</td>
<td>AE0.3-FA45</td>
<td>26</td>
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<td>0.89</td>
<td>2.17</td>
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<td>AE0.08-FA15</td>
<td>80</td>
<td>69.0</td>
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<td>0.25</td>
<td>2.35</td>
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<td>7.00</td>
<td>0.88</td>
<td>2.15</td>
<td>26.70</td>
</tr>
</tbody>
</table>

5.2. Hardened Concrete Properties

The hardened properties of SCC mixes evaluated at 14 days of age were compressive strength, permeability tests, ultrasonic pulse velocity and resonant frequency, and the results are given in Table 5.2. As seen from the table, an increase in fly ash and air entraining agent content resulted in a reduced compressive strength. Moreover, increasing in the fly ash content resulted in a reduction in rapid chloride permeability and sorptivity index values.

Figure 5.1, 5.2 and 5.3 present the effect of fly ash content on the compressive strength, permeability properties and elastic properties, respectively. In these figures, y-axis shows the difference between moist cured and air cured specimens in percent. As seen from Figure 5.1, increase in the fly ash content resulted in an increased difference between compressive strength values of moist cured and air cured specimens which were also affected by the amount of air entraining agent. On the other hand, from Figure 5.2, it can be concluded that air cured specimens have higher permeability. However, in permeability results, there are more variations which can be mostly attributed to the test
procedures [Yaman, 2000]. Another cause for these variations could also be due to the number of specimens tested in the test program. In Figure 5.3, it can be seen that an increase in fly ash content raised the difference between UPV and resonant frequency values of air cured and moist cured specimens and this result is also compatible with the compressive strength results.
<table>
<thead>
<tr>
<th>Mix #</th>
<th>Mix Design</th>
<th>Curing Condition</th>
<th>Compressive Strength (kgf/cm²)</th>
<th>RCPT</th>
<th>Sorptivity Index (mm/min(^{1/2}))</th>
<th>Water Absorption (%)</th>
<th>UPV (m/s)</th>
<th>Resonant Frequency (Hz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>AE0-FA0</td>
<td>M-C</td>
<td>670 -</td>
<td>2741</td>
<td>0.0107</td>
<td>4.79</td>
<td>4902</td>
<td>10218</td>
</tr>
<tr>
<td></td>
<td>AE0-FA0</td>
<td>A-C</td>
<td>520 -</td>
<td>2864</td>
<td>0.0113</td>
<td>4.31</td>
<td>4884</td>
<td>9848</td>
</tr>
<tr>
<td>2</td>
<td>AE0-FA15</td>
<td>M-C</td>
<td>577 579</td>
<td>2714</td>
<td>0.0110</td>
<td>4.74</td>
<td>4923</td>
<td>9958</td>
</tr>
<tr>
<td></td>
<td>AE0-FA15</td>
<td>A-C</td>
<td>411 502</td>
<td>3328</td>
<td>0.0128</td>
<td>5.33</td>
<td>4727</td>
<td>9471</td>
</tr>
<tr>
<td>3</td>
<td>AE0-FA30</td>
<td>M-C</td>
<td>474 529</td>
<td>1934</td>
<td>0.0086</td>
<td>4.71</td>
<td>4888</td>
<td>9613</td>
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<tr>
<td></td>
<td>AE0-FA30</td>
<td>A-C</td>
<td>334 381</td>
<td>3615</td>
<td>0.0122</td>
<td>4.95</td>
<td>4555</td>
<td>9032</td>
</tr>
<tr>
<td>4</td>
<td>AE0-FA45</td>
<td>M-C</td>
<td>345 413</td>
<td>1916</td>
<td>0.0093</td>
<td>4.74</td>
<td>4706</td>
<td>9439</td>
</tr>
<tr>
<td></td>
<td>AE0-FA45</td>
<td>A-C</td>
<td>211 231</td>
<td>3752</td>
<td>0.0095</td>
<td>5.12</td>
<td>4547</td>
<td>8865</td>
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<tr>
<td>5</td>
<td>AE0.15-FA15</td>
<td>M-C</td>
<td>291 301</td>
<td>-</td>
<td>0.0156</td>
<td>6.19</td>
<td>4489</td>
<td>8857</td>
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<tr>
<td></td>
<td>AE0.15-FA15</td>
<td>A-C</td>
<td>163 162</td>
<td>-</td>
<td>0.0155</td>
<td>5.00</td>
<td>4259</td>
<td>8564</td>
</tr>
<tr>
<td>6</td>
<td>AE0.15-FA30</td>
<td>M-C</td>
<td>361 437</td>
<td>3179</td>
<td>0.0101</td>
<td>4.54</td>
<td>4722</td>
<td>9371</td>
</tr>
<tr>
<td></td>
<td>AE0.15-FA30</td>
<td>A-C</td>
<td>289 293</td>
<td>5142</td>
<td>0.0105</td>
<td>5.10</td>
<td>4459</td>
<td>8773</td>
</tr>
<tr>
<td>7</td>
<td>AE0.15-FA45</td>
<td>M-C</td>
<td>308 367</td>
<td>3392</td>
<td>0.0086</td>
<td>4.67</td>
<td>4691</td>
<td>9118</td>
</tr>
<tr>
<td></td>
<td>AE0.15-FA45</td>
<td>A-C</td>
<td>214 211</td>
<td>2648</td>
<td>0.0101</td>
<td>5.48</td>
<td>4301</td>
<td>8481</td>
</tr>
<tr>
<td>8</td>
<td>AE0.3-FA45</td>
<td>M-C</td>
<td>286 291</td>
<td>2565</td>
<td>0.0086</td>
<td>5.51</td>
<td>4613</td>
<td>8932</td>
</tr>
<tr>
<td></td>
<td>AE0.3-FA45</td>
<td>A-C</td>
<td>115 114</td>
<td>4496</td>
<td>0.0115</td>
<td>5.35</td>
<td>4309</td>
<td>8451</td>
</tr>
<tr>
<td>9</td>
<td>AE0.08-FA15</td>
<td>M-C</td>
<td>497 583</td>
<td>5237</td>
<td>0.0061</td>
<td>4.95</td>
<td>4752</td>
<td>9535</td>
</tr>
<tr>
<td></td>
<td>AE0.08-FA15</td>
<td>A-C</td>
<td>386 343</td>
<td>6007</td>
<td>0.0113</td>
<td>4.92</td>
<td>4669</td>
<td>9205</td>
</tr>
<tr>
<td>10</td>
<td>AE0.3-FA30</td>
<td>M-C</td>
<td>290 423</td>
<td>3762</td>
<td>0.0110</td>
<td>5.28</td>
<td>4479</td>
<td>8808</td>
</tr>
<tr>
<td></td>
<td>AE0.3-FA30</td>
<td>A-C</td>
<td>180 157</td>
<td>9437</td>
<td>0.0109</td>
<td>5.24</td>
<td>4244</td>
<td>8021</td>
</tr>
</tbody>
</table>
Figure 5.1. Relationship between Compressive Strength and Fly Ash Content
Figure 5.2. Relationship between Permeability and Fly Ash Content

