

**UNIVERSITY OF GAZİANTEP  
GRADUATE SCHOOL OF  
NATURAL & APPLIED SCIENCES**

**EFFECT OF METAKAOLIN AND  
SILICA FUME ON THE MECHANICAL  
PROPERTY, SHRINKAGE, AND GAS  
PERMEABILITY OF HIGH  
PERFORMANCE CONCRETE**

**M.Sc. THESIS  
IN  
CIVIL ENGINEERING**

**BY  
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**Effect of Metakaolin and Silica Fume on the  
Mechanical Property, Shrinkage, and Gas  
Permeability of the High Performance Concrete**

**M. Sc. Thesis  
in  
Civil Engineering  
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**TO MY FAMILY**  
**CAN, SEMA, NURI KARAOGLU**

## ABSTRACT

### EFFECT OF METAKAOLIN AND SILICA FUME ON THE MECHANICAL PROPERTY, SHRINKAGE, AND GAS PERMEABILITY OF HIGH PERFORMANCE CONCRETE

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Using mineral admixtures as substituting additives in concrete has a tendency to increase by the future in order to provide greater sustainability in construction industry. The issues regarding the cost, recycling the industrial wastes, rehabilitation in durability and mechanical performance of concrete will therefore put a pressure on utilization of such materials. This study investigates the effectiveness of metakaolin (MK) and silica fume (SF) in contribution to the mechanical, shrinkage, and gas permeability of high performance concretes. Replacement levels of 5 and 15% of MK or SF by the weight of cement were considered in this study. The other experimental parameter investigated in the study was water-to-cementitious material ratios (w/cm) which were 0.25 and 0.35. The performance characteristics of the plain and mineral admixture incorporated concretes were evaluated by measuring the compressive strength, splitting tensile strength, static modulus of elasticity, drying shrinkage, restrained shrinkage, water sorptivity, and gas permeability. Test results revealed that replacement level of MK and SF had significant effects on the mechanical and especially durability characteristics of high performance concretes.

**Key Words:** Free shrinkage, Gas permeability, High performance concrete, Mechanical properties, Metakaolin, Restrained shrinkage, Silica fume.

## ÖZET

### METAKAOLİN VE SİLİS DUMANININ YÜKSEK PERFORMANSLI BETONDA MEKANİK ÖZELLİK, RÖTRE VE GAZ GEÇİRGENLİĞİ ÜZERİNDEKİ ETKİSİ

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Gelecekte inşaat sektörünün daha iyi bir sürdürülebilir gelişim sağlayabilmesi için betonda mineral katkı kullanımını artıran bir eğilime sahiptir. Maliyet, endüstriyel atıkların geri dönüşümünün sağlanması, betonun mekanik ve dayanıklılık özelliklerinin iyileştirilmesi gibi konular bu tür malzemelerin kullanımını zorunlu hale getirmektedir. Bu çalışma yüksek performanslı betonda, silis dumanı (SD) ve metakaolinin (MK) mekanik, rötre ve gaz geçirimsizliği etkisini incelemektedir. Beton üretiminde MK ve SD oranları %5 ve %15 oranlarında çimento ile yer değiştirilerek kullanıldı. Diğer deneysel parametre olarak su-bağlayıcı oranı 0.25 ve 0.35 olarak alındı. Kontrol ve katkıli betonların karakteristik performansları, basınç dayanımı, yarmada çekme dayanımı, statik elastik modülü, serbest rötre, sınırlanmış rötre, kılcal su ve gaz geçirimsizliği ölçülerek değerlendirilmiştir. Deney sonuçları MK ve SD oranlarının değişmesinin, yüksek performanslı betonun mekanik özellik ve dayanıklılık özellikleri üzerinde önemli etkilerinin olduğunu göstermiştir.

**Anahtar Kelimeler:** Gaz geçirimsizliği, Mekanik özellikler, Metakaolin, Serbest rötre, Sınırlanmış rötre Silis dumanı, Yüksek performanslı beton.

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## LIST OF SYMBOLS/ABBREVIATIONS

AC	Air curing
ASR	Alkali-silica reaction
ASTM	American society for testing and materials
CH	Calcium hydroxide
CR	Cement replacement
CSF	Condensed silica fume
DIN	Deutched institut für normng
FA	Fly ash
HPC	High performance concrete
HR	High resolution
HRM	High reactivity metakaolin
MK	Metakaolin
OPC	Ordinary portland cement
SEM	Scanning electron microscope
SF	Silica Fume
SP	Superplasticizer
SR	Sand replacement
WC	Water curing
$\sigma$	Compressive strength

## CHAPTER 1

### INTRODUCTION

#### 1.1. General

The purpose of this study is to further examine the influence of the supplementary cementing materials such as metakaolin (MK) and silica fume (SF) on the properties of high performance concrete. Concrete is the most important element of the infrastructure and well-designed concrete can be a durable construction material. However, the environmental aspects of portland cement are a growing concern, as cement manufacturing is responsible for about 2.5% of the total worldwide emissions from industrial sources. One effective way to diminish the environmental impact is to use natural pozzolans and/or supplementary cementing materials, as a partial replacement. This strategy will have the potential to reduce costs, conserve energy, and reduced waste volumes (Aldea, 2000). The cementing materials that are widely used, concrete constituents, are fly ash, ground granulated blast furnace, silica fume, metakaolin, etc. (Neville, 1996). Recently, there has been a growing interest in the utilization of high-reactivity metakaolin as a supplementary cementitious material in concrete construction industry.

The term 'mineral admixtures' is often used to cover all pozzolanic and cementitious materials, irrespective of their sources, and hence it is used in this study. Mineral admixtures used in the cement and concrete industries have different pozzolanic characteristics. Theoretical background is available in the literature to explain the pozzolanic activity of mineral admixtures on the basis of their mineral composition. The difference in free energy between source system and products system in the chemical reaction can be a reason for the pozzolanic activity of mineral admixtures. The concrete performance depends mainly on the environmental conditions, the microstructures, and the chemistry of the concrete. Here, the continuation of hydration reactions in portland cement is essential to improve the potential strength and durability of concrete. The type and the amount of supplementary material



present, the water – to – cementitious material ratio (w/cm) and the curing conditions. It is obvious that those factors are essential to obtain the durable concrete structures (Frias and Cabrera, 2001). Curing is necessary for concrete to fully realize its potential properties. The curing becomes more important for concrete incorporating pozzolans especially in hot and dry environments (Bisonette and Pierre, 1999). Based on test results, the effects of replacement level of metakaolin and silica fume, w/cm ratio and age upon strength and particularly durability properties of the concrete were discussed.

In this thesis, MK and SF were used as a replacement for portland cement (PC), with the levels of 5 and 15% to evaluate the efficiency of these materials upon the concrete properties. For this purpose, two groups of concrete mixtures with w/cm ratios of 0.25 and 0.35 were designed. The main focus of the study is to assess the influence of MK and SF on mechanical, permeability, and shrinkage properties of the concrete. Permeability related durability properties were examined through water sorptivity and gas permeability tests. Mechanical properties of the concrete investigated in this study were compressive and splitting tensile strengths with static modulus of elasticity at 3, 7 and 28 days. Restrained shrinkage properties of concrete produced by w/cm ratio of 0.35 was measured as well as free shrinkage and weight loss due to the corresponding drying. According to the test results the effects of replacement levels, w/cm ratios, and age upon mechanical and particularly shrinkage and permeability properties of the concrete were discussed.

## **1.2. Organization of the Thesis**

The thesis consists of five chapters.

Chapter 1 is describing the aim and objectives of the thesis.

Chapter 2 presents a literature review and gives a general background on admixtures, especially metakaolin and silica fume.

Chapter 3 presents the experimental program, briefly explains the test procedures, and summarizes the experimental data.

Chapter 4 includes the performed test results; evaluation and discussion exist in this chapter.

Chapter 5 is the conclusion of the thesis and possible further research studies complementing this thesis are also in this chapter.

## CHAPTER 2

### LITERATURE REVIEW AND BACKGROUND

#### 2.1 Mineral Admixtures

Mineral admixtures are finely divided siliceous materials which are added to concrete in relatively large amounts, generally in the range of 5 to 80 percent by weight of portland cement (Mehta and Monterio, 1997). Although pozzolans in the raw state or after thermal treatment are being used in some parts of the world, for economic reasons many industrial by-products are fast becoming the primary source of mineral admixtures in concrete.

Power generation units using coal as fuel, metallurgical furnaces producing cast iron, silicon metal, and ferrosilicon alloys are the major source of by-products which are being produced at the rate of millions of tons every year in many industrial countries. Dumping away these by-products, represents a waste of the material and causes serious environmental pollution problems. Disposal as aggregates for concrete and roadbase construction is a low-value use which does not utilize the potential of these pozzolanic and cementitious materials. With proper quality control, large amounts of many industrial by-products can be incorporated into concrete, either in the form of blended portland cement or as mineral admixtures. When the pozzolanic or/and cementitious properties of a material are such that it can be used as a partial replacement for portland cement in concrete, this results in significant energy and cost savings (Mehta and Monterio, 1997). Types of mineral admixtures according to their origin and type of production are shown in Table 2.1.

Table 2.1 Classification of mineral admixtures (Mehta and Monterio, 1997)

<b>Mineral Admixtures</b>	
<b>Materials</b>	<b>Sources</b>
Fly Ash	Volcanic Glasses
Blast Furnace Slag	Volcanic Tuffs
Condensed Silica Fume	Calcined Clays and Shales
Rice Huger Ash	Diatomaceous Earth

In the literature there are so many studies covering the investigation on the effects of mineral admixtures on the properties of concrete produced or cured under various conditions (Moon and Shin, 2007). Generally the effects on mineral admixtures may be assessed under the following titles;

- Improvement in mechanical properties
- Improvement in workability
- Durability to thermal cracking
- Durability to chemical attacks
- Production of high performance concrete

### **2.1.1. Natural mineral admixtures**

Natural mineral admixtures have been used for a long time in cement and concrete. Some of these admixtures, such as volcanic ash, trass, zeolite and diatom, exert a pozzolanic activity but their chemical and mineral composition vary depending on the source and conditions under which they were formed. These mineral admixtures consist predominantly of silica with as additional constituents of aluminium and ferric oxide. The term ‘pozzolan’ is derived from the volcanic ash mined at Pozzuoli, a village near Naples in Italy. A similar natural ash, formed as a result of volcanic eruption near Santorin around 1500 B.C., had been used for making lime-pozzolan mortars in Greece far before the word pozzolan was coined. Trass is a volcanic tuff that was used extensively in Germany, Greece, India, and Egypt a long time ago. Diatoms can contain silica contents as high as 94%. Diatomaceous soils can exhibit reactivity either before or after grading. (Malhotra and Mehta, 1986) More frequently, the diatoms are mixed with clay and other sediments; therefore, they need

to be calcined to exhibit adequate pozzolanic characteristics. Large deposits of diatomaceous earth are found in several parts of the world, including Algeria, California, Denmark, and France (Cook, 1986). In the past, diatoms were only used to a limited extent because of impurities and the large water requirement to obtain proper consistency.

Natural mineral admixtures are employed in many countries around the world, especially in developing ones. The activities of pozzolans show wide variation as a result of their natural origin. Some of the main types are zeolite and volcanic rock.

Pyroclastic rocks from Sardinia and Italy have been investigated for use as natural mineral admixture in concrete. Results show some rock materials to lead to satisfactory pozzolanic behaviour. In Iran, trass is used to substitute 20% by weight of Portland cement in mortar and concrete (Sistu, 1991). Although pozzolanic activity is low, the natural mineral admixtures were used for a long time to produce mortar and replace a part of Portland cement due to low costs involved.

The effects of zeolite on properties of cement and concrete depend on blending percentage (Vaughan 1978). The water demand of concrete in which Portland cement has been blended with 30% zeolite by weight is similar to that of plain Portland cement concrete with the same slump, but segregation will be reduced. The water demand will rise, however, at higher replacement levels. An addition of zeolite to concrete leads to increased strength and reduced pore content in the blended paste. More specifically, a 10 to 15% increase in compressive strength can be achieved by 10% zeolite blending in combination with a superplasticizer in concrete with water to binder ratios of 0.31 to 0.35. Additionally, this causes a significant decrease in bleeding (Naiqian, 1993). Also, the strength of concrete, in which 40% of Portland cement was replaced by zeolite, was higher at all ages than that of blast-furnace slag cements concrete with the same mixture proportions and under similar experimental conditions. Zeolite can decrease the degree of orientation of  $\text{Ca(OH)}_2$  in the interfacial transition zone between cement paste and aggregate, thereby improving the structure and properties of this zone. Adding natural zeolite to concrete can prevent expansion due to alkali-silica reaction of aggregate by reducing the concentrations of alkali in the pore water solution (Grutzeck, 1993).

In summary, several conclusions can be drawn from such studies as to the effects of bleeding with natural mineral admixtures:

- Significant cost reductions of cement and concrete.
- Conservation of energy, and of precious raw materials.
- Promotion of environmental protection.
- Delayed setting time.
- Modified normal consistency of cement and concrete depending on the kind and content of admixture used.
- Reduced permeability and thereby improved durability of mortar and concrete in aggressive environmental such as seawater and sodium-water.
- Reduced alkali-silica reaction.

### **2.1.2 Artificial mineral admixtures**

Predominantly, artificial mineral admixtures are employed nowadays in cement and concrete, because of the rapid exhaustion of natural resources for mineral admixtures. With progress in science and technology, some artificial mineral admixtures with high pozzolonic activity have been developed.

Mineral admixtures of artificial origin already in use in many countries around the world primarily include blast-furnace slag (BFS), fly ash (FA) and, to a smaller degree, calcined clay. More recently, particularly effectively mineral admixtures such as silica fume (SF), rice husk ash (RHA) and metakaolin (MK) have been introduced by the cement and concrete industry. The success of cement blending with industrial waste such as slag and FA is due to cost reductions and to improvements in certain characteristics of mortar and concrete behaviour that can be realised simultaneously. Chemical and mineral compositions of mineral admixtures vary with the source and with the conditions during their formation, inevitably leading to differences in pozzolanic activity. Hence, the efficiency of such technological measures can vary.

Fly ash is –like slag- an industrial waste that is widely used as mineral admixture in the cement and concrete industry. The chemical composition of FA depends on the

composition of the original coal ash. Fly ash with high glass content is used for blending with Portland cement (Thomas, 1999). The glass content in the fly ash depends on burning temperature, the source of the coal, and the cooling rate. Compared to BFS, ordinary and high calcium FA is low in CaO and MgO, and is rich in SiO<sub>2</sub>. The main chemical components of fly ash are varying amounts of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO, Fe<sub>2</sub>O<sub>3</sub>, and MgO. Fly ash is the main component in the waste mixture of fly ash and slag, which is produced by power stations that use coal as fuel. The amount of unburned carbon in the fly ash primarily depends on the combustion method. In a fly ash-slag mixture, the pozzolanic activity of the fly ash is lower than that of the slag.

Silica fume (SF) is a highly reactive mineral admixture composed of very fine particles of silica predominantly in the amorphous state. Indeed, the major chemical compound, usually taking up more than 80% of the silica fume, is SiO<sub>2</sub>, details depending on the manufacturing process (Pistille, 1985). The particles of silica fume have a size smaller than 0.1 μm.

Rice husk ash (RHA) is a product produced by burning rice husks at temperatures up to 750°C. The main component in RHA is silica oxide, generally ranging from 80 to 95%, depending on the unburnt carbon content (Bui, 2001). RHA usually contains a relatively high amount of unburnt carbon depending on the production conditions (Yu and Shoya, 1999). However, the reactivity of RHA is not diminished by the presence of carbon. Tridimite and cristobalite can occur in RHA as a result of conversion of the amorphous silica, depending on the temperature and the duration of the burning process, as well as on the cooling conditions. The pozzolanic activity of RHA is due to the amorphous state of the SiO<sub>2</sub>. Because of low soda, potash, and phosphate contents in the RHA, it can be used as a high quality mineral admixture by the cement and concrete industry.

