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# Sulfuric acid resistance of fly ash mortar

Walairat Bumrongjaroen  
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## **ABSTRACT**

### **SULFURIC ACID RESISTANCE OF FLY ASH MORTAR**

**by**  
**Walairat Bumrongjaroen**

Crown corrosion of concrete pipe and its subsequent repair is a topic much discussed in recent years. Present protective methods have not worked effectively. Due to its properties and its low cost, fly ash was used as a cement replacement of mortar to investigate its sulfuric acid resistance.

Different types and different percentages of fly ashes were mixed with and without admixtures including microsilica and superplasticizer. The main parameters investigated included the chemical compounds, the fineness and the volume of fly ash. 2"x2" cubes were immersed in a 10% sulfuric acid pond. The weight of each cube was determined continuously up to 28 days. The results indicated that fly ash can be used effectively to improve sulfuric acid resistance of mortar in term of weight loss when using the finest fly ash in the optimum percentage, 50%. However the strength of these samples under the same conditions was significantly deteriorated. Microsilica and superplasticizer cannot inhibit the strength deterioration when mixed with fly ash mortar.

**SULFURIC ACID RESISTANCE OF FLY ASH MORTAR**

by  
**Walairat Bumrongjaroen**

**A Thesis  
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New Jersey Institute of Technology  
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**Department of Civil and Environmental Engineering**

**January 1994**

**APPROVAL PAGE**

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**The author dedicates this thesis to her  
parents for their constant inspiration.**



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# CHAPTER 1

## INTRODUCTION

### 1.1 Statement of Problems

In recent years, it has been recognized that there are great damages on sewage pipe caused by crown corrosion. This problem occurs all over the world but no effective protection is developed. This paper studies on a method to improve the acid resistance of mortar. Since fly ash possesses pozzolanic behavior which can enhance the properties of concrete, it is the potential material to enhance the acid resistance of mortar.

To properly design a durable mortar structure requires a thorough understanding on the corrosive process of concrete. Many articles are consistent that this corrosion causes by sulfuric acid. This corrosive acid is generated by anaerobic bacteria which covers on surface concrete. When concrete is in contact with such acidic solution, the calcium hydroxide reacts with the sulfuric acid to form gypsum, which can be readily washed away. More over, this solution is particular corrosive due to the hydrogen ion attack which can corrode any compounds in concrete.

To improve sulfuric resistance of concrete, many methods have been suggested. In general, the durable concrete must be dense and have low permeability. Such low permeability concrete can be obtained by using fine particle admixture or lowering water:cement ratio of the mixes. Other methods suggested to improve the quality of concrete are the use of polymer materials as additive, sulfate-resisting cement, high alumina cement or pozzolanic materials, etc. These potential solutions can only be feasible depending largely on their cost.

The present study investigated on sulfuric acid resistance of fly ash mortar. It

is found that the acid resistance is contributed by both of its physical and chemical properties. Physical property of fly ash provides acid resistance mostly in initial phase. This very fine particles of fly ashes fill up the voids between cement and aggregate. While chemical property, pozzolanic action, contributes acid resistance in later phase. The silica content in the fly ash reacts with free lime or calcium hydroxide generated from the hydration process of cement to form calcium silicate hydrate compound. The gel helps filling up the remaining air voids in between fine aggregates and cement particles, making concrete denser, more impermeable and durable.

The results of this study indicate that concrete with a large volume percentage of fly ash as cement replacement exhibits excellent resistance to sulfuric acid attack. It is founded that the fly ash with 50% as cement replacement provides the optimum sulfuric resistance but its compressive strength is still very low. Thus, microsilica and superplasticizer are used to improve this durability. The result shows that microsilica can enhance the initial compressive strength and sulfuric acid resistance but it cannot reduces the strength reduction of fly ash mortar immersed in sulfuric acid. And superplasticizer gives the adverse effect as it provides higher deterioration than normal cement mortar. However, this present study has proposed the important key that leads to the new material which has high sulfuric acid resistance. These findings are major breakthroughs which have never been reported elsewhere.

In addition, strength contribution of fly ash is investigated here. It is founded that the higher percentage of fly ash is used as cement replacement, the lower it contributes compressive strength to the mix. It is also noted that for each fly ash percentage used, the higher the age of the mix is, the more compressive strength the fly ash provides. Details of all the investigations mentioned above are further elaborated in this thesis.