(a) RCPT

(b) Sorptivity

(c) Water Absorption
Figure 5.3. Relationship between Elastic Properties and Fly Ash Content
5.3 Resistance to Freezing-Thawing

In this study, the resistance of SCC specimens to freezing-thawing was evaluated through traditional freezing-thawing test and also image processing used for the determination of air void characteristics.

Traditional freezing-thawing test depends on computing the durability factor obtained by measuring the resonant frequency values of the specimens from the amplitude-frequency graph. The accuracy of these frequency values were controlled via SAP 2000 software for structural analysis and design. In this analysis, first the moduli of elasticity values were estimated by ultrasonic pulse velocity test. For example for Mix 1, UPV was found as 4902 m/s from where modulus of elasticity can be calculated as 57.4 GPa. After inserting the modulus of elasticity values to the modal analysis program, concrete cylinder specimen was modeled as seen in Figure 5.4. Modal frequency of each mode was computed using this structural model and compared with the data obtained from resonant frequency test. Using modulus of elasticity and a typical poisson ratio of 0.20 modal analysis reveals a frequency of 10100 Hz for the following mode shape in Figure 5.4.

In the resonant frequency test, the specimen was impacted and data were collected by LABVIEW computer program, and amplitude-time graph was obtained by the program. By the amplitude-frequency graph through fast fourier analysis, the peak value which shows the resonant frequency value of the concrete specimen was obtained as 10250 Hz as seen in Figure 5.5. Therefore, at the end of this comparison, it was seen that the results were consistent and it was concluded that the developed resonant frequency testing apparatus was working properly.
Figure 5.4. Modal Analysis of the Concrete Specimen