Calcined materials are usually produced from raw materials as clay, soil, and kaolin. Clay and soil consist of small particles (less than 2 μm in size) and water molecules that are attached electrically to the surface of these particles. Clay minerals consist predominantly of silica and aluminum oxide, and of minor contents of alkaline. The pozzolanic activity of the calcined clay depends on the burning temperature and on

the composition. Basically, most soils and clays are mixtures and the dehydrating temperatures will depend not only on the dehydrating temperatures of individual components, but also on the interaction between the components in the dehydrating process. The optimum temperature of the calcination process of clay is around 800°C. This should last between 1 to 2 hours (Ambroise, 1993). Calcination significantly improves the strength development of natural pozzolans by replacing the original ingredients with new ones that have a higher pozzolanic activity. Metakaolin is the modern terminology for calcined kaolin. It has been used as highly reactive mineral admixture in cement and concrete.

Presently, a variety of highly reactive mineral admixtures are at our disposal for use in cementitious composites, such as silica fume, rice husk ash, and metakaolin. Many studies are available on the effect of such mineral admixtures on the performance of cement and concrete. These mineral admixtures are employed in normal concrete, as well as in high performance concrete either alone or in combination with more traditional mineral admixtures as slag and fly ash. So far, of course the traditional mineral admixtures play a more dominant role in the cement and concrete industry. The maximum blending percentage varies among the national and international standards, e.g. 65% in UK and USA, 70% in Japan and 80 % in Germany and the Russian republic (Roy, 1993).

A substantial number of studies cover aspects of influences exerted by slag additions on material properties of cement and concrete. However, the reactivity of slag varies and is not very high. As a consequence of the aforementioned high blending percentages, the characteristics of the blended portland cement mortar or concrete can be strongly influenced by those of the slag. Several conclusions as to effects of blending with BFS can be drawn from such studies:

- Lower cost of cement and concrete.
- Reduced thermal stresses during hardening due to the lower heat of hydration.
- Delayed setting times.
- Reduced water permeability.
- Improved performance in seawater and in a sodium-rich environment.



- Improved resistance to steel reinforcement corrosion.
- Lower alkali concentration in pore solution.
- Conservation of energy, and of precious raw materials.
- Promotion of environmental protection.

World-wide, large amounts of fly ash are added to concrete, despite its relatively low pozzolanic activity. Fly ash can be introduced into concrete in one of the following ways:

- To replace part of the Portland cement.
- Used as an additional component in the concrete.
- To replace part of the fine aggregate in concrete.

Fly ash in cement and concrete has been reported to affect both the properties of fresh and hardened concrete. The following effects are mentioned resulting from PC blending with FA (Bijen, 1996):

- Reduced water demand and setting process.
- Diminished segregation, bleeding and heat of hydration.
- Lower early-age strength of cement and concrete.
- Higher strength of matured mortar and concrete.
- Improved durability of mortar and concrete in salt and sulphate solution.
- Conservation of precious raw materials and energy.
- Increased corrosion risks due to high carbon contents.
- Economical and ecological benefits.

Among the artificial admixtures, calcined kaolin takes up a dominant position, due to its efficiency of improving aspects of concrete performance. The calcined kaolin, commonly referred to as metakaolin, is nowadays produced as a commercial high-quality product in several countries around the world. The nature and composition of metakaolin, as well as the effects exerted by blending with this mineral admixture on properties of cement and concrete are presented in detail as follows.

## **2.2. Metakaolin**

Many relatively new and useful products are nowadays at the disposal of the concrete industry, among which a range of mineral admixtures. Commercial, technological and sustainability arguments can be at the basis of employing such mineral admixtures. Primarily, technological potentialities are reflected by the use of mineral admixtures, such as silica fume, metakaolin, and rice husk ash, for the production of high performance concrete. Due to its white color, metakaolin is also a suitable candidate for application in architectural concrete. The commercial production of metakaolin is therefore rapidly growing.

Metakaolin (MK) is a reactive alumino-silicate pozzolan formed by burning purified kaolin or kaolinite clays at a specific temperature range and by grinding it to a high fineness. Metakaolin can be combined with calcium hydroxide to form hydrates. This contributes to improving properties of mortar and concrete. The reaction capacity of metakaolin depends mainly on its mineral composition, on the raw kaolin source and on the conditions of production.

### **2.2.1 Nature of metakaolin**

Metakaolin is produced from raw kaolin or kaolinite clay. Chemically, metakaolin encompasses as main components oxides as  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and in smaller quantities the oxides  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$ . The efficiency of metakaolin as a mineral admixture in cement and concrete is governed by high contents of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  (Wild, 1996).

Commercial metakaolin consists of active components as  $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  (metakaolinite) and  $\text{SiO}_2$  in amorphous or poorly crystalline form. However, the precise nature of dehydration products of kaolin or kaolinite clay is a matter of dispute. Formerly, metakaolin was conceived as a mixture of amorphous aluminium and silica oxides. Latter it was demonstrated that the metakaolin revealed still some of the original structural features of the kaolinite. Nowadays, most researchers share the opinion that the metakaolin consists of metakaolinite in amorphous and poorly crystalline form.

The behaviour of clay minerals under a regime of higher temperatures depends on structure, crystal size, and degree of crystallinity. Kaolinite decomposes above about 450<sup>0</sup>C, losing its hydroxyl groups in the form of water. At a temperature of 900<sup>0</sup>C or higher, the metakaolin undergoes structural changes to form crystalline compounds consisting of free silica oxide and mullite. At optimum temperatures (in the range of 650<sup>0</sup>C to 800<sup>0</sup>C), the main mineral of calcined kaolin is metakaolinite. This is the very reason calcined kaolin is generally attributed as metakaolin. The pozzolanic activity of calcined kaolin increases with the content of metakaolinite.

### **2.2.2. Metakaolin in cement and concrete**

Strength properties of paste and mortar are strongly influenced by kind, shape, size, and spatial dispersion of hydration products and by the pore characteristics. The reaction mechanisms underlying pozzolanic activity and the resulting effects of metakaolin on properties of blended Portland cement mortar and concrete have been the subject of many reports. Partial replacement of Portland cement (PC) by metakaolin (MK) was applied mid 1960s to improve the durability of concrete dams in Brazil. Since those days, the use of metakaolin in cement and concrete has increased significantly. Many companies nowadays produce highly reactive metakaolin used in the cement and concrete industry. At present, metakaolin is not only used in Portland cement and in normal concrete but also in high performance mortar and concrete (Curcio, 1998). The optimum content of metakaolin replacing an equal amount of Portland cement in mortar and concrete was subject of study in a couple of investigations. Additionally, the reactive ability of metakaolin in combination with C<sub>3</sub>S has also been envisaged experimentally. The result of these studies has improved the general knowledge about metakaolin application in mortar and concrete. The presence of metakaolin affects most properties of fresh and hardened paste, mortar, and concrete.

#### **2.2.2.1. Effect of metakaolin additions in fresh state**

The effects exerted by metakaolin additions during the first period of hardening on paste, mortar, and concrete will be effective also in the hardened state. Metakaolin increases the normal consistency of the cement, the extend of the effect depending on

the origin of the metakaolin, as well as on its fineness (Maximilien, 1993). Differences in calcination temperature and duration lead to differences in the normal consistency of metakaolin-blended cement pastes. As a consequence, the presence of metakaolin in concrete increases the water demand, but does not increase the risk of aggregate segregation in mortar and concrete. However, the reduction in slump of concrete due to MK-blending was less than experienced with silica fume at the same replacement level. The effect of metakaolin on water demand is smaller for mortar and concrete than for Portland cement paste containing the same amount of metakaolin.

Wong and Razak (Razak and Wong, 2005) studied efficiency of calcined kaolin and silica fume as cement replacement material on the fresh properties of concrete. They noted the following results about workability properties of concrete produced by different water-cementitious ratio (w/cm) at different replacement levels of SF and MK.

The workability characteristics of fresh concrete were assessed with respect to slump and Vebe time. Results are summarized in Table 2.2. The mixtures had slump values ranging from 30 to 260 mm while Vebe time was in the range of 1 to 15 s. The large variation of workability across mixtures was due to the constant superplasticizer dosage used for mixtures with the same w/cm ratio. The superplasticizer content was fixed with the intention of maintaining a standard material proportion and avoiding any effects of variation in superplasticizer content on fresh properties. Consequently, the slump reduced systematically as the amount of mineral admixture used in the mixture increased. It was observed that SF caused a more severe loss of workability compared with MK. This is attributed to its extremely high surface area of 21.3 m<sup>2</sup>/g, measured via nitrogen adsorption, which is double that MK (9.5 m<sup>2</sup>/g).

Table 2.2. Workability characteristics with some MK and SF replacement levels for different W/CM ratios (Razak and Wong, 2005)

W/CM	Mixture	Slump(mm)	Vebe(s)
0.27	PC	165	8
	MK5	155	8
	MK10	150	10
	MK15	115	10
	SF5	100	8
	SF10	50	12
	SF15	35	15
0.30	PC	225	3
	MK5	220	3
	MK10	210	3
	MK15	205	4
	SF5	215	3
	SF10	117	5
	SF15	30	16

#### 2.2.2.2. Effect of metakaolin additions in hardened state

Metakaolin additions exert influences on the main properties of matured paste, mortar, and concrete, such as strength and durability. Strength is one of the basic properties of cement and concrete. As a general conclusion, it can be stated that the optimum is different for paste, mortar, and concrete (Caldarone, 1995).

Based on such studies, it can be concluded that metakaolin additions to cement and concrete can increase:

- Flexural and compressive strength;
- Density;
- Chemical resistance;
- Alkali-silica resistance.

The underlying mechanisms are:

- Chemical improvements due to pozzolanic reactions;

- Physical improvements due to the particke packing (filler) effect.

Metakaolin addition leads to the formation of new cementitious compounds that influence the hydration process of the blended Portland cement, and thereby the strength and density of the matured material. Additionally, the metakaolin has a particle size distrubution deviating from that of the Portland cement. The significance of the latter effect depends on the gap between both particle size distribution functions. Hence, the finer the mineral admixture with respects to that of the Portland cement, the more prominent will be the filler effect in giving the material a higher density and improved strength capacity (Goldman, 1993).

One of the most important effects of the pozzolans is pore size refinement. Filling up large capillary spaces is important in terms of improving the strenght and impermeability of the concrete (Mehta and Monteiro, 1997). Thus, modifiers should be used in order to provide pore size refinement in concrete.

Bredy (Bredy et al., 1989) conducted a study on prosity and pore size distribution of pastes containing MK. Pastes were prepared with MK content of 0-50% at different water to binder (w/b) ratios to maintain the same consistency. They concluded that the total porosity of paste decreased if the MK content was below 20%. Beyond 30%, an increse in porosity was found. Larbi and Bijen (Larbi and Bijen, 1992) reported that at 100 days of curing the pore volume of mortar and the threshold diameter in the presence of MK were decreased. Khatib and Clay (Khatib and Clay, 2003) found a slight increase in pore volume for pastes containing up to %15 MK as partial replacement of cement and at a constant water/ (cement + MK), and this increase is dependent uopn the MK content. The presence of MK, however, causes refinement in pore structure, in that pastes containing MK devrease the threshold diameter and increase the percentage of small pores.

Poon (Poon, 2006) conducted a study on pore structure of high performance MK and SF concrete. They used mercury intrusion porosimeter (MIP) to measure pore size distribution of the concrete. Their finding about pore sturcture of concrete incorporated with MK and SF was the incorporation of MK in the cement pastes resulted in a very dence microstructure of the paste, with a lower porosity and finer

pore size distribution compared with the plain Portland cement pastes and SF blended cement pastes.

In the study of Singh and Garg (Singh and Gard, 2004), the microstructure of the metakaolin modified mortar has been reported. The effect of addition of metakaolin up to 25% in the Portland cement mortars was investigated. An increase in compressive strength and decrease of porosity and pore diameter of cement mortars containing metakaolin (10%) was reported over the cement mortars without metakaolin. The hydration of metakaolin blended cement mortars was investigated by differential thermal analysis (DTA) and scanning electron microscopy (SEM). DTA of hydrated cement showed (Figure 2.1) formation of endotherms of major hydraulic products such as C-S-H (130-135 °C), hexagonal calcium aluminate hydrate ( $C_4AH_{13}$ ) (180-200°C), and  $Ca(OH)_2$  (480-500°C) due to their decomposition reactions. The endotherms at 750-770°C is due to decomposition of calcium carbonate ( $CaCO_3$ ). An increase in peak areas of C-S-H and  $C_4AH_{13}$  was observed with increase in metakaolin up to 10%. SEM (Fig. 2.2.) showed formation of dense subhedral to euhedral crystals of C-S-H and  $C_4AH_{13}$  interspersed with short length ettringite at 3 days (a) of curing. Appearance of partially hydrated amorphous  $SiO_2$  and  $Al_2O_3$  can also be seen. At 7 and 28 days of hydration (b and c), well-crystallized CSH formed.

With the increase in curing period (d), well-developed euhedral CSH crystals with little unhydrated metakaolin are formed. The formation of these crystals is responsible for increase in strength of OPC-metakaolin mortar than the plain OPC mortar. Replacement of metakaolin with silica fume (up to 5%) did not show appreciable improvement in compressive strength of the cement.

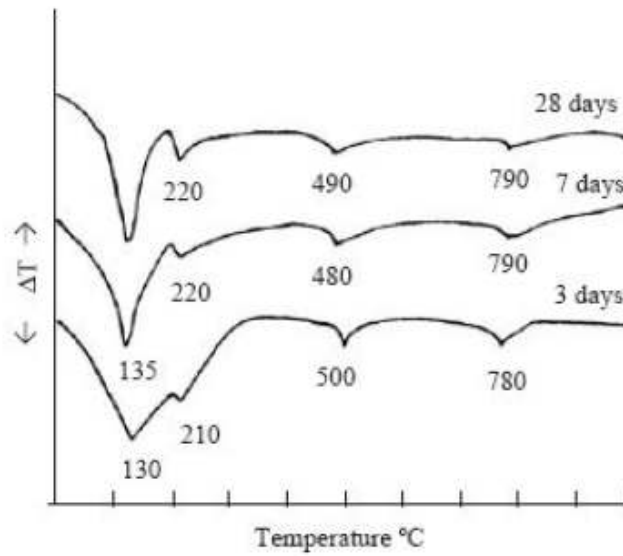


Figure 2.1 Differential thermograms of OPC-metakaolin mix hydrated for different periods (Singh and Gard, 2004)

They also used mercury intrusion porosimeter in order to observe the average pore diameter and porosity of mortars with and without MK. The mortar samples were moulded into 2.5×2.5×2.5 cm cubes at normal consistency and hydrated for different periods, crushed, and soaked in acetone and then placed in a vacuum oven at 60<sup>0</sup> to remove acetone and dried for 48 h. The cement mortars were reduced to 3 mm in size for pore-size distribution and porosity using a ‘Pore sizer 9320’ mercury intrusion porosimeter (micromeritics) with a maximum mercury intrusion pressure of 210 MPa. They summarized the result of mercury intrusion porosimeter in Table 2.4.