## 1.2 Objectives

The purpose of this study is to study sulfuric acid resistance of fly ash mortar. Different types of fly ashes which are known physical and chemical characteristics are introduced as pozzolans into mortar to investigate the effect of those characteristics. Different percentages of the finest fractionated fly ash were mixed to find the optimum mix which gives the highest acid resistance. In addition, microsilica and superplasticizer were added into the fly ash mortar to investigate the corrosion and strength effect. They were immersed into a concentrated sulfuric acid solution to evaluate for their resistance to acid attack. The strength and weight losses due to acid attack will be monitored. The results should provide a definite rate of degradation and the quantity of fly ash needed to maintain the strength and integrity of the fly ash concrete. Also, the strength contribution of fly ash is studied.

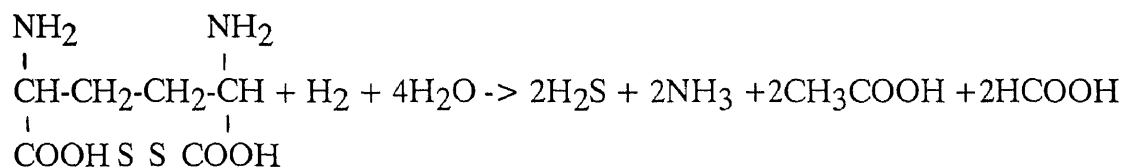
## CHAPTER 2

### LITERATURE SURVEY

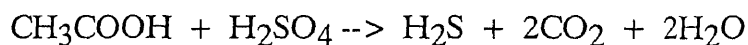
#### 2.1 Crown Corrosion in Sewer System

Many papers have been published dealing with the subject of crown corrosion of sewer pipe. They all reported that concrete is corroded by sulfuric acid caused by sulfur bacteria. The process forming sulfuric acid is divided into two steps; H<sub>2</sub>S production and H<sub>2</sub>SO<sub>4</sub> generation.

H<sub>2</sub>S production is the anaerobic processes occurring in wastewater. Certain bacteria obtain the energy needed for their metabolism from the oxidation of sulfur in this process then produce hydrogen sulfide as a waste product. There are two sources of sulfur in sewage. One is derivative from hydrolysis of protein. Bacteria, including *E.Coli*, *Proteus Valgaris* and *Pseudomonas pycocyanca*, reduces these compounds and produces H<sub>2</sub>S according to the following equation.



The other source, which provides most of sulfur in sewage is sulfate compound. It is oxidized to sulfide by anaerobic bacteria by the following equation:



Not only H<sub>2</sub>S is generated from sewage water but it can be generated in natural waters (Thornton 1978). This phenomena was found in the deterioration of

concrete pipes through a dam. The investigator concluded that this bacteria living in anoxic region oxidized sulfate discharging from nearby plant to produce  $H_2S$ . This  $H_2S$ -rich water was siphoned into the dam then  $H_2S$  was emitted to air in the concrete pipe. It caused severe corrosion that was difficult to remedial.

Presently, the crown corrosion problem has become more topical because of environmental concerns. EPA has mandated law that affects  $H_2S$  generation. One striking problem results from water conservation program including the installation of low-flow plumbing devices. It decreases flow rate significantly and increases Biochemical Oxygen Demand (BOD). Reducing flow lets organic matter settle to the bottom of the pipe. Increasing the BOD lowers dissolved oxygen and facilitates the generation of anaerobic bacteria. These two conditions are enough to enhance corrosion rate in sewer pipe.

Hersch (1989) found problem caused by heavy metal control act. Generally, wastewater from treatment plant has some amounts of heavy metal which can kill bacteria. But the new environmental law mandates the removal of all heavy metal from the wastewater before discharged to the sewers. Therefore, the natural protection of sewer pipe does not occur resulting in considerable corrosion in concrete pipe.

$H_2S$  emission is also important. There can be considerable  $H_2S$  produced in the sewage but no crown corrosion will occur if the  $H_2S$  is not emitted to air in the pipe. There are three factors influencing the rate of emission; the thickness of laminar layer between air and sewage, presence of floatation on surface, and presence of  $H_2S$  in sewage (Parker 1951). Moreover, Kienows (1991) and Sand and Bock (1984) found corrosion in turbulence areas such as rising mains, outlets, and manholes. They concluded that the turbulence helped the  $H_2S$  escaped from the sewage more easily than stagnant water. This gas arises from wastewater and covers the crown of concrete.