(a) Undeformed Shape

(b) 10th Modal Shape
Figure 5.5. Resonant Frequency Determination
The effect of freezing-thawing is commonly assessed on the basis of the change in the dynamic modulus of elasticity. The dynamic methods employ ultrasonic velocity and mechanical resonant frequency tests. In this study, resonant frequency test was employed to determine the dynamic modulus of elasticity values of SCC cylinder specimens. The dynamic modulus of elasticity of each concrete specimen was calculated at initial condition, and for each 30 freezing-thawing cycles. In Figures 5.6 and 5.7, resonant frequency values of each mix for both moist cured and air cured specimens for each 30 freezing-thawing cycles are given, respectively.
Figure 5.6. Resonant Frequency Test Results for Moist Cured Specimens
Figure 5.7. Resonant Frequency Test Results for Air Cured Specimens
As seen from Figure 5.6 and 5.7 for those mixes that do not contain any air-entrainment the initial resonant frequency values are higher. As the air-entrainment level is increased there is a reduction in the initial resonant frequency of the concrete simply because of the increase in the porosity leading to a decrease in the modulus of elasticity. Moreover, except for the mix those do not contain any air entrainment and maximum amount of fly ash, most of the mixes are resistant to freezing and thawing cycles.

The durability factor was computed using the resonant frequency values of the specimens according to the following equation proposed by ASTM C 666 [ASTM C 666].

\[
DF = \frac{P_N}{M}
\]  

where \(DF\) is the durability factor, \(P\) is relative dynamic modulus of elasticity at \(N\) cycles in %, and \(M\) is specified number of cycles at which the exposure is to be terminated and in this study, \(M\) was chosen as 700 since freezing-thawing test was conducted to 700 cycles. In Table 5.3, durability factors of each mix are presented.
Table 5.3. Durability Factor

<table>
<thead>
<tr>
<th>Mix #</th>
<th>Mix Design</th>
<th>Curing</th>
<th>DF&lt;sub&gt;700&lt;/sub&gt;</th>
</tr>
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<tbody>
<tr>
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<td>AE0-FA0</td>
<td>M-C</td>
<td>100.6</td>
</tr>
<tr>
<td></td>
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<td>A-C</td>
<td>107.4</td>
</tr>
<tr>
<td>2</td>
<td>AE0-FA15</td>
<td>M-C</td>
<td>101.7</td>
</tr>
<tr>
<td></td>
<td>AE0-FA15</td>
<td>A-C</td>
<td>107.6</td>
</tr>
<tr>
<td>3</td>
<td>AE0-FA30</td>
<td>M-C</td>
<td>102.4</td>
</tr>
<tr>
<td></td>
<td>AE0-FA30</td>
<td>A-C</td>
<td>110.0</td>
</tr>
<tr>
<td>4</td>
<td>AE0-FA45</td>
<td>M-C</td>
<td>63.0</td>
</tr>
<tr>
<td></td>
<td>AE0-FA45</td>
<td>A-C</td>
<td>100.8</td>
</tr>
<tr>
<td>5</td>
<td>AE0.15-FA15</td>
<td>M-C</td>
<td>93.1</td>
</tr>
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<td>AE0.15-FA15</td>
<td>A-C</td>
<td>104.4</td>
</tr>
<tr>
<td>6</td>
<td>AE0.15-FA30</td>
<td>M-C</td>
<td>98.0</td>
</tr>
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<td>A-C</td>
<td>106.7</td>
</tr>
<tr>
<td>7</td>
<td>AE0.15-FA45</td>
<td>M-C</td>
<td>101.4</td>
</tr>
<tr>
<td></td>
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<td>A-C</td>
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<td>M-C</td>
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<td>A-C</td>
<td>112.3</td>
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<td>9</td>
<td>AE0.08-FA15</td>
<td>M-C</td>
<td>100.7</td>
</tr>
<tr>
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<td>A-C</td>
<td>105.3</td>
</tr>
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<td>M-C</td>
<td>100.7</td>
</tr>
<tr>
<td></td>
<td>AE0.3-FA30</td>
<td>A-C</td>
<td>110.0</td>
</tr>
</tbody>
</table>

The relationship between durability factor and fly ash and air entraining agent content was also evaluated and it is given in Figure 5.8 (a) and (b), respectively. As seen from the figure, the highest degree of reduction in durability factor was observed at mixes including maximum fly ash content, and no air entraining agent and maximum air entraining agent content. Furthermore, when fly ash was used in the range of certain limits, it was observed that SCC specimens were resistant to the freezing-thawing.
Figure 5.8. Relationship between Durability Factor and Fly Ash and Air Entraining Agent Content
5.4. Air Void Characteristics Obtained by Image Processing

Air void characteristics were determined using image processing based on linear traverse method defined in ASTM C 457. According to this standard, for durable concrete against freezing-thawing, the specific surface (\(\alpha\)) is usually in the range 24 to 43 mm\(^{-1}\), and the spacing factor (\(\bar{L}\)) is usually in the range 0.1 to 0.2 mm [ASTM C 457].