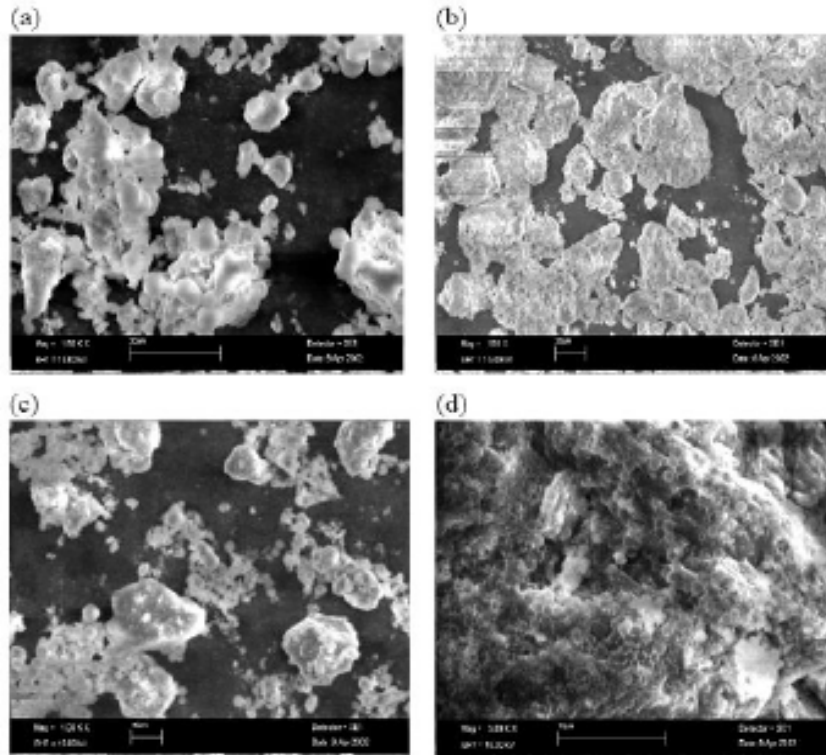


Figure 2.2 SEM of OPC-metakaolin mixture hydrated for different periods (a) 3 days (b) 7 days, (c) 28 days, and (d) 90 days (Singh and Gard, 2004)

Table 2.3 Average pore diameter and porosity of cement mortars with and without MK (Singh and Gard, 2004)

Curing Period	Average pore diameter ( $\mu\text{m}$ )		Total porosity (%v/v)	
	OPC-10% MK	OPC mortar	OPC-10% MK	OPC mortar
3 days	0.0266	0.0371	15.22	21.11
7 days	0.0252	0.0362	15.10	18.10
28 days	0.0187	0.0342	13.38	16.58
90 days	0.0176	0.0336	11.20	13.40

### 2.3. Fly ash

Fly ash is a by-product of the combustion of coal in thermal power plants, which is capable of reacting with  $\text{Ca}(\text{OH})_2$  at room temperature. The pozzolanic activity of fly ash depends on the presence of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  in the amorphous form (Baker,

1984). The use of fly ash in concrete technology dates back 1930s (Şengül, 2005). It is estimated that about 450 million tons of fly ash is produced worldwide annually, but only 6% of the total available is used as pozzolan in blended cements or in concrete mixtures (Baker, 1984). In Turkey, there are twelve active coal-burning power plants with annual fly ash production of about 15 millions tons.

According to ASTM C 618 (2002) fly ash is categorized in two classes: Class F and Class C. The classification is mainly based on if the fly ash in question carries only pozzolanic (Class F) or pozzolanic and cementitious (Class C) properties. The former is normally generated due to combustion of anthracite or bituminous coal whereas the latter is produced by means of burning of lignite or sub-bituminous coal. From a physical point of view, fly ash can also be very different from one another. They can appear in shapes such as spherical, rounded, irregular and angular. The particle size distribution of fly ash obtained by scanning electron microscope is provided in Figure 2.3.

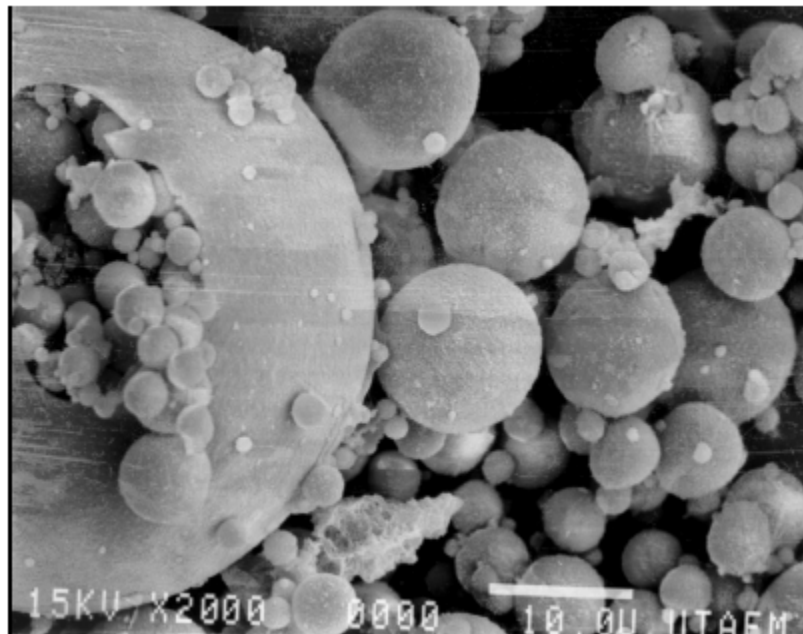


Figure 2.3 Scanning electron microscope image of fly ash particles (ACI 232,2 R-96, 1996)

### **2.3.1. Effects of fly ash on the fresh properties of concrete**

The absolute volume of cement plus fly ash normally exceeds that of cement in similar concrete mixtures not containing fly ash. This is because the fly ash normally is of lower density and the mass of fly ash used is usually equal to or greater than the reduced mass of cement. While it depends on the proportions used, this increase in paste volume produces a concrete with improved plasticity and better cohesiveness. In addition, the increase in the volume of fines from fly ash can compensate for deficient aggregate fines. Fly ash changes the flow behaviour of the cement paste; in general spherical shape of fly ash particles normally results in a reduction in the water demand in the concrete for a given workability (ACI232.2 R-96, 1996).

Bouzoubaa and Lachemi (2001) produced self compacting concrete (SCC) with a cement replacement of 40%, 50%, and 60% by Class F fly ash. They demonstrated that incorporating fly ash improved the slump flow diameter and decreased the V-funnel flow time of SCCs. Besides, the temperature rise of the SCC was 5 to 10°C lower than that of the control concrete and the setting times of SCCs were 3 to 4 hours longer than those of the control concrete.

Sonebi (2004) reported that the incorporation of pulverized fuel and limestone powder lessened the requirement of superplasticizer necessary to obtain the desired slump. The use of these materials also improved the rheological properties and reduced the risk of cracking of concrete due to the heat of hydration, thus led to more durable concrete. According to ACI 232.2R-96 (1996) report, the use of fly ash may extend the time of setting of concrete if the portland cement content is reduced.

Sekino and Narita (2003) conducted a study to propose the mix design method for SCCs containing high volume fly ash, and to evaluate the effects to replacing cement by fly ash on the strength and durability of concrete. They concluded that SCC at 50% replacement level of FA had excellent workability and filling at the site of construction. Xie (2002) studied the preparation technology of high-strength self-compacting concrete containing ultra-pulverized fly ash and superplasticizer. They found out that the effect of ultra-pulverized fly ash on fresh concrete is improve the viscosity of fresh concrete, and the effect being the same as that of a viscosity agent.

It does not decrease the flowability of fresh concrete. Kim (1996) studied the properties of super flowing concrete containing fly ash and reported that the replacement of cement by 30% fly ash resulted in excellent workability and flowability. Miura (1993) evaluated the influence of supplementary cementitious materials on workability and concluded that the replacement of cement by 30% fly ash significantly improves rheological properties.

### **2.3.2. Effects of fly ash on the hardened properties of concrete**

Fly ash concrete generally has lower early strength due to the slow pozzolanic reaction of fly ash. This strength reduction depends on the content and properties of the fly ash used. In the 1980s, CANMET designed the so-called high-volume fly ash (HVFA) concrete in which 55-60% of the portland cement is replaced by Class F fly ash and such concrete eventually demonstrated excellent mechanical and durability properties (Canada Energy Mines and Resources, 1986; Bilodeau and Malhotra, 2000; Malhotra, 1986).

Yazıcı (2007) performed a study to find out the effects of high volume FA replacement ratio on the properties of SCCs. In that study, cement was replaced by a Class C FA in various proportions from 30 to 60%. Similar tests were carried out with the incorporation of 10% SF to the same mixtures. The results showed that the compressive strength decreased with the increasing FA content at all ages. The compressive strength of control (0%FA) and 60% FA mixtures were 61.8 MPa and 28.4 MPa, respectively at 28 days. All mixes showed strength gain beyond 28 days and the control mixture reached to 72.5 MPa at 90 days while this value was 38 MPa for 60% FA content. However, it was possible to produce SCC with a compressive strength value of 50 MPa with 30-40% FA replacement. 10% SF addition to the system positively affected the compressive str. and contributed to the production of SCC mixtures that developed high-mechanical properties incorporating high-volume of FA. At 30% and 40% FA replacement levels, compressive strength values even exceeded the compressive strength of the control specimens at 28 days.

Saraswathy et al. (2003) prepared concrete specimens with 10%, 20%, 30%, and 40% of activated fly ash replacement levels and evaluated their compressive strength

at 7, 14, 28, and 90 days and the results were compared with that the ordinary portland cement concrete (without fly ash). They also measured electrical resistivity and ultrasonic pulse velocity of the concrete to understand the quality of concrete. In that study, it was noted that, curing time increased the compressive strength of concrete, irrespective of amount of fly ash replaced. A decrease in compressive strength of 1.5 times was observed as the percentage of fly ash replacement increased from 10 to 40 %. Saraswathy et al. (2003) also found that the electrical resistance of activated fly ash system showed less resistivity values than ordinary portland cement at replacement levels of 20% and above. This behaviour was observed due to the fact that, in activated fly ash the compatibility with hydrated cement phases and the remaining minerals phases act as more porous in the concrete formulation. With this result, the decrease in resistivity was observed. In addition, activated fly ash contains impurities like unburnt carbon, sulphur etc, increases the electrical conductivity and consequently decreases the resistivity values. Ultrasonic pulse velocity values observed for ordinary portland cement is found to be 4.35 km/s, whereas that for activated fly ash system ranged between 4.36 and 4.00 km/s.

#### **2.4. Silica Fume**

Silica fume is a by-product resulting from the reduction of high-purity quartz with coal or coke and wood chips in an electric arc furnace during the production of silicon metal or ferrosilicon alloys. The silica fume, which condenses from the gases escaping from the furnaces, has a very high content of amorphous silicon dioxide and consists of very fine spherical particles (Figure 2.4).

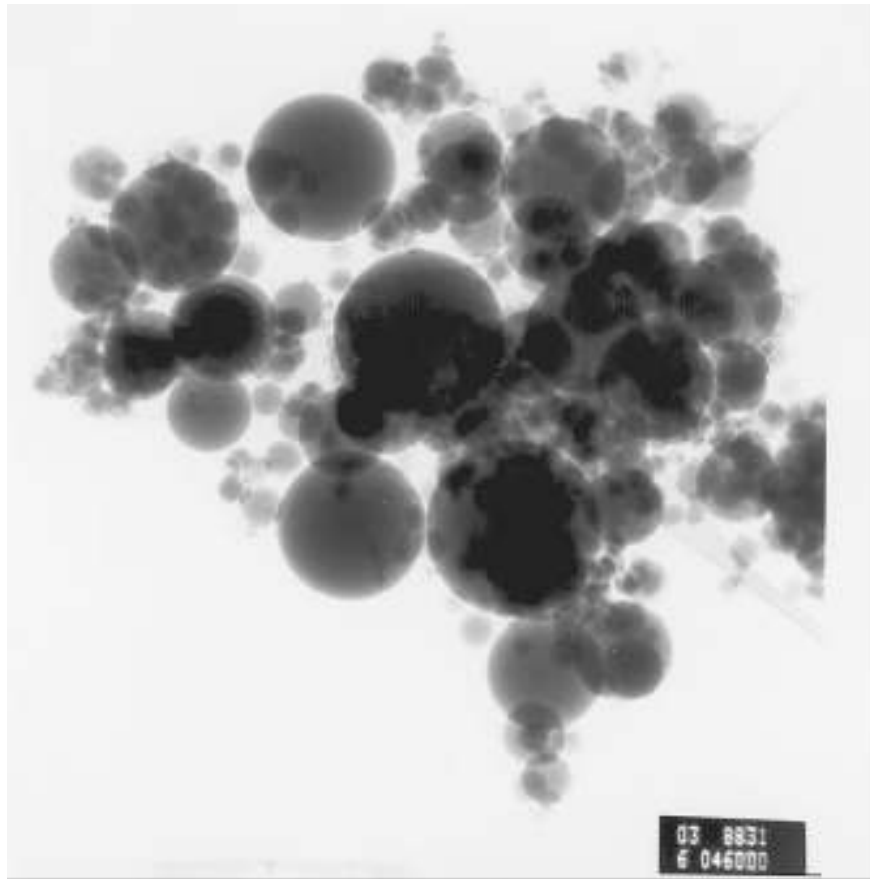


Figure 2.4 SEM micrograph of silica fume (ACI 234 R-96, 1996)

Ferrosilicon alloys are produced with nominal silicon contents of 61 to 98 percent. When the silicon content reaches 98 percent, the product is called silicon metal rather than ferrosilicon. As the silicon content increases in the alloy, the  $\text{SiO}_2$  content will increase in the silica fume (see Table 2.5). The majority of published data and field use of silica fume have been from production of alloys of 75 percent ferrosilicon or higher. Limited applications have been made using silica fume from production of 50 percent ferrosilicon alloys (ACI 234 R-96, 1996).

Table 2.4 Alloy types and their silica fume contents (ACI 234 R-96, 1996)

Alloy type	SiO <sub>2</sub> content of silica fume
50 percent ferrosilicon	61 to 84 percent
75 percent ferrosilicon	84 to 91 percent
silicon metal (98 percent)	87 to 98 percent

Most silica fumes range from light to dark grey in colour. Because SiO<sub>2</sub> is colourless, the colour is determined by the non-silica components, which typically include carbon and iron oxide. In general, the higher the carbon content, the darker the colour of the silica fume. The carbon content of silica fume is affected by many factors relating to the manufacturing process such as: wood chip composition, wood chip use versus coal use, furnace temperature, furnace exhaust temperature, and the type of product (metal alloy) being produced. The degree of compaction may also affect the colour.

The average diameter of silica fume particles are 0.1 µm, and their specific surface area is about 20000 m<sup>2</sup>/kg, as compared to 250 to 450 m<sup>2</sup>/kg for an ordinary portland cement or a fly ash. Silica fume contains 85 to 95% SiO<sub>2</sub> depending on the type of silicon alloy which is produced. Its chemical composition is generally very constant because of the high purity of the two main raw materials used in the process (quartz and coal). Its carbon content is generally less than 2 % (Delage and Aitcin, 1983).

#### **2.4.1. Effects of silica fume on the fresh properties of concrete**

The water demand of concrete increases with increasing the amounts of silica fume used due primarily to its high surface area (Scali et al., 1987; Caretta and Malhotra, 1983). Fresh concrete containing silica fume is more cohesive and less prone to segregation than concrete without silica fume. As the silica-fume content increases, the concrete may appear to become sticky. Concrete containing silica fume shows significantly reduced bleeding. This effect is caused primarily by the high surface area of the silica fume to be wetted; there is very little free water left in the mixture for bleeding. Additionally, the silica fume reduces bleeding by physically blocking the pores in the fresh concrete (ACI 234 R-96, 1996).