In this study, firstly, the concrete specimens were sawed into three Ø100x25-mm disc specimens with a water-cooled diamond saw, and for analysis two moist cured and two air cured disc specimens from each mixes were used. Then the specimen surface to be observed follows several grinding steps in order to remove the marks of the saw blade and any damage to the surface that might have been introduced during the cutting process, and to obtain a smooth flat surface. After cutting the specimens, they were first ground with silicon carbide of 220 grits and then 600 grits. Grit size is the nominal size of abrasive particles corresponding to the number of openings per inch in a screen through which the particles can just pass. After the 600 grit grinding, the surface of the specimens were polished with felt including aluminum oxide (Al\(_2\)O\(_3\)) of 1500 grits. Polishing residues were removed from the specimen surface between each step and after the final step by gently blowing on the surface with an air compression and rinsing with water. The grinding and polishing of concrete surfaces were performed in General Directorate of Mineral Research and Exploration.

After grinding and polishing the surface, a blue board marker ink was used to color the surface blue. A series of slightly overlapping parallel lines were used to color the surface and second coat was applied at an orientation of 90° to the first coat, and dried in a 50°C oven for 4 hrs. After the ink was dried, zinc oxide was spread onto the polished surface, and worked into the voids with the flat face of a glass slide. Next, most of the excess paste was scraped away with a cloth. The remaining powder was wiped away with a liquid paraffin, leaving only the powder that had been worked into the recesses or voids.
For air void characteristics determination, the inked surface of SCC specimens were scanned and obtained pictures were introduced to the developed tool. However, the results according to ASTM method could not be obtained due to the employed board marker ink type and/or because of the aggregate type, as the aggregates could not be distinguished from the paste as shown in the pictures provided in Appendix B. Therefore, the results are obtained following the TS EN 480-11 standard, in which the aggregate/paste ratio is obtained from the mix design as presented in Table 5.4.
### Table 5.4. Air Void Characteristics

<table>
<thead>
<tr>
<th></th>
<th>Mix #</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>6</td>
<td>7</td>
<td>9</td>
<td>5</td>
</tr>
<tr>
<td>Mix Design</td>
<td></td>
<td>AE0-FA0</td>
<td>AE0-FA15</td>
<td>AE0-FA30</td>
<td>AE0-FA45</td>
<td>AE0.15-FA30</td>
<td>AE0.15-FA45</td>
<td>AE0.08-FA15</td>
<td>AE0.15-FA15</td>
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<tr>
<td>Aggregate/Paste Ratio</td>
<td>2.03</td>
<td>1.93</td>
<td>1.84</td>
<td>1.86</td>
<td>1.9</td>
<td>2.05</td>
<td>2.24</td>
<td>1.98</td>
<td>2.04</td>
</tr>
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<td>Aggregate Content (%)</td>
<td>66.1</td>
<td>64.27</td>
<td>63.65</td>
<td>64.49</td>
<td>64.82</td>
<td>65.97</td>
<td>67.53</td>
<td>64.5</td>
<td>65.72</td>
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<tr>
<td>Paste Content (%)</td>
<td>32.53</td>
<td>33.3</td>
<td>34.59</td>
<td>34.67</td>
<td>33.44</td>
<td>32.18</td>
<td>32.22</td>
<td>33.59</td>
<td>32.22</td>
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<tr>
<td>Air Content (%)</td>
<td>1.37</td>
<td>2.43</td>
<td>1.76</td>
<td>0.84</td>
<td>1.74</td>
<td>1.86</td>
<td>2.05</td>
<td>1.98</td>
<td>2.07</td>
</tr>
<tr>
<td>Void Frequency (mm-1)</td>
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<td>0.15</td>
<td>0.09</td>
<td>0.03</td>
<td>0.07</td>
<td>0.12</td>
<td>0.14</td>
<td>0.09</td>
<td>0.06</td>
</tr>
<tr>
<td>Specific Surface (mm-1)</td>
<td>29.92</td>
<td>30.79</td>
<td>27.26</td>
<td>21.04</td>
<td>23.61</td>
<td>30.06</td>
<td>29.52</td>
<td>20.63</td>
<td>20.5</td>
</tr>
<tr>
<td>Paste-Air Ratio</td>
<td>23.77</td>
<td>13.72</td>
<td>19.75</td>
<td>44.86</td>
<td>19.25</td>
<td>17.4</td>
<td>12.98</td>
<td>11.39</td>
<td>15.6</td>
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<td>Spacing Factor (mm)</td>
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<td>0.24</td>
<td>0.31</td>
<td>0.56</td>
<td>0.36</td>
<td>0.27</td>
<td>0.24</td>
<td>0.32</td>
<td>0.4</td>
</tr>
</tbody>
</table>
5.5. Air Void Characteristics vs. Freeze-Thaw Resistance