Silica-fume concrete usually includes chemical admixtures that may affect the time of setting of the concrete. Previous experiences indicate that the setting time is not significantly affected by the use of silica fume alone. Practical control of the setting time may be achieved by using appropriate chemical admixtures. In the literature, there is a general agreement on the retardation of initial and final setting times of the concrete containing FA and GGBFS. The behaviour seen in the SF concrete was different from those with FA and GGBFS in that the addition of SF generally reduced the initial and final setting times of the concrete, especially at 10 and 15% replacement levels. No general agreement could be found in the literature considering the effect of SF content on the setting times of concrete (Gesoglu and Özbay, 2007). Some researchers (Brooks et al., 2000; Alshamsi et al., 1993; Khedr and Abou-Zeid, 1994) stated that SF has retarding effect while some others reported its accelerating effect on the setting times. According to Pistilli et al. (1984) and De Almeida and Goncalves (1990), for a lower water/binder ratio concrete containing a superplasticizer, the effect of SF was to reduce the setting times when compared to those of control concrete containing the same amount of superplasticizer.

Park et al. (2005) investigated the influence of the cementitious materials containing fine particles on the rheology of the pastes. The mineral admixtures used were finely GGBFS, FA, and SF. Cementitious pastes were designed as one component Portland cement (PC), two-components (PC-GGBFS, PC-FA, and PC-SF), and threecomponents (PC-GGBFS-SF and PC-FA-SF) systems. It was found for the two component system that yield stress and viscosity decreased with replacing PC with GGBFS and FA. In the case of PC-SF system, however, yield stress and viscosity steeply increased with increasing SF. For both of the three-component systems the rheological properties of the cementitious pastes improved compared with the PC-SF binary systems.

#### **2.4.2. Effects of silica fume on the hardened properties of concrete**

Since silica fume improves the bond between the paste and aggregate, the influence of the quality of the aggregate on the mechanical properties of concrete becomes more important in silica-fume concrete.



Bhanja and Sengupta (2005) carried out an extensive experimental study over the water–binder ratios ranging from 0.26 to 0.42 and silica fume–binder ratios from 0.0 to 0.3. They were determined, compressive, flexural, and split-tensile strengths at 28 days. The compressive, as well as the tensile strengths increased with silica fume incorporation, and the results indicated that the optimum content is not constant but depends on the water–cementitious material (w/cm) ratio of the mixture as seen in Figure 2.5. When compared to splitting tensile strength, flexural strength has exhibited greater improvements.

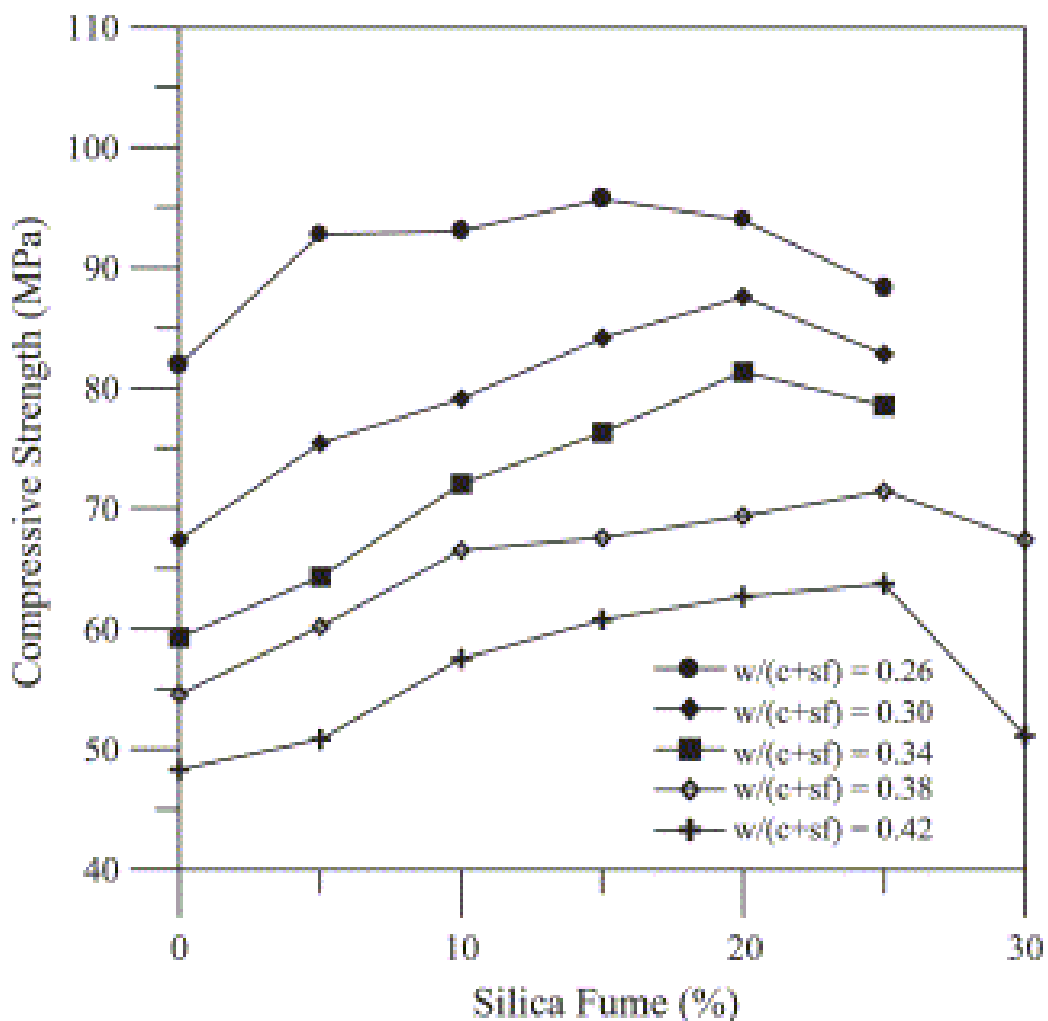


Figure 2.5 Influence of silica fume replacement and water binder ratio on the compressive strength of concrete (Bhanja and Sengupta, 2005)

According to ACI committee report (ACI 234 R-96, 1996), the static elastic modulus of silica fume concrete is apparently similar to that of portland cement concrete of

similar strength. Helland et al. (1983) concluded that the stress-strain behaviour of silica-fume concrete was similar to that of portland-cement concrete. Sellevold and Nilsen (1987) found that the dynamic modulus of elasticity increased with increasing silica-fume content in pastes.

Mazloom et al. (2004) demonstrated that the compressive strength development of the concrete mixtures containing silica fume was negligible after the age of 90 days; however, there were 26% and 14% strength increase in the control concrete after one year compared to its 28 and 90 days strengths, respectively. At the age of 28 days, the strength of concrete containing 15% silica fume was about 21% higher than that of the control concrete. Therefore, the inclusion of silica fume in concrete mixture, mainly affects short-term strength of concrete. The difference in strength development in ordinary portland cement concrete and silica fume concrete can be attributed (Wild et al., 1995) to the rapid formation of an inhibiting layer of the reaction products preventing further reaction of SF with calcium hydroxide beyond 90 days. In the case of the control concrete, hydration is at a less advanced stage and strength still shows significant improvement.

## CHAPTER 3

### EXPERIMENTAL STUDY

#### 3.1. Materials

##### 3.1.1 Cement

The cement used in this study is Portland cement (CEM I 42.5R) conforming to the Turkish standard TS EN 197-1 (which mainly based on the European EN 197-1). It has a specific gravity of 3.13 and a surface area (Blaine) of 339 m<sup>2</sup>/kg. The physical and chemical properties of the cement used are given in Table 3.1. It was supplied by Çimko Cement Factory.

Table 3.1 Chemical composition and physical properties of cement and mineral admixtures

Chemical analysis [%]	Portland cement	Metakaolin	Silica Fume
CaO	63.60	0.78	0.45
SiO <sub>2</sub>	19.49	52.68	90.36
Al <sub>2</sub> O <sub>3</sub>	4.54	36.34	0.71
Fe <sub>2</sub> O <sub>3</sub>	3.38	2.14	1.31
MgO	2.63	0.16	-
SO <sub>3</sub>	2.84	-	0.41
K <sub>2</sub> O	0.58	0.62	1.52
Na <sub>2</sub> O	0.13	0.26	0.45
Loss of ignition	2.99	0.98	3.11
Specific gravity	3.13	2.5	2.2
Specific Surface [m <sup>2</sup> /kg]	339	12000	21080

### **3.1.2 Metakaolin**

The metakaolin used in this study is a white powder. It has a specific gravity of about 2.50, a specific surface area (Nitrogen BET Surface Area) of 12000 m<sup>2</sup>/kg, and an average particle size less than 2 μm. Physical and chemical properties of MK used in this study are given in Table 3.2. The origin of the raw material is from USA.

### **3.1.3 Silica Fume**

A commercial grade silica fume (SF) obtained from Norway was utilized in this study. It had a specific gravity of 2.2 g/cm<sup>3</sup> and the specific surface area (Nitrogen BET Surface Area) of 21080 m<sup>2</sup>/kg. In Table 3.1, both the chemical analysis and physical properties of SF is provided.

### **3.1.4 Aggregate**

Both fine and coarse aggregates were used to produce the plain, metakaolin and silica fume modified concrete. The fine aggregate was a mix of river sand and crushed sand whereas the coarse aggregate was river gravel with a maximum particle size of 22 mm. Both aggregates were obtained from local sources. Properties of the aggregates are presented in Table 3.3. Grading of the aggregates is shown in the Figure 3.1.

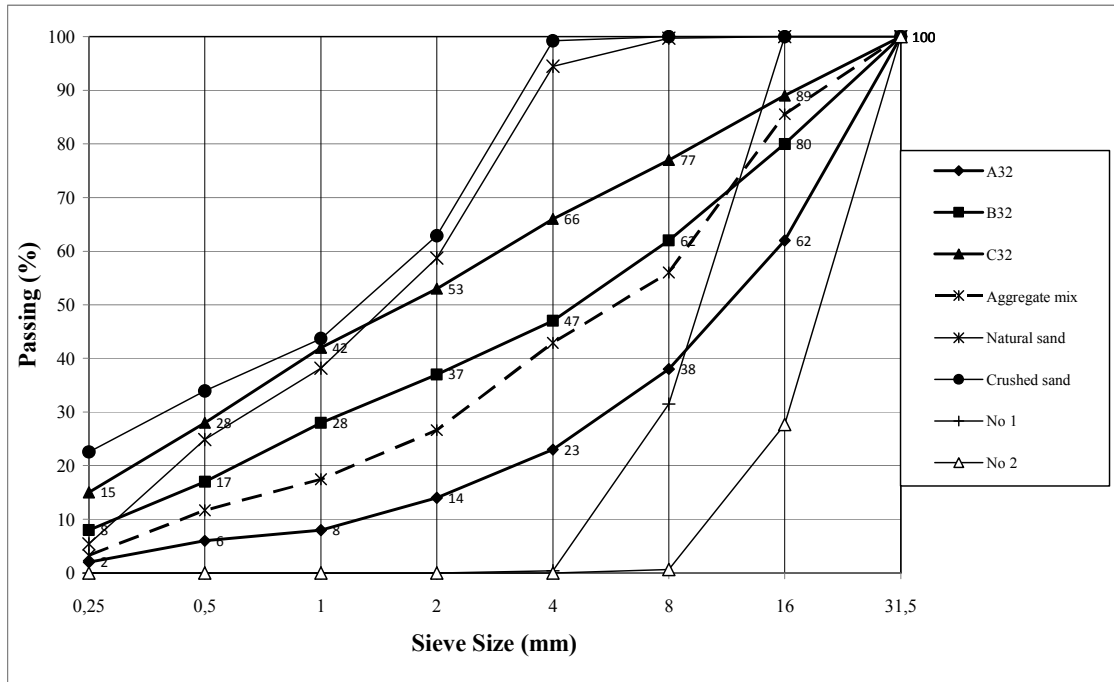


Figure 3.1 Grading of the aggregates

### 3.1.5 Superplasticizer

A polycarboxylic-ether type superplasticizer (SP) with a specific gravity of 1.07 and pH of 5.7 was used in all mixtures. The properties of superplasticizer are given in Table 3.2 as reported by the local supplier.

Table 3.2 Properties of superplasticizer

Properties	Superplasticizer
Name	Glenium 51
Color tone	Dark brown
State	Liquid
Specific gravity (kg/l)	1.07
Chemical description	Modified polycarboxylic type polymer

Table 3.3 Sieve analysis and physical properties of aggregates

Sieve size (mm)	Fine aggregate		Coarse aggregate	
	River sand	Crushed sand	No I	No II
31.5	100	100	100	100
16.0	100	100	100	27.74
8.0	99.7	100	31.5	0.6
4.0	94.5	99.2	0.4	0
2.0	58.7	62.9	0	0
1.0	38.2	43.7	0	0
0.50	24.9	33.9	0	0
0.25	5.40	22.6	0	0
Fineness modulus	2.79	2.38	5.68	6.72
Specific gravity	2.66	2.45	2.72	2.73

### 3.2. Details of Concrete Mixtures

Two series of control mixtures with w/cm ratios of 0.25 (Series 1) and 0.35 (Series 2) were designed. To develop the metakaolin and silica fume modified concrete mixtures, the portland cement was partially replaced with 5% and 15% mineral admixture by weight for both series. Thus, totally ten different mixtures were prepared including plain concretes. Details of the mixtures are given in Table 3.4. Grading of the aggregate mixture was kept constant for all concretes. The mixtures given in Table 3.4 were designed to have slump values of  $100 \pm 20$  mm and  $150 \pm 20$  mm for w/cm ratios of 0.25 and 0.35, respectively for the ease of handling, placing, and consolidation. The superplasticizer was added at the time of mixing to achieve the specified slump at each w/cm ratio.

Table 3.4 Concrete mixture proportioning for 1 m<sup>3</sup>

<b>w/cm ratio</b>	<b>Mix proportions(kg/m<sup>3</sup>)</b>	<b>Control I</b>	<b>5MK</b>	<b>15MK</b>	<b>5SF</b>	<b>15SF</b>
<b>w/cm=0.25</b>	Cement	550	522.5	467.5	522.5	467.5
	Water	137.5	137.5	137.5	137.5	137.5
	MK	0	27.5	82.5	0	0
	SF	0	0	0	27.5	82.5
	No1	736.2	733.7	728.6	732.0	723.6
	No2	257.7	256.8	255.0	256.2	253.3
	Nat. Sand	579.5	576.4	570.3	578.3	575.9
	Cr. Sand	177.9	177.0	175.1	177.5	176.8
	SP	4.23	5.40	8.40	5.62	9.60
	Theo. Density	2447.1	2439.7	2424.8	2439.8	2425.3
<b>w/cm=0.35</b>	<b>Mix proportions</b>	<b>Control II</b>	<b>5MK</b>	<b>15MK</b>	<b>5SF</b>	<b>15SF</b>
	Cement	470	446.5	399.5	446.5	399.5
	Water	164.5	164.5	164.5	164.5	164.5
	MK	0	23.5	70.5	0	0
	SF	0	0	0	23.5	70.5
	No1	735.70	733.54	729.21	732.11	724.93
	No2	257.50	256.74	255.22	256.24	253.72
	Nat. Sand	579.09	576.47	571.23	578.05	575.98
	Cr. Sand	177.79	176.99	175.38	177.47	176.84
	SP	1.86	3.00	4.31	2.80	4.47
	Theo. Density	2391.63	2385.28	2372.60	2385.43	2373.02

### 3.3. Test Procedures

#### 3.3.1. Mechanical Properties

##### 3.3.1.1. Compressive Strength

The concrete cubes (150×150×150 mm) were used for the compressive strength test at various ages. The compression test was carried out on the specimens by a 3000 kN capacity test machine according to ASTM C39. The test performed at the ages of 3, 7, and 28 days.