In Table 5.5, specific surface, spacing factor and number of voids calculated for both entrained air voids and total air voids, and also durability factor of each mix are presented for comparison. As seen from the table, specific surface values are between 24 to 28.9 mm$^{-1}$ for durable concrete against freezing-thawing, and are compatible with durability factor results. Although ASTM standard recommends that frost resistant concrete should have a calculated spacing factor less than 0.2 mm, there are few studies that this value is not suitable for all concrete types. One of those studies was reported by Khayat (2003) who concluded that depending on the type of cement, type and concentration of admixture in use, water-cement ratio, rate of freezing, etc., concrete with spacing factor larger than 0.2 mm can exhibit good frost resistance. At the end of this thesis, it was also concluded that the range of 0.1 to 0.2 mm for spacing factor should not be restricted for SCC since the concrete with spacing factor smaller than 0.4 mm could still exhibit good freezing-thawing resistance. In SCC, when spacing factor is approximately 0.4 mm, fly ash content and strength value should also be taken into account.
### Table 5.5. Air Void Characteristics and Durability Factor

<table>
<thead>
<tr>
<th>Mix #</th>
<th>Mix Design</th>
<th>Entrained Air Voids</th>
<th>Total Air Voids</th>
<th>Durability Factor</th>
</tr>
</thead>
<tbody>
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<td></td>
<td></td>
<td>Specific Surface</td>
<td>Spacing Factor</td>
<td>Void Frequency</td>
</tr>
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<td>AE0-FA0</td>
<td>29.92</td>
<td>0.31</td>
<td>0.09</td>
</tr>
<tr>
<td>2</td>
<td>AE0-FA15</td>
<td>30.79</td>
<td>0.24</td>
<td>0.15</td>
</tr>
<tr>
<td>3</td>
<td>AE0-FA30</td>
<td>27.26</td>
<td>0.31</td>
<td>0.09</td>
</tr>
<tr>
<td>4</td>
<td>AE0-FA45</td>
<td>21.04</td>
<td>0.56</td>
<td>0.03</td>
</tr>
<tr>
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<td>AE0.15-FA15</td>
<td>20.63</td>
<td>0.32</td>
<td>0.09</td>
</tr>
<tr>
<td>6</td>
<td>AE0.15-FA30</td>
<td>23.61</td>
<td>0.36</td>
<td>0.07</td>
</tr>
<tr>
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<td>AE0.15-FA45</td>
<td>30.06</td>
<td>0.27</td>
<td>0.12</td>
</tr>
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<td>AE0.3-FA45</td>
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<td>0.4</td>
<td>0.06</td>
</tr>
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<td>AE0.08-FA15</td>
<td>29.52</td>
<td>0.24</td>
<td>0.14</td>
</tr>
<tr>
<td>10</td>
<td>AE0.3-FA30</td>
<td>18.41</td>
<td>0.34</td>
<td>0.09</td>
</tr>
</tbody>
</table>
From Table 5.5, mixes were classified according to their fly ash content and the relationship between the durability factor and specific surface, spacing factor and void frequency was investigated as seen in Figure 5.9 (a), (b) and (c), respectively. From those figures, it can be concluded that when fly ash was used in the range of certain limits, SCC specimens were resistant to freezing-thawing and there is a very good correlation between the durability factor and air void characteristics.
Figure 5.9. Relationship between Durability Factor and Air Void Characteristics
6.1. Conclusions

In this thesis, a software tool was developed to determine the air void parameters of concrete based on image analysis and two different standards, ASTM C 457 and TS EN 480-11. After that, an experimental program was conducted to investigate the air void characteristics of self consolidating concrete. The effectiveness of fly ash and air entraining admixtures in the freeze-thaw resistance of self consolidating concrete was evaluated and for this purpose, durability factor of each mix was computed. To determine the durability factor, resonant frequency values were also computed by an apparatus which was also developed within the scope of this thesis. Furthermore, the effect of curing conditions on the durability of self consolidating concrete was also aimed to be evaluated. However, this was not able to be accomplished due to the unavailability of a freeze-thaw cycle cabinet that allows freezing and thawing of specimens in water.

The following conclusions are drawn as a result of this experimental program:

- As for the fresh properties of SCC mixes, it can be concluded that both fly ash and air entraining agent content have an effect on the workability properties of self consolidating concrete. During mixing it was observed that the addition of air entraining agent increased the flowability and decreased V-funnel time of SCC mixes due to the workability
enhancement of the small air-bubbles formed by the chemical agent. Moreover, an increase in the fly ash content decreased the effect of air entraining agent due to the unburned carbon contained in the fly ash since the unburned carbon absorbs a portion of the air entraining agent, which limits its ability for producing the needed stable air bubbles.

- When the hardened properties of the SCC was considered, it was observed that addition of both fly ash and air entraining agent content resulted in a reduction in the 14th day compressive strengths.

- The difference between the compressive strength, UPV and resonant frequency values of moist cured and air cured specimens increased with increasing fly ash and air entraining agent content. Therefore, special attention should be paid to cure the concrete mixes incorporating higher amounts of fly ash and air entraining agent.

- From the permeability tests, it was concluded that air cured specimens have higher permeability. Furthermore, an increase in fly ash content resulted in a reduction in the permeation properties of self consolidating concrete. On the other hand, more variations were observed in permeability results since only two specimens were used for permeability tests.

- During the freezing-thawing test, it was observed that air cured specimens were not affected by freezing-thawing and did not indicate any degradation since they were not totally saturated because of the lack of the saturation period that was employed.

- The highest degree of reduction in durability factor was observed for mixes including maximum fly ash content and no air entraining agent because of the lack of a sufficient air void system and a weak microstructure before the start of freeze-thaw cycles. Additionally, when
fly ash was used in the range of certain limits, it was observed that SCC specimens were resistant to the freezing-thawing cycles because of the lowered permeability properties.

- In the air void characteristics determination, it was concluded that spacing factor - which is the most important air void characteristic that is utilized for determination of the resistance to freezing-thawing - should not be restricted to 0.2 mm for SCC, since SCCs with spacing factors smaller than 0.4 mm could still exhibit good freeze-thaw resistance. In SCC, when spacing factor is approximately 0.4 mm, fly ash content and the initial strength of the concrete should be taken into account to assess its performance against freeze-thaw cycles.

6.2. Recommendations

The effect of curing conditions on the freezing-thawing resistance of SCC should be further studied utilizing the method, freezing-thawing in water, defined in ASTM. Moreover, since surface preparation procedure’s aforementioned importance on the quality of analysis, more study is required for surface preparation method. On the other hand, in this thesis, a limited number of specimens was used for permeability tests whereas these tests should be utilized by using lots of specimens.
REFERENCES


ACI Committee Report 308; *Guide to Curing Concrete*; ACI, USA; (2001).


Akhras, N.M.; *Detecting freezing and thawing damage in concrete using signal energy*; Cement and Concrete Research, Vol. 28, Issue 9, pp. 1275-1280; (Sep., 1998).


ASTM C 231; Standard Test Method for Air Content Freshly Mixed Concrete by the Pressure Method; Annual Book of ASTM Standards; (2002).

ASTM C 403; Standard Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance; Annual Book of ASTM Standards; (2002).

ASTM C 597; Standard Test Method for Pulse Velocity Through Concrete; Annual Book of ASTM Standards; (2002).


ASTM C 666; Standard test Method for Resistance of Concrete to Rapid Freezing and Thawing; Annual Book of ASTM Standards; (2002).

ASTM C 1202; Standard test Method for Electrical Indication of Concrete’s Ability to Ion Penetration; Annual Book of ASTM Standards; (2002).


Blick, R.L., Petersen, C.F. and Winter, M.E.; *Proportioning and Controlling High-Strength Concrete*; ACI, USA, SP-46, pp. 141-163; (1974).


Blitz, J.; *Ultrasonics: Methods and Applications*; Van Nonstrand Reinhold Company, New York, USA; (1971).

Brown, A.E.; *Rationale and summary of methods for determining ultrasonic properties of materials at Lawrence Livermore National Laboratory*; UCRL-ID-119958, Lawrence Livermore National Laboratory, California, USA; (1997).