##### 3.3.1.2. Splitting Tensile Strength

Splitting tensile strength of the concrete will be measured on 150x300 mm cylinder specimens at 28 days as recommended by ASTM C 496. The splitting tensile strength reported herein is the average of three cylinders.

By definition the tensile strength is obtained by the direct uniaxial tensile test. But the tensile test is difficult and expensive for routine application. The splitting tensile test appears to offer a desirable alternative, because it is much simpler and inexpensive. The splitting tensile strength test is one of the simplest tests in which such stress fields occur. Since it is widely used in practice, a uniform test method is needed for data to be comparable. A uniform test is also needed to ensure that the disk specimens break diametrically due to tensile pulling along the loading diameter.

### **3.3.1.3. Modulus of Elasticity**

Cylinders with a dimension of 150x300 mm were tested for determining the static modulus of elasticity as per ASTM C469. Each of the specimens was fitted with a compressometer containing a dial gage capable of measuring deformation to 0.002 mm and then loaded three times to 40% of the ultimate load of companion cylinder. The first set of readings of each cylinder was discarded and the modulus was reported as the average of the second two sets of readings. For each parameter, three specimens were used.

### **3.3.2. Shrinkage**

#### **3.3.2.1. Drying Shrinkage (Free shrinkage) and weight loss**

Free shrinkage test specimens having a dimension of 70×70×280 mm for each mixture were cured for 24 h at 20 °C and 100% relative humidity and then were demoulded. After that, the specimens were exposed to drying in a humidity cabinet at  $23 \pm 2$  °C and  $50 \pm 5\%$  relative humidity, as per ASTM C157 for about 42 days. The length change was measured by means of a dial gage extensometer with a 200 mm gage length. The shape of the shrinkage specimens as well as the location of the reference pins are shown in Figure 3.2. Measurements were carried out every 24 h for the first 3 weeks and then 3 times a week. At the same time, weight loss measurements were also performed on the same specimens. Variations in the free shrinkage strain and the weight loss were monitored during the 42-day drying period (at  $23 \pm 2$  °C and  $50 \pm 5\%$  relative humidity) and the average of four prism specimens were used for each property.



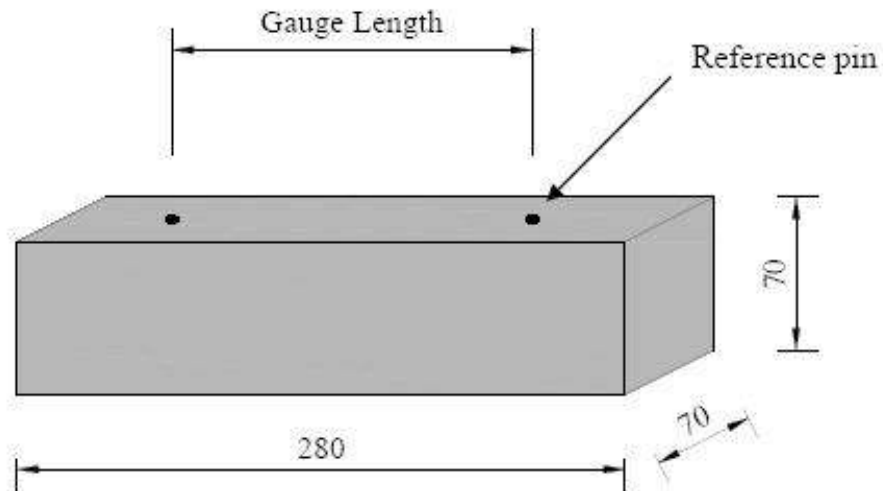


Figure 3.2 Shrinkage sample

### 3.3.2.2. Restrained Shrinkage

Ring-type specimens were used in this study to observe the restrained shrinkage-induced cracking of concrete. The scheme and the dimensions of the ring mould is shown in Figure 3.3., respectively. For such a ring, as the concrete was subjected to an internal pressure induced by the restraining inner steel tube, the difference between the values of the tensile hoop stress on the outer and the inner surface of the concrete was only 10%. Also, the maximum value of the radial stress was 20% of the maximum hoop stress. Thus, it can be assumed that the concrete annulus was essentially subjected to a uniform, uniaxial tensile stress when it was internally restrained by the steel ring. In addition, the width of the specimen (140 mm) was four times its thickness (35 mm), so that a uniform shrinkage along the width of the specimen can be assumed (Wiegrink et al., 1996; Grzybowski et al., 1990; Shah et al., 1993).

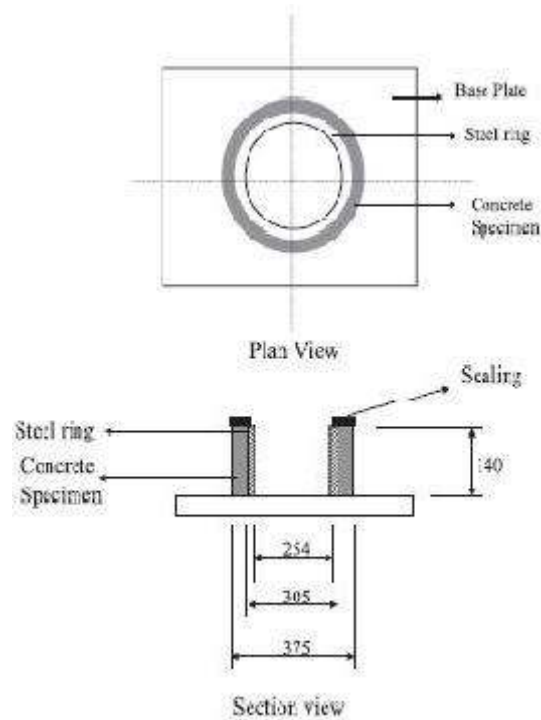


Figure 3.3 Restrained shrinkage ring specimen (in mm)

Free and restrained shrinkage specimens were cured for 24 h at 20 °C and 100% relative humidity and were then demoulded. After the outer steel ring had been stripped off, the top surface of the concrete ring was sealed off using silicon rubber, so that the drying would be allowed only from the outer circumferential surface. After that, the specimens were exposed to drying in a humidity cabinet at  $23 \pm 2$  °C and  $50 \pm 5$  % relative humidity, as per ASTM C157-75 for about 42 days. All the other test specimens were first maintained under plastic sheets for 24 h and were then demoulded. Specimens for compressive strength, splitting-tensile strength, and modulus of elasticity were water-cured for 28 days after demoulding.

### 3.3.3. Permeability

#### 3.3.3.1. Gas Permeability

Gas permeability of concrete can be used to characterize durability properties of concrete. The gas permeability of each concrete mixture was measured at the end of 28 days water curing period. Two specimens of each type of concrete were tested. After curing period, two 50 mm height and 150 mm diameter disk specimens were

cut from the middle of each 150\*300 – mm cylinder. Then, the following steps of the gas permeability test are utilized;

1. Measure the diameter of the test specimen in 4 positions (two perpendicular diameters in both top and bottom faces) with a precision of 0.1 mm. The diameter  $D$  is the mean value of the four readings. The thickness  $L$  of the test specimen is determined in four positions equally distributed along the perimeter.
2. Place the test specimen in the cell and assemble the apparatus.
3. Build up a minimum lateral pressure of 7 bar (0.70 MPa) on the rubber tube.
4. Select 3 pressure stages: start with 1.5 bar (0.15 MPa) and increase to 2.0 (0.20 MPa) and then 3.0 bar (0.30 MPa) absolute gas pressure. Correct the input pressure of gas if necessary within 10 minutes.
5. Wait for 30 minutes before measuring the first flow.
6. Measure the flow at each pressure stage until it becomes constant, as follows:
  - a. Moisten the capillary of the soap bubble flow meter 1 minute before creating the bubble for measurement.
  - b. Always start the time measurement when the bubble is at the lowest marking of the calibrated tube.
  - c. Select the measuring volume by choosing the appropriate soap bubble flow meter such that the time reading is more than 20 seconds.
  - d. Take provisional readings of the flow rate. If the difference between successive readings within 5 to 15 minutes is less than 3%, take at least 2 readings in quick succession and determine the flow rate  $Q_i$  (passed volume  $V_i$  at pressure stage  $i$  divided by average time reading of the previous two readings  $t_i$ ):  $Q_i = V_i/t_i$  (m<sup>3</sup>/s) for the given pressure stage. If this condition is not reached within 3 hours (no constant

flow is attained, *e.g.* very low-permeability concrete), take the previous value of the flow rate.

7. Increase the pressure to the next pressure level and repeat the procedure with steps (6a) through (6d). Ensure that there are no leaks during the tests: the coefficient  $K_i$  should decrease when the pressure increases. If this is not the case, check the test setup for possible leaks and repeat the measurements.

$$K_i = \frac{2P_2QL\mu}{A(P_1^2 - P_2^2)}$$

Where,

$K$ : Gas permeability coefficient ( $m^2$ )     $P_1$ : Inlet gas pressure ( $N/m^2$ )

$P_2$ : Outlet gas pressure ( $N/m^2$ )     $A$ : Cross-sectional area of the sample ( $m^2$ )

$L$ : Height of sample (m)     $\mu$ : Viscosity of oxygen ( $2.02 \times 10^{-5} \text{ Nsn}/m^2$ )

$Q$ : Rate of flow of air bubble ( $m^3/sn$ )



a)

b)

Figure 3.4 Photographic view of the gas permeability test set up

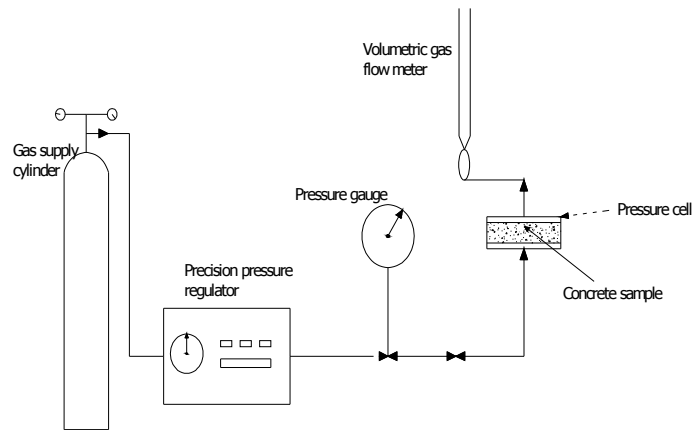


Figure 3.5 Schematic presentation of the gas permeability test set up

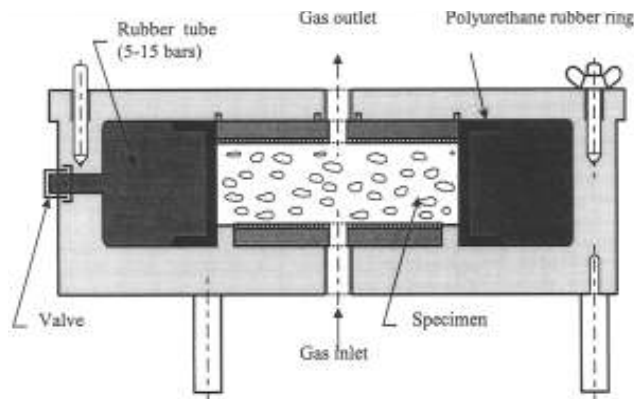


Figure 3.6 Schematic Presentation of the Pressure Cell and test specimen

### 3.3.3.2. Water Sorptivity

The sorptivity test measures the rate at which water is drawn into the pores of concrete. For this, three test specimens having a dimension of 100×100×100 mm were employed. The specimens were dried in an oven at about 105 °C until constant mass and then allowed to cool to the ambient temperature in a sealed container. Afterwards, the sides of the specimens were coated by paraffin and as shown in Figure 3.8. The sorptivity test was carried out by placing the specimens on glass rods in a tray such that their bottom surface up to a height of 5 mm is in contact with water. This procedure was considered to allow free water movement through the bottom surface. The total surface area of water within the tray should not be less than 10 times that of the specimen cross-sectional area. The specimens were removed

from the tray and weighed at different time intervals up to 1 hour to evaluate mass gain. The volume of water absorbed was calculated by dividing the mass gained by the nominal surface area of the specimen and by the density of water. These values were plotted against the square root of time. The slope of the line of the best fit was defined as the sorptivity coefficient of concrete. For each test, the measurements were obtained from three specimens and the average values were reported. The test will be conducted at the ages of 28 days.

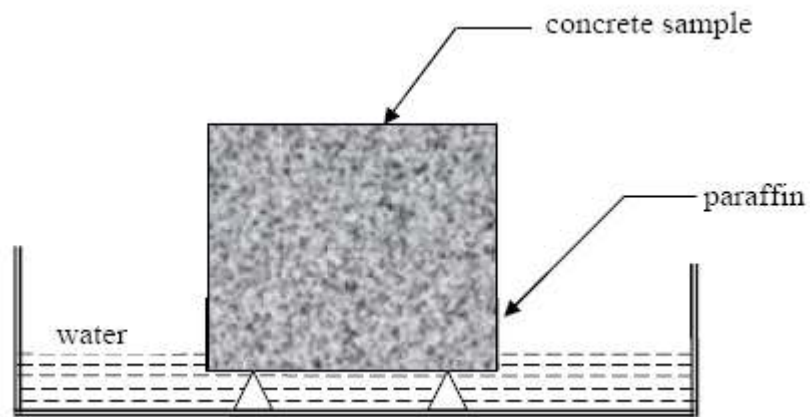


Figure 3.7. Measurement of concrete sorptivity

## CHAPTER 4

### TEST RESULTS AND DISCUSSIONS

#### 4.1 Compressive Strength

The data concerning the compressive strength development with w/cm ratio and concrete age for control and mineral admixture incorporated concrete were presented in Figure 4.1. The effect of MK and SF on compressive strength of concrete can clearly be observed from the Figure. The SF and MK concrete had consistently higher compressive strength than the control concrete. The 28 day compressive strength of the concrete with w/cm ratios of 0.25 and 0.35 were ranged between 75.4 - 85.8 and 61.8 - 73.3 MPa, respectively, without depending on the type of the mineral admixture and replacement level. The figure indicated that there was a systematic increase in compressive strength with the increase in MK and SF content for both of the concrete groups. This is more pronounced for concrete with w/cm ratio of 0.35. For example, observing 28-days compressive strength, we calculate the strength gain of 35%, 43%, 59%, 39%, and 44% for plain, SF5, SF15, MK5 and MK15 concrete with w/cm ratio of 0.35, respectively, in reference to 3-days compressive strength. Whilst, 13%, 27%, 39%, 18% and 29% compressive strength gains were observed for the concrete group with w/cm ratio of 0.25. In both cases, SF incorporated concrete showed higher rate of strength gain than plain and MK concrete. It must be kept in mind that, the degree of strength enhancement due to MK and SF is in direct relation to the parameters of age and the replacement level. In the study of Poon et al. (2006) the strength properties of the high performance metakaolin and silica fume was also investigated. Regarding the use of MK, they demonstrated that MK concrete had relatively higher strength development than control concrete. It was reported that the ratio of the 90 day compressive strength to the 28 day compressive strength for the MK concrete was found as up to 1.18, depending mainly on replacement level of MK and w/cm ratio.

It was also noted that for w/cm ratio of 0.35, the concrete with MK had higher compressive strength than the plain and SF concrete. Nevertheless, the compressive strength development of MK and SF concretes produced by 0.25 w/cm ratio, followed a close trend. The addition of metakaolin into the matrix improves the bond between the cement paste and aggregate particles as well as increasing the density of the cement paste which in turn significantly improves the compressive strength of the concrete. According to the literature, the main factors that affect the contribution of metakaolin in the strength are a) the filling effect, b) the dilution effect, and c) the pozzolanic reaction of metakaolin with CH (Wild et al. 1996). Wild et al. (1995) reported that the inclusion of silica fume in concrete mixture, mainly affects short-term strength of concrete. The difference in strength development in ordinary portland cement concrete and silica fume concrete can be attributed to the rapid formation of an inhibiting layer of the reaction products preventing further reaction of SF with calcium hydroxide.

Table 4.1 Compressive strength of plain, silica fume, and metakaolin modified concrete with w/cm ratios of 0.25 and 0.35

w/cm ratio	Mixture details	Compressive strength, MPa		
		3 days	7 days	28 days
w/cm=0.25	Control I	66.5	68.5	75.4
	5MK	67.1	72.5	78.8
	15MK	64.8	79.4	83.4
	5SF	62.9	72.4	79.6
	15SF	61.7	63.9	85.8
w/cm=0.35	Control II	45.7	51.7	61.5
	5MK	47.5	58.9	66.2
	15MK	50.9	65.2	73.4
	5SF	43.4	53.4	61.8
	15SF	41.7	50.6	66.2



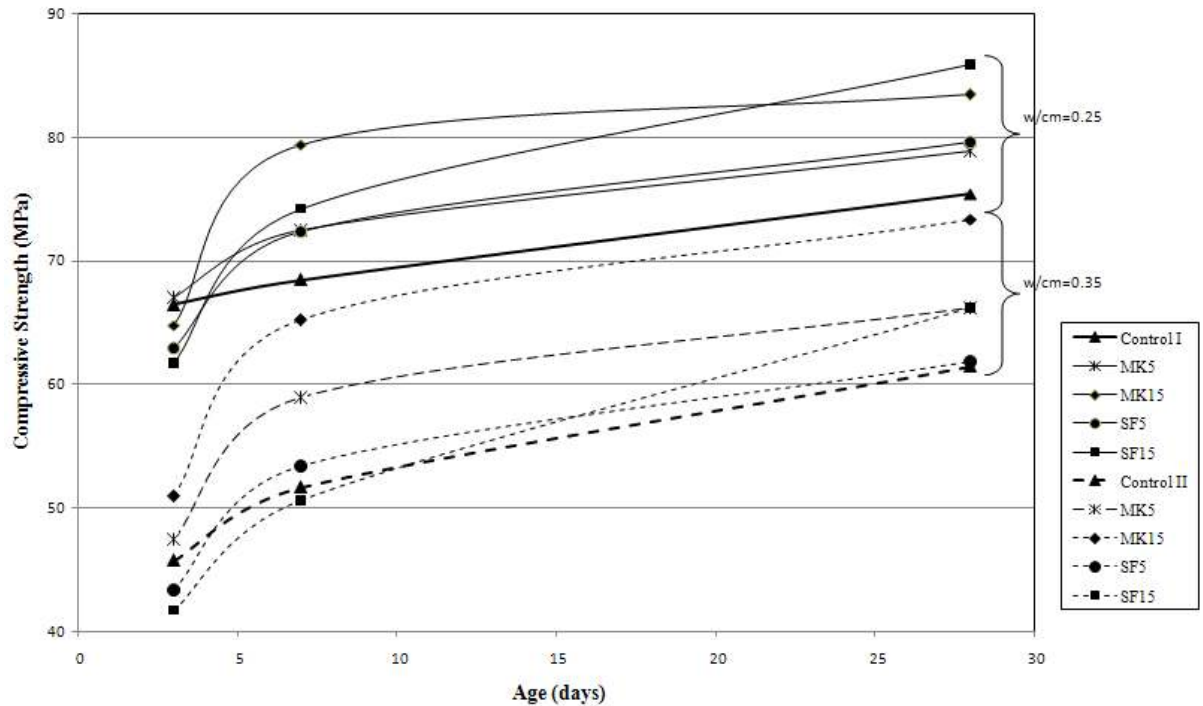


Figure 4.1. Variation in compressive strength of plain, silica fume, and metakaolin concretes with w/cm ratios of 0.25 and 0.35

## 4.2 Splitting Tensile Strength

The indirect tensile strength of the concrete measured by splitting test at 3, 7, and 28 days were graphically depicted in Figure 4.2. This figure illustrates the strength development pattern for splitting tensile strength is similar to that of compressive strength. Neville (2006) reported that there is a direct proportionality between splitting tensile and compressive strength of concrete strength. In other words, while the compressive strength increased, the tensile strength also increased at a lower rate.

The highest tensile strength values were obtained for the concrete with 15% replacement of SF and MK for w/cm ratios of 0.25 and 0.35, respectively. 3-days splitting tensile strength of SF and MK concrete with w/cm ratio of 0.25 were generally less than that of control. However, at later ages all of the splitting tensile strength of concrete with SF or MK was greater than control mixes in both w/cm ratios, independent of the amount of mineral admixture used. However, this increase in the splitting tensile strength was relatively smaller compared to that obtained in the compressive strength. For instance, control concrete had 28-days splitting tensile

strengths of 5.26 and 3.52 MPa for w/cm ratios of 0.25 and 0.35, respectively, while the highest values were observed as 5.92 and 4.39 MPa for the concrete mix contain 15% of MK and SF. The SF5 concrete with to w/cm ratios had similar splitting tensile strength values compared with control concrete at all ages.

Table 4.2 Splitting tensile strength of plain, silica fume, and metakaolin concretes with w/cm ratios of 0.25 and 0.35

w/cm ratio	Mixture details	Splitting tensile strength, MPa		
		3 days	7 days	28 days
w/cm=0.25	Control I	4.6	4.7	5.3
	5MK	4.7	5.3	5.8
	15MK	3.7	5.5	5.9
	5SF	4.7	5.3	5.2
	15SF	3.6	4.5	5.9
w/cm=0.35	Control II	2.3	3.2	3.5
	5MK	2.7	3.2	3.9
	15MK	3.5	4.1	4.4
	5SF	3.4	3.8	3.8
	15SF	3.1	3.3	3.9

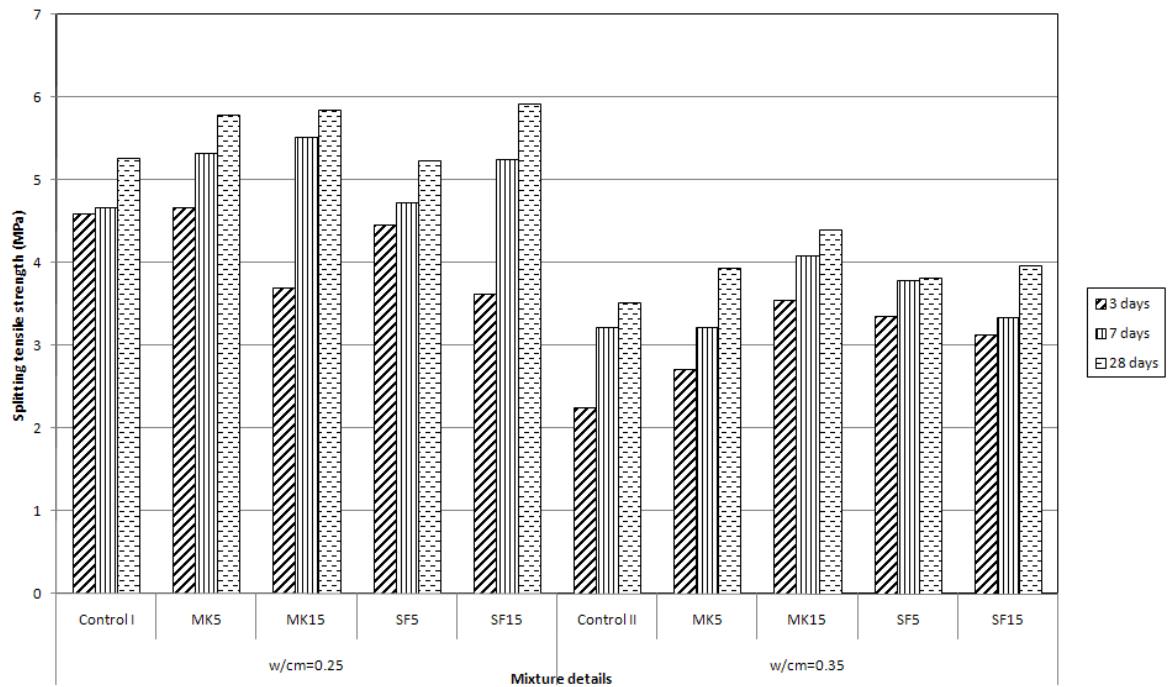


Figure 4.2. Variation in splitting tensile strength of plain, silica fume, and metakaolin concretes with w/cm ratios of 0.25 and 0.35

### 4.3 Modulus of Elasticity

The variations in modulus of elasticities of concrete dealt with in this study were presented in figure 4.3. By critical observation of the figure, it can be seen that the elasticity modulus increases with increase in MK and SF replacement, but the rate of increase is less than that for the compressive strength. It has rather a similar trend with splitting tensile strength development. The ranges of the modulus elasticity values were observed to be 43.9-51.2 GPa, and 24.3-48.8 GPa for concrete groups of 0.25 and 0.35 w/cm ratios, respectively. There is prominent difference between the developments of the modulus of elasticities of concrete groups. The concrete group with 0.25 w/cm ratio has a gradual rate of increase, while the concrete group with higher w/cm ratio has dramatically rise in modulus of elasticity values as time passes. This situation can be attributed to the variation in hydration kinetics due to the amount of water that exists in the medium of reactions (Wojcik et al. 2001).

Addition of MK and SF had an important contribution to the elastic modulus of concrete at all ages, without depending on the w/cm ratio and replacement level. This situation leads an improvement in mechanical behavior of concrete for responding the applied stresses. The maximum modulus of elasticity values were achieved at SF15 and MK15 concrete at concrete groups of 0.25 and 0.35 w/cm ratios, respectively.

Table 4.3 Modulus of elasticity of plain, silica fume, and metakaolin concretes with w/cm ratios of 0.25 and 0.35

w/cm ratio	Mixture details	Modulus of Elasticity (GPa)		
		3 days	7 days	28 days
w/cm=0.25	Control I	46.4	44.0	48.8
	5MK	43.9	47.3	50.2
	15MK	46.5	48.0	50.9
	5SF	46.0	48.0	49.1
	15SF	44.0	45.5	51.2
w/cm=0.35	Control II	24.3	31.7	42.7
	5MK	24.1	31.5	40.6
	15MK	30.5	40.2	48.8
	5SF	25.0	39.6	42.3
	15SF	26.2	28.4	39.5

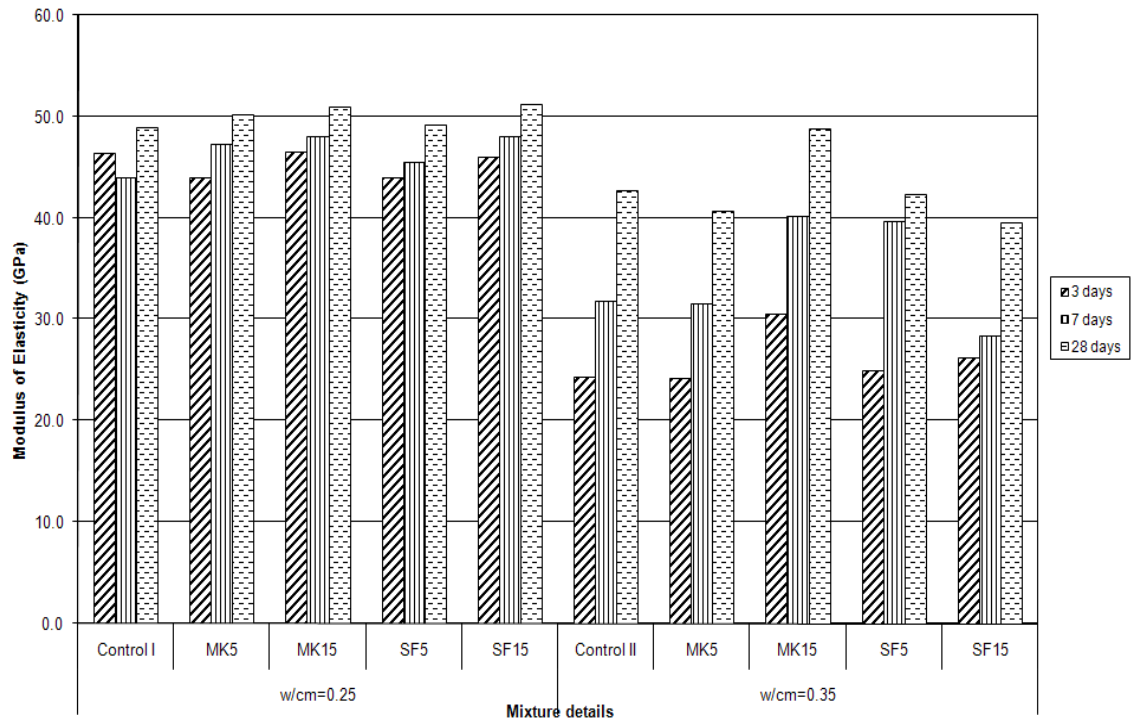


Figure 4.3 Variation in modulus of elasticity of plain, silica fume, and metakaolin concretes with w/cm ratios of 0.25 and 0.35

#### 4.4. Drying Shrinkage and Weight Loss

Drying shrinkage tests can provide necessary information on how the drying shrinkage stresses develop (Wiegrink et al. 1996). As a result, the observation of free drying shrinkage of the concrete may direct the investigators to find out the deterioration mechanism in concrete due to the time dependent deformations.

The strain developments versus time of the drying shrinkage specimens produced with w/cm ratio of 0.35 was presented in Figure 4.5. It was observed from the figure that drying shrinkage of the concrete had decreasing tendency with little fluctuations through the time passed. Even at early test ages, the shrinkage of the plain concrete specimens developed quickly and diverged from MK and SF modified concrete. The difference between amount of drying shrinkage of MK and SF incorporated concrete was small throughout the test period. Especially after 1 week of drying the difference in shrinkage strain values of plain and mineral admixed concrete became clearer and the shrinkage of concrete with MK or SF decreased with increasing replacement levels. When the shrinkage values at 7 weeks were considered, the shrinkage of MK

and SF concrete especially at 15% level of replacement exhibited remarkable reduction in comparison to plain concrete shrinkage. Although the mineral admixed concrete showed a similar trend in drying shrinkage strain development, it was notified that the more the replacement of MK or SF, the more the reduction in shrinkage, irrespective of time. For example, the drying shrinkage of the concrete mixtures MK5 and SF5 were approximately 33 and 29% less than that of the plain concrete, respectively. However, MK15 and SF15 concrete provided the reductions in drying shrinkage values of 42 and 35%, respectively. These findings are all compatible with the observations of Al-Khaja (1994), Jainyong and Yan (2001) and Güneyisi et al. (2008) for the effect of the ultrafine mineral admixtures. In the study of Al-Khaja (1994), it was concluded that the shrinkage and creep of plain concrete were considerably or moderately reduced with the incorporation of silica fume, showing a one-month reduction in strain of 34.9 and 18.5% for shrinkage and creep, respectively, which led to a reduction in the total deformation of 20.8%. Jainyong and Yan (2001) have also showed that ultrafine ground granulated blast-furnace slag and silica fume can substantially promote hydration of cement and increase in the amount of AFt crystal hydrates and C-S-H gel hydrates in cement paste, which offers hardened concrete a stronger structure and higher resistance to deformation caused by applied forced. As well, these two binders may fill small pores and voids harmful to the structure of concrete. That might be the mechanism of reducing effect of ultrafine mineral admixtures (i.e. GGBS, SF) on creep and drying shrinkage of concrete. Güneyisi et al. (2008) used MK as a supplementary cementing material to improve strength, pore structure and drying shrinkage of concrete. They chose two different replacement levels, namely, 10% and 20% in reference to plain concrete. They observed that, the inclusion of MK as a partial cement replacement material provided an excellent improvement in the pore structure and MK modified concrete had remarkably lower drying shrinkage strain than the plain concrete, irrespective of the w/cm ratios.