Carino, N.J. and Meeks, K.W.; *Curing of High performance Concrete for Strength: What is Sufficient?*; Repair, Rehabilitation and Maintenance of Concrete Structures and Innovations in Design and Construction, ACI SP-193, The 4th International Conference, Korea; (Sep., 2000).


Davis, R.E.; *Pozzolanic Materials with Special Reference to their use in Concrete Pipe*; Technical Memo., American Concrete Pipe Assoc., Texas, USA; (1954).


EFNARC; *Specification & Guidelines for Self-Consolidating Concrete*; European Federation for Specialist Construction Chemicals and Concrete Systems, Norfolk, UK; (Feb., 2002).

Erdogan, T.Y.; *Beton*; METU Press, Ankara, Turkey; (May, 2003).


John, D.A., Poole, A.W. and Sims, I.; *Concrete Petrography*; John Wiley and Sons Inc, New York, USA; (1998).


Kaufmann, J.P.; *Experimental Identification of Ice Formation in Small Concrete Pores*; Cement and Concrete Research, Vol. 34, pp. 1421-1427; (Jan., 2004).


Kodama, Y.; *Current Condition of Self-Compacting Concrete*; Cement Shimbun, Tokyo, Japan; (Dec., 1997).
Lachemi, M. and Bouzoubaa, N.; *Self-Compacting Concrete Incorporating high volumes of class F fly ash preliminary results*; Cement and Concrete Research, Vol. 31, pp. 413-420; (Dec., 2001).


Lee, S.; *Petrographic Evaluation of Bridge Deck Concrete Durability*; Ph.D. Thesis, Graduate Faculty of Texas Tech University; (Aug., 2001).


Malhotra, V.M.; *In Situ/Nondestructive Testing of Concrete*; American Concrete Institute, Detroit, USA; (1984).


Mehta, P. K. and Monteiro, P.J.M.; *Concrete: Microstructure, properties, and materials*; Indian Ed., Indian Concrete Institute, Chennai, India; (1997).
Meyer, A.; *Experiences with the Use of Superplasticizers in Germany*; ACI SP-62, USA, pp. 21-36; (1981).

Okamura, H. and Ouchi M.; *Self Compacting Concrete*; Journal of Advanced Technology, Concrete Institute, Osaka, Japan, Vol. 1, pp. 5-15; (Apr., 2003).


Özyıldırım, Ç., and Halstead, W.; *Resistance to chloride ion Penetration of concrete containing fly ash, silica fume, or slag*; ACI SP-108, Permeability of Concrete, Concrete Inst., Detroit, Michigan, USA, pp. 35-62; (1988).


Şahmaran, M.; *Self-Compacting Concrete with High Volumes of Fly Ash*; PhD. Thesis, Department of Civil Engineering, METU, Ankara, Turkey; (Jan., 2006).


Sutter, L. and Van Dam, T.; *Scanners Takes Air Void Analysis Out From Under Microscope*; Wisconsin Highway Research program, Wisconsin, USA; (Sep., 2007).


Tucker, G.R.; *Concrete and Hydraulic Cement*; United States Patent No. 2141569; (1932).

Yaman, İ.Ö.; *Finite Element Simulation of Wave Propagation in Concrete for the Evaluation of Ultrasonic Testing Procedures*; Ph.D. Thesis, Department of Civil and Environmental Engineering Wayne State University, Detroit, USA, (2000)

APPENDIX A

PART I - Converting an RGB image to grayscale

%% Converting an RGB image to grayscale
GrayScaleImage = rgb2gray(CroppedImage); % Converting RGB image to grayscale
figure, imshow(GrayScaleImage), title('GrayScale Image'); % Show grayscale image as a new figure
clear OriginalImage % Clear original image from memory
clear CroppedImage % Clear cropped image from memory
% Adjusting image intensity values
AdjustedGrayScaleImage = imadjust(GrayScaleImage, stretchlim(GrayScaleImage), [0 1]); % Adjusting image intensity values by finding limits to contrast stretch the grayscale image.
figure, imshow(AdjustedGrayScaleImage), title('Adjusted GrayScale Image'); % Show adjusted grayscale image
clear GrayScaleImage % Clear grayscale image
PART II - Thresholding of an Image