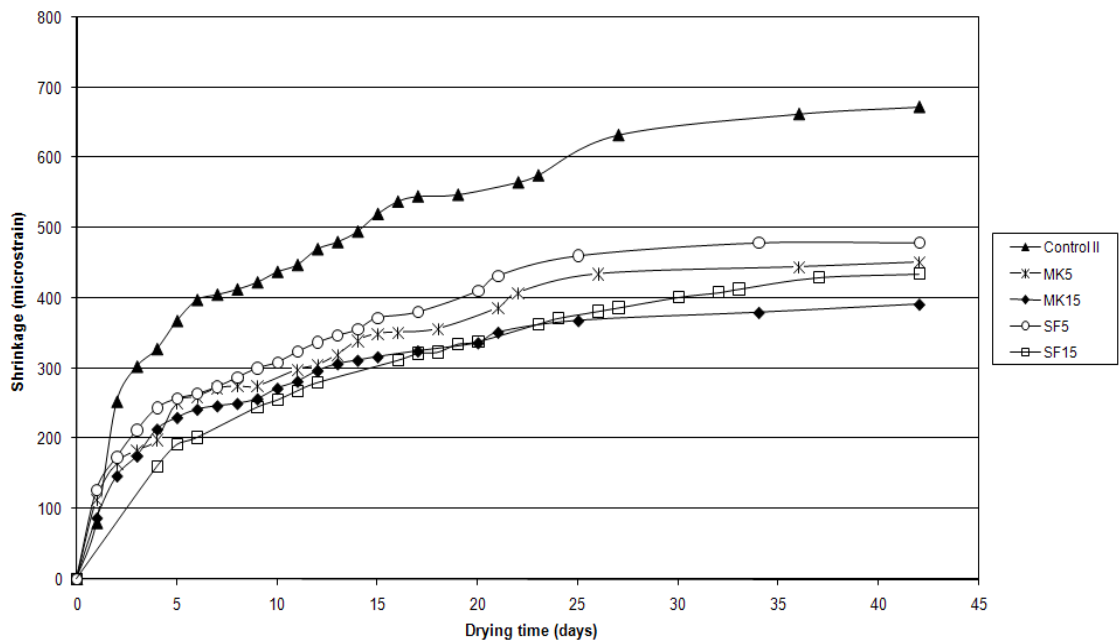


Figure 4.4 Drying shrinkage vs. drying time graph of plain, MK, and SF concretes having w/cm ratio of 0.35

Due to the shrinkage-decreasing or compensating properties of MK and SF as well as improvement in mechanical properties, may lead the practitioners to use these materials as an additive where high shrinkage should be avoided or undesirable.

The results of weight loss due to drying for the concrete mixtures were also shown in Figure 4.6. It was clear from the figure that concrete mixes incorporated with mineral admixtures showed lower weight loss. Similar to the drying shrinkage test results, inclusion of MK and SF to the concrete mixes decreased the weight loss considerably. During 42 days drying period, the difference of weight loss between plain and mineral admixture blended cement concrete became more distinguishable after two weeks, irrespective of the replacement level. The differences were then observed to have an increasing tendency with increasing the drying time. It was observed that after 42 day drying, the MK and SF concrete exhibited up to 18% and 26%, respectively, less weight loss compared to plain concrete, depending mainly on replacement level. It seems that the rate of weight loss for MK and SF concrete slows down earlier than that for plain concrete.

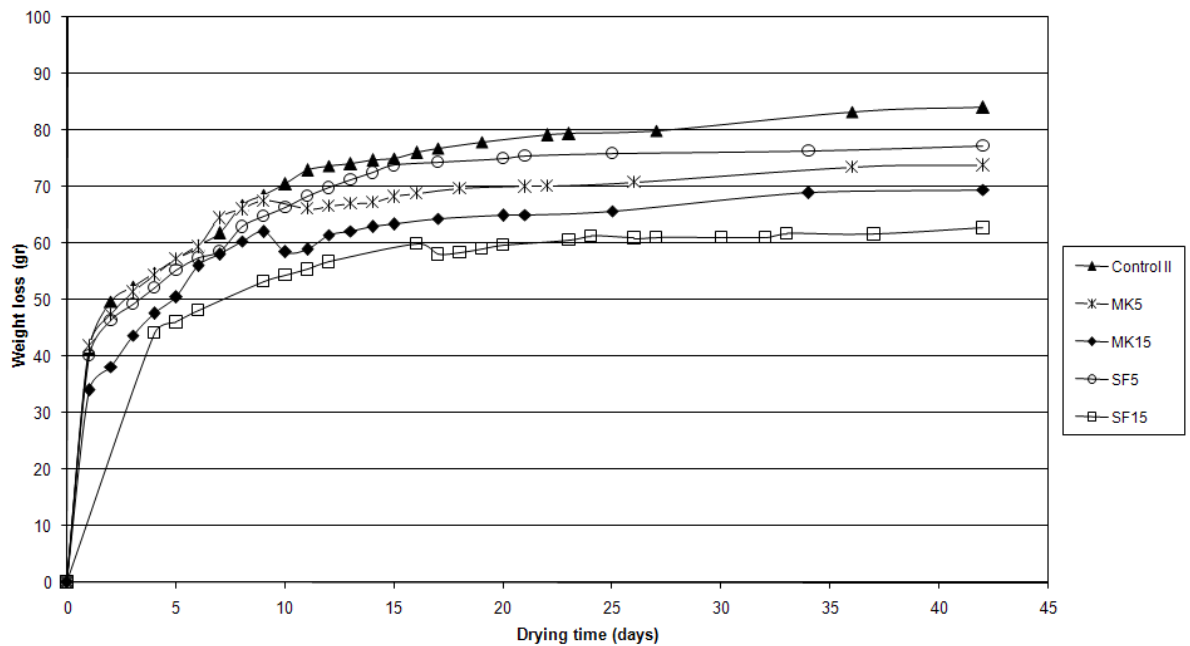


Figure 4.5 Weight loss vs. drying time graphs of plain, MK, and SF concretes having w/cm ratio of 0.35

#### 4.5. Restrained Shrinkage

Since drying shrinkage tests alone can not offer sufficient information on the behavior of concrete structures since virtually all concrete structures are restrained in some way, either by reinforcement or by the structure. Therefore, the observation of restrained shrinkage cracking behavior of concrete gains a prominent importance. The crack developments of the restrained shrinkage specimens produced by w/cm ratio of 0.35 were illustrated in Figure 4.7. As it can be seen from the figure that initial cracking of the specimen were observed at 7<sup>th</sup> day for control, MK5 and MK15 concrete, however, the crack initialization of SF5 and SF15 concrete was observed to occur at 8<sup>th</sup> and 9<sup>th</sup> days, respectively. Considering the overall drying period, the lowest crack propagation was observed in SF15 concrete, irrespective of the drying time. Nevertheless, MK15 concrete showed remarkable reduction in crack width at the end of 42 days, as well. Although MK5 concrete had higher crack width than plain concrete at early ages of crack propagation, a similar trend as in the drying shrinkage and weight loss was observed, nonetheless. Taking into consideration the

overall drying period, the more the amount of MK or SF used, the lower the restrained shrinkage crack widths were observed. The findings of the current study agree with the observations of Ding and Li (2002). They concluded that the incorporation of both MK and SF in concrete can reduce the free drying shrinkage and restrained shrinkage cracking width. They also reported that the initial cracking appeared earlier in the SF and MK modified concrete compared with the control concrete.

It is known that, cracking of concrete under restrained conditions is a major serviceability problem for field pavements and structures. The current study highlighted the utilization of MK and SF to provide additional performance in terms of reduction in restrained shrinkage cracking of concrete.

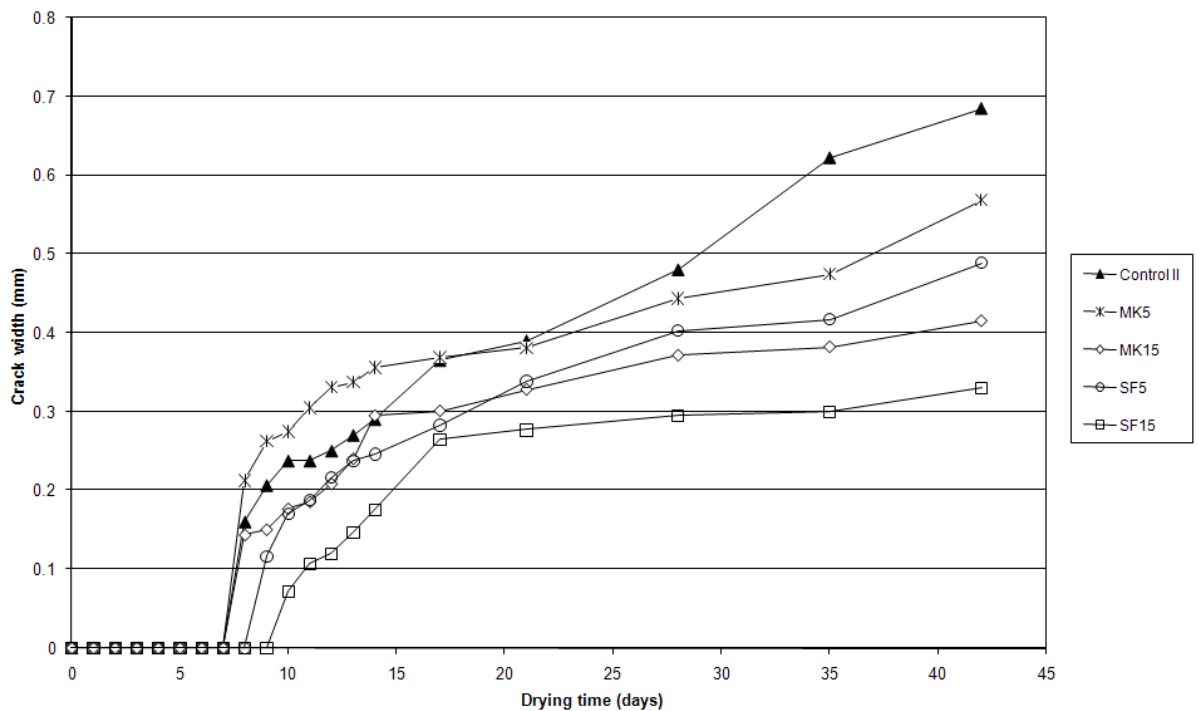


Figure 4.6 Restrainted shrinkage cracking of concretes produced with w/cm ratio of 0.35

#### 4.6. Gas Permeability

The coefficients of apparent oxygen gas permeability determined according to RILEM (1999) on completely dry specimens are presented in Figs. 4.7. The ranges



of permeability coefficients were found to be 0.97-2.04 ( $\times 10^{-16}$ )  $m^2$  and 1.32-3.45 ( $\times 10^{-16}$ )  $m^2$  for the concrete groups with w/cm ratios of 0.25 and 0.35, respectively, without depending on the type and amount of mineral admixture. As it can be seen from the figure the highest values in both of the concrete groups were observed at control concrete. The influence of MK addition was seem to be somehow more effective than SF in reduction of gas permeability coefficient. Figure 4.8 also clearly shows how effective the incorporation MK and SF in decreasing the gas permeability coefficient. Although reduction effects of MK and SF seem to be similar, there is an evident variation in effectiveness due to the w/cm ratio. For example, considering 15% replacement level, MK and SF provided 56 and 61% reduction at concrete group produced with w/cm ratio of 0.25. However, at concrete group of 0.35 w/cm ratio the percentages of decrease were measured as 52% for the former and 56% for the latter. The degree of change became lower when w/cm ratio increases.

Since the transport properties of concrete are strongly depending on its pore structure (Boel et al. 2008), reduction of gas permeability of concrete can be attributed to the refinement in pore structure of concrete due to MK or SF addition.

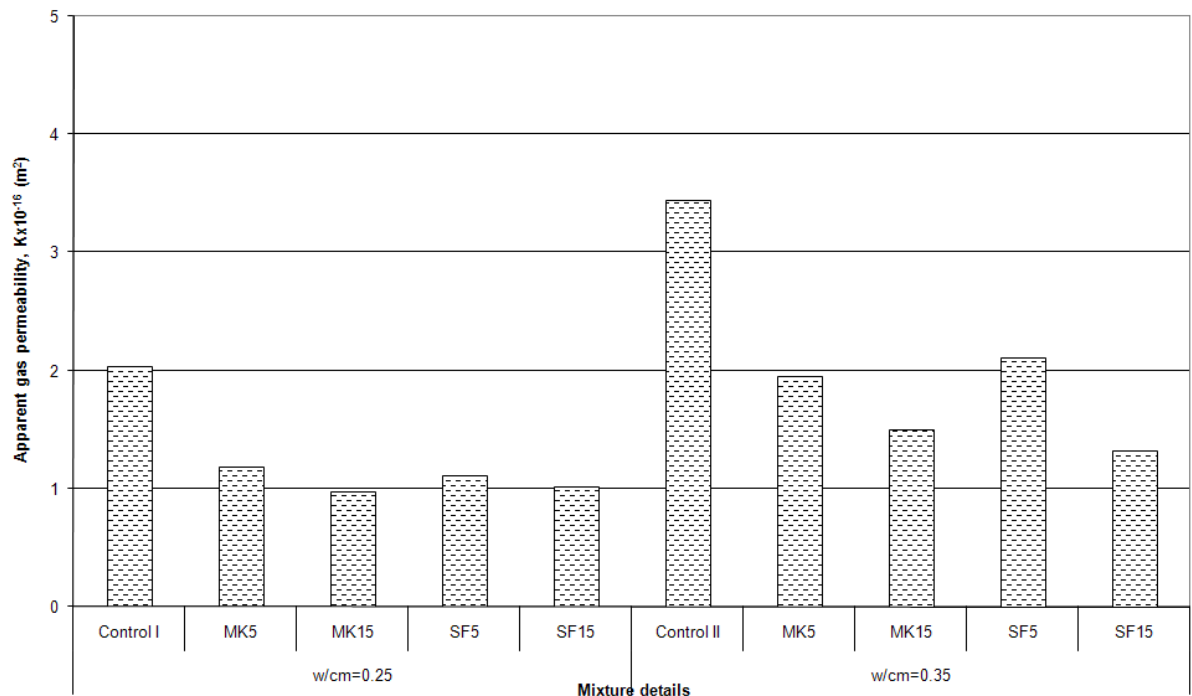


Figure 4.7 Variation of apparent gas permeability coefficients of the concrete with respect to w/cm ratio and composition of the concrete

Table 4.4 Gas permeability coefficients of the concrete with respect to w/cm ratio and composition of the concrete

w/cm ratio	Mixture details	Gas permeability coefficient, $K \times 10^{-16} (\text{m}^2)$
w/cm=0.25	Control I	2.04
	5MK	1.19
	15MK	0.97
	5SF	1.12
	15SF	1.01
w/cm=0.35	Control II	3.45
	5MK	1.95
	15MK	1.49
	5SF	2.11
	15SF	1.32

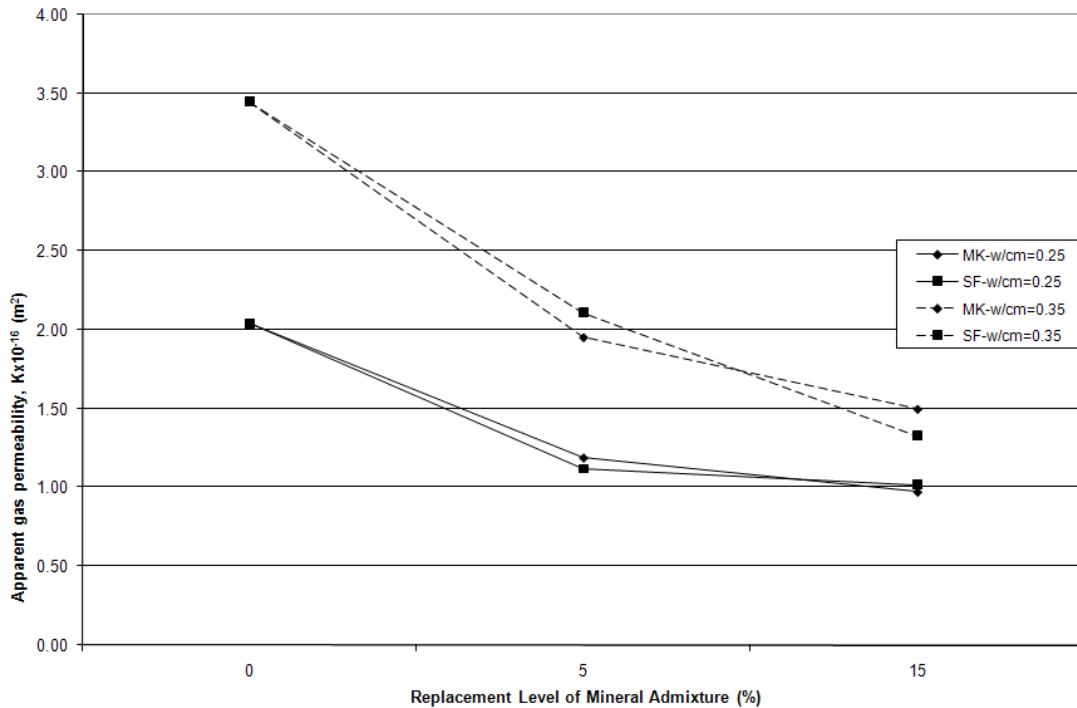
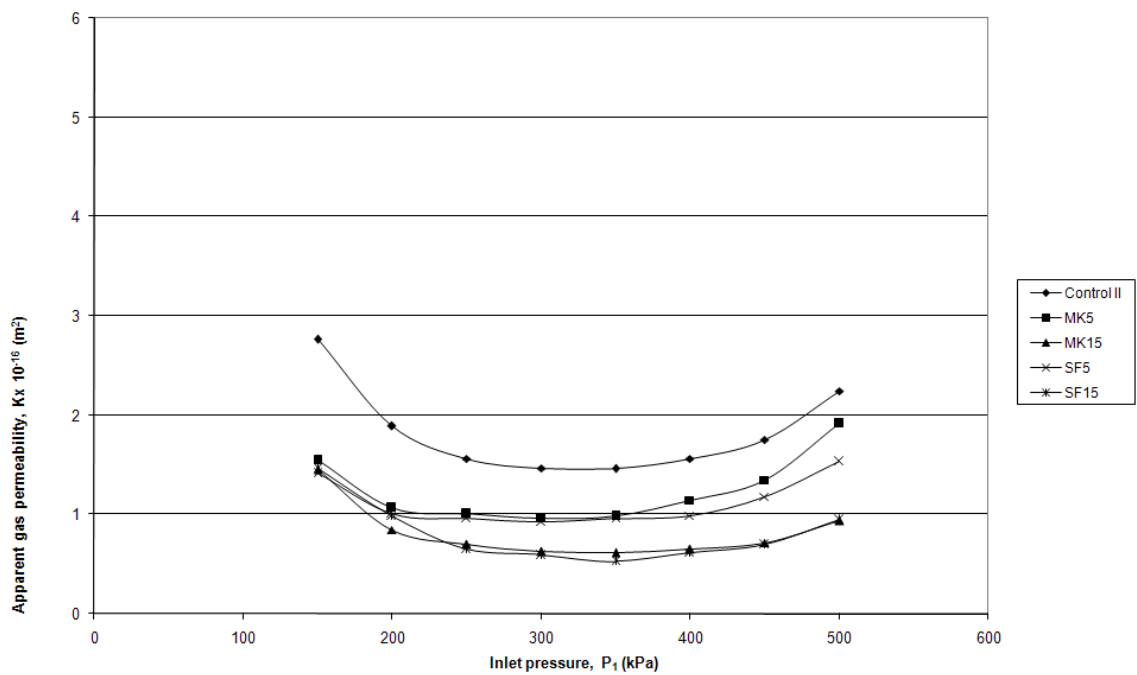


Figure 4.8. The change in replacement level and apparent gas permeability

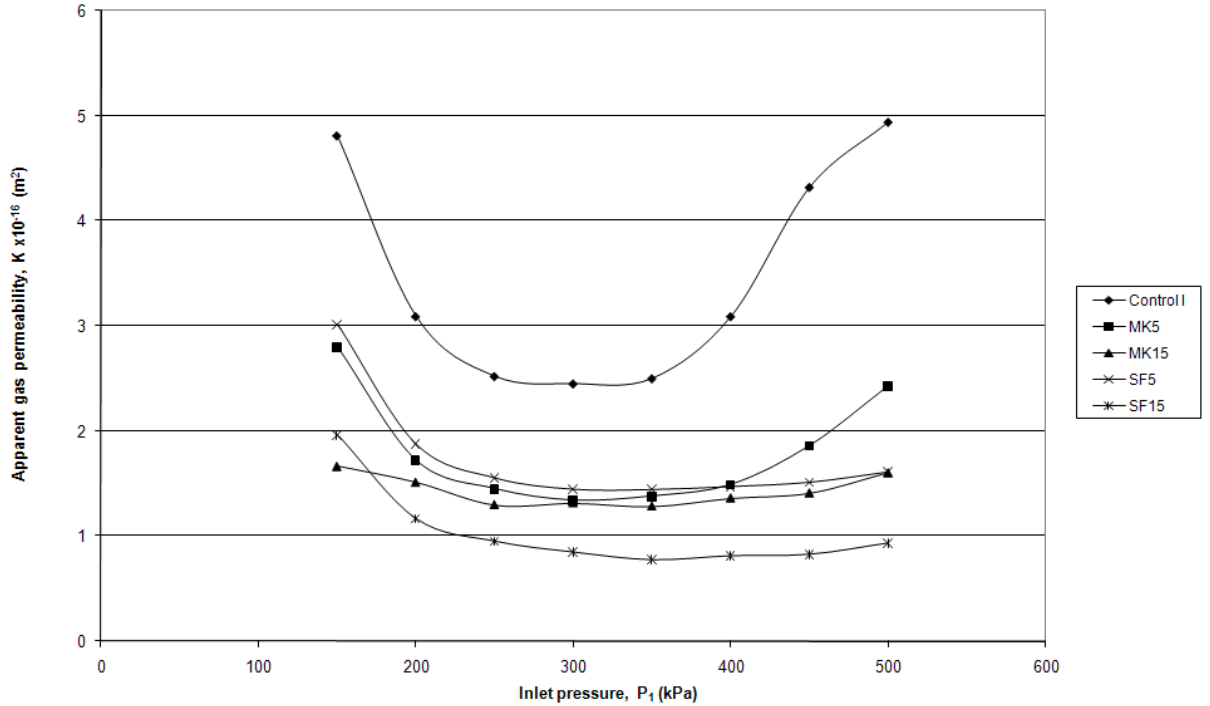
To evaluate the behavior of concrete according to the inlet pressure head, Figure 4.9 were illustrated. It can be observed from the figure that the gas permeability coefficient begins to rise after the inlet pressure of 350 kPa. The apparent gas permeability calculation was carried out on the basis of the Hagen-Poiseuille relationship for laminar flow of a compressible fluid through a porous body with

small capillaries under steady-state conditions. The inlet pressures greater than 300 kPa did not obey the inverse proportionality between  $P_1$  and apparent gas permeability. Maybe, for this reason RILEM (1999) proposes the use of 150, 200 and 300 kPa inlet pressures for determination of the gas permeability. At lower pressures the contribution of the slip flow increases, which causes increased apparent gas permeability (Boel et al. 2007). The higher apparent gas permeability values were observed at plain concrete at each level of inlet pressure, irrespective w/cm ratio.

Abbas et al. (1999) stated that permeability, along with diffusion and absorption, can be used to quantify durability characteristics of a concrete. In this study it was demonstrated that the concrete with MK and SF especially at replacement level of 15% showed better performance than plain concrete in terms of mechanical shrinkage and capillary water transport which were all compatible with gas permeability characteristics.



(a)



(b)

Figure 4.9 Apparent gas permeability plotted versus the change of inlet pressure for the concrete produced by w/cm ratios of a) 0.25 and b) 0.35

#### 4.7. Sorptivity

The 28 day sorptivity coefficients of concrete for different w/cm ratios, type and amount of mineral admixtures were given in Figure 4.4. Generally, MK and SF concrete performed better than the control concrete and marked improvements in terms of lower rate of water penetration through capillary suction were apparent. It is apparent that sorptivity decreases systematically with the decrease in w/cm ratio, and the gradients of the sorptivity tend to decrease with increase in the replacement level of MK and SF. Considering 28 days, it can be observed that there is an inverse proportionality between the sorptivity values and mechanical properties. The higher the mechanical property concrete has, the lower the sorptivity coefficient was observed. This is due to the pore refinement through filling and secondary hydration reaction of the mineral admixtures. Pore refinement results in lower capillarity and higher strength. Thus, the concrete with the lowest sorptivities have the highest strengths, especially for those having 15% MK or SF irrespective of the w/cm ratio.

Increasing the MK and SF content reduced the 28-day sorptivities of the concrete. This situation is more pronounced for the high w/cm ratio MK modified concrete. 15% MK incorporation provided 29% reduction in sorptivity values of both w/cm ratios, whereas, same level of SF replacement achieved 30% and 20% decrease in w/cm ratios of 0.25 and 0.35 respectively. Thus, it can be said that SF concrete are more vulnerable to change in w/cm ratio.

Reduced sorptivity reflects a finer pore structure that would, for example, inhibit ingress of aggressive elements into the pore system (Chindaprasirt et al. 2005). Taşdemir (2003) have also shown that type and fineness of mineral admixtures (such as fly ash, limestone filler, sandstone filler, and silica fume) together with curing condition affect greatly the pore structure of concrete and it was reported that a significant reduction in sorptivity for the blended mixtures was obtained when the proper curing was applied.

Table 4.5 Type and amount of mineral admixture on 28-days sorptivity coefficients of concrete

w/cm ratio	Mixture details	Sorptivity coefficients (mm/min <sup>1/2</sup> )
w/cm=0.25	Control I	0.0639
	MK5	0.0522
	MK15	0.0464
	SF5	0.0603
	SF15	0.0448
w/cm=0.35	Control II	0.0811
	MK5	0.0736
	MK15	0.0585
	SF5	0.0752
	SF15	0.0652

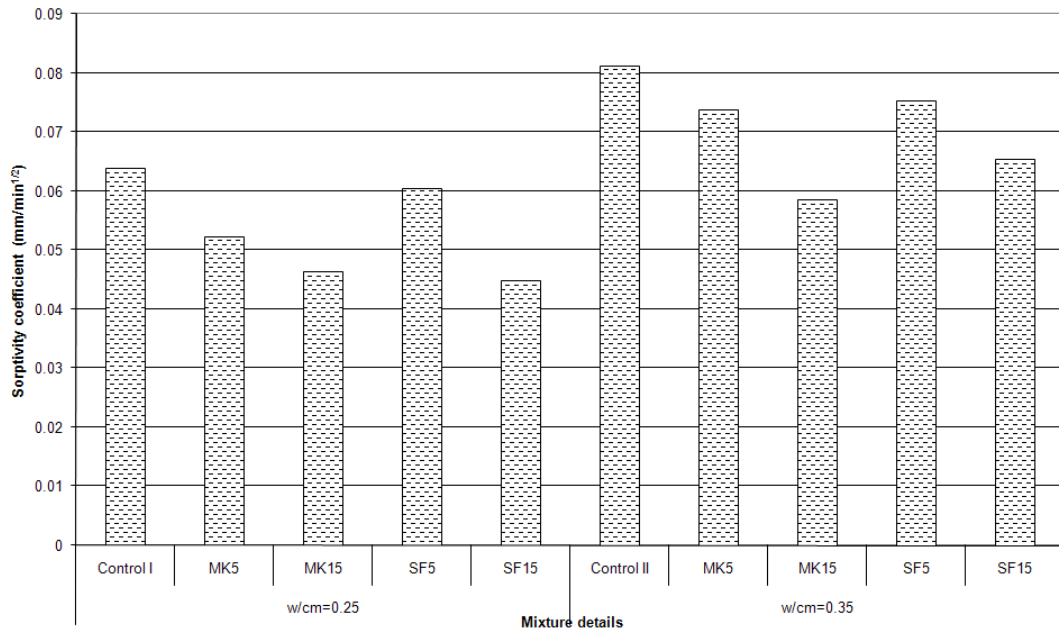


Figure 4.10. Influence of w/cm ratio, type, and amount of mineral admixture on 28-days sorptivity coefficients of concretes

## CHAPTER 5

### CONCLUSION

In this thesis, the effectiveness of metakaolin and silica fume on the hardened properties of high performance concrete was investigated. Based on the results obtained from the experimental study, the following conclusions can be drawn:

- This study specified that the SF and MK concrete had consistently higher compressive strength than the control concrete. There was a systematic increase in compressive strength with the increase in MK and SF content for both w/cm ratios. In both w/cm ratios, SF and MK concretes showed higher rate of compressive strength development than that of plain concrete.
- According to the test results obtained in this study it was proved that the splitting tensile strength development of the concretes had similar tendency with compressive strength. However the differences between plain and blended concretes were not as high as compressive strength. At the concretes having w/cm ratios of 0.25 and 0.35, the maximum 28-day splitting tensile strength were measured at the concretes contained 15% SF and MK, respectively.
- Elasticity modulus increases with increase in MK and SF replacement, but the rate of increase is less than that for the compressive strength. It has rather a similar trend with splitting tensile strength development. MK and SF addition had an important contribution to the development of the modulus of elasticity of concretes. Utilizations of these materials provide an improvement in early age mechanical properties.
- For all replacement levels, MK and SF modified concretes exhibited lower shrinkage in comparison to the plain concretes. Nonetheless the lowest

shrinkage strains were measured at the concrete containing 15% SF. It is known that the drying shrinkage is influenced by many factors. The results demonstrated that the inclusion of mineral admixture had a prominent effect in reduction of drying shrinkage of concretes. The drying shrinkage rates of the concretes had a decreasing tendency with passing drying time, particularly for the MK and SF concretes.

- Addition of the MK and SF decreased the rate of weight loss due to the drying of the concretes. Especially for the concretes having higher rate of replacement levels, the weight losses observed to be relatively lower than that of plain concrete.
- Incorporation of MK and SF was proved to be effective in improvement of the pore structure of the concretes. Gas permeability and sorptivity tests reflect the permeability characteristics of the concretes. The lower the permeability of the concretes, the higher the enhanced durability can be attained. Especially, the concrete produced with low w/cm ratio had the lowest sorptivity and apparent gas permeability coefficients. Moreover, the concretes with MK or SF had relatively lower gas permeability coefficients in comparison to plain concretes for both w/cm ratios.



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