%% Thresholding Air Content Image
level = graythresh(NewAir);  %Compute global image threshold using Otsu's method
AirContentBinaryImage = im2bw(NewAir,level);  %Converting adjusted grayscale image to a binary image, based on threshold
AirContentBinaryImage=~AirContentBinaryImage;
figure, imshow(AirContentBinaryImage), title('Air Content Binary Image');  %Show binary image
clear AirContent  %Clear adjusted grayscale image
se90 = strel('line',1,90);
se0 = strel('line',1,0);
BWsdilAir = imdilate(AirContentBinaryImage, [se90 se0]);
figure, imshow(BWsdilAir), title('Dilated Gradient Mask in Air Content Image')
FilledAirBinaryImage = imfill(~BWsdilAir,'holes');
figure, imshow(FilledAirBinaryImage), title('Air Content Binary Image with Filled Holes');
seD = strel('line',1,0);
BWfinalAir = imerode(FilledAirBinaryImage,seD);
SegmentedAirBinaryImage = imerode(BWfinalAir,seD);
AirBinaryImage=SegmentedAirBinaryImage;
figure, imshow(AirBinaryImage), title('Air Content Segmented Binary Image');

%% Thresholding the Original Image
level2 = graythresh(AdjustedGrayScaleImage);  %Compute global image threshold using Otsu's method
BinaryImageOrigin = im2bw(AdjustedGrayScaleImage,0.35);  %Converting adjusted grayscale image to a binary image, based on threshold
figure, imshow(BinaryImageOrigin), title('Original Binary Image');  %Show binary image
clear AdjustedGrayScaleImage  %Clear adjusted grayscale image
se90 = strel('line',1,90);
se0 = strel('line',1,0);
BWsdl = imdilate(BinaryImageOrigin, [se90 se0]);
figure, imshow(BWsdl), title('Dilated Gradient Mask in Original Image')
FilledBinaryImage = imfill(BWsdl,'holes');
figure, imshow(FilledBinaryImage), title('Original Binary Image with Filled Holes');
seD = strel('line',1,0);
BWfinal = imerode(FilledBinaryImage,seD);
SegmentedBinaryImage = imerode(BWfinal,seD);
BinaryImage=SegmentedBinaryImage;
figure, imshow(BinaryImage), title('Original Segmented Binary Image');
PART III - Determination of the Entrained Voids

%% Determination of the Entrained Voids
VoidArea=0; %Variable set to zero
mmMaximumArea=(0.5/(mmPixelSize))^2*pi(); %Specifies the maximum
area value depending on the standards
mmMinimumArea=(0.001/(mmPixelSize))^2*pi(); %Specifies the minimum
area value depending on the standards
for i=1:numObjects %Adding areas of all objects
    VoidArea=voiddata(i).Area; %Specifies the area of the void
    Diameter=voiddata(i).EquivDiameter; %Specifies the diameter of a
circle with the same area as the void
    Perimeter=voiddata(i).Perimeter; %Specifies the perimeter of the
void
    %Removing voids that don't satisfy area or circular shape
conditions
    %by changing their color into the background color
    if (VoidArea >mmMaximumArea) | (VoidArea< mmMinimumArea) %Remove
voids that have an area greater than a maximum value or smaller than
a minimum value
        VoidArea;
        mmMaximumArea;
        labeled(voiddata(i).PixelIdxList)=0;
    elseif (Perimeter>(1.2*Diameter*pi())) %Remove voids that have a
perimeter greater than the perimeter of the equivalent circle
perimeter by 1.2
        Perimeter;
        EquivPerimeter=1.2*Diameter*pi();
        labeled(voiddata(i).PixelIdxList)=0;
    end
end
EntrainedAirBinaryImage=~labeled;
figure, imshow(EntrainedAirBinaryImage), title('Entrained Air');
[labeled2, numObjects2] = bwlable(~EntrainedAirBinaryImage,8); %Labeling 8-connected components in binary image
numObjects2;
EntrainedAirVoidNumber=numObjects2;
EntrainedAirImage=labeled2;
(c) Mix 3

(d) Mix 4
Figure B. Typical Image Processing Pictures
CURRICULUM VITAE

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Education

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<tr>
<th>Degree</th>
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<tr>
<td>M.Sc.</td>
<td>METU Civil Engineering</td>
<td>2006</td>
</tr>
<tr>
<td>B.S.</td>
<td>Cukurova University Civil Engineering</td>
<td>2002</td>
</tr>
</tbody>
</table>

Work Experience

- Sept 2009 – Present Materials Technology Unit, Qatar University, have been working as a Researcher
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**Publications**

- **Ozerkan, N.G.** Yaman, I.O., “Effect of Freezing-Thawing on Self Consolidated Concrete Including Fly Ash”, 33rd Conference on Our World in Concrete & Structures, Singapore, August 2009

**Languages**

Turkish (Native Language), English (Fluent)