Anaerobic bacteria on concrete surface oxidizes  $\text{H}_2\text{S}$  and change it to be  $\text{H}_2\text{SO}_4$ . This reaction needs not only available sulfur but also needs proper environment and nutrient. Moisture is also an important factor of this reaction. In high relative humidity (RH) the rate of corrosion is higher than the rate of corrosion in low RH (Islander, Devanny, Mansfeld, Postyn, and Shih 1991).

Oxidation reaction can be divided into three pathways. First, abiotic neutralization occurs in pH from 12 to 9. It is a carbonation process that  $\text{CO}_2$  in the air reacts with  $\text{H}_2\text{O}$  to produce  $\text{H}_2\text{CO}_3$  and  $\text{HCO}_3^-$ . These products neutralize  $\text{Ca}(\text{OH})_2$  and forms  $\text{CaCO}_3$  at the concrete surface. Concurrently,  $\text{H}_2\text{S}$  is reduced to  $\text{S}_2\text{O}_3^{2-}$  and  $\text{S}_4\text{O}_6^{2-}$ . At the end, pH of surface concrete drops to 9.

Second pathway, thiosulfate oxidation, takes place from pH 9 to 5 by two kinds of bacteria. *Thiobacillus thioparus* consumes  $\text{S}_2\text{O}_3^{2-}$  and produces  $\text{S}_4\text{O}_6^{2-}$ . And *Thiobacillus neapolitanus* uses those products to produce sulfur. At the end of this pathway, pH of the concrete surface is about 5.

Third pathway, sulfur oxidation by *Thiobacillus concretivorans*, takes place between pH 5 to 1. It reduces sulfur to sulfate,  $\text{H}_2\text{SO}_4$ . This end product is a corrosive substance that destroys the surface concrete in the highest rate. Generally, the surface of destructive concrete is dominant by this bacteria. Sand and Bock (1984) measured the corrosion rate by counting *Thiobacillus concretivorans* concentration. They found that this method is more precise than simply measuring pH.

$\text{H}_2\text{SO}_4$  which is generated from the oxidation reaction reacts with  $\text{Ca}(\text{OH})_2$  of concrete. The products of this reaction are  $\text{CaSO}_4$ ,  $\text{CaCO}_3$ , and  $\text{CaO}$  that have no strength. They do, however, act as barriers slowing the penetration of  $\text{H}_2\text{SO}_4$  into concrete and also slowing the leaching of  $\text{Ca}(\text{OH})_2$  out of concrete. If these products drop off from surface concrete, they will reveal the new concrete surface that is vulnerable to acid attack. The whole process repeats again and again until the



concrete pipe completely collapses

## **2.2 Protective Measures Against Crown Corrosion**

Many technical and economical protective measures against crown corrosion have been proposed. Each method can be applied correctly by the careful consideration of the particular set of factors and conditions. They are summarized into four forms; prevention of hydrogen sulfide emission, prevention of hydrogen sulfide condensation, using acid resistant concrete, and coating concrete surface by special material.

Normal sewage contains enough sulfur compounds to produce hydrogen sulfide. Chemical and physical methods have been used to prevent it from emission. For chemical, chloride has been used successfully to prevent sulfide production. The addition of lime to raise the pH of the sewage to above 10 inhibits the sulfide production. Salts of copper, zinc, ferrous ion can also be added to form insoluble sulfides but this option is only occasionally used due to its cost. Removal of slimes and silt accumulations, reduction of detention time by increasing velocities, and the avoidance of points of turbulence are physical measures found useful for preventing H<sub>2</sub>S evolution.

Prevention of hydrogen sulfide condensation can be accomplished by two methods. First method is using forced air ventilation of sewer pipe. It dries the exposed surface of concrete and prevents absorption of hydrogen sulfide. It also introduces additional oxygen to reduce the possibility of developing anaerobic condition feasible to H<sub>2</sub>S production. Another method is flushing sewer pipe periodically. Islander, Devanny, Mansfeld, Postyn, and Shih (1991) successfully used flushing of the crown by sewage periodically to wash away the acid and change the ecological balance among the microorganisms.

Considerable attentions have been given to the testing of constructions

resistant to sulfuric acid. One research found that no cement type offers sufficient corrosion resistance except through the use of limestone aggregates. It has been found to reduce the rate of sulfuric acid attack three to five times compared to portland cement concretes made with siliceous aggregates. That is because the aggregate itself is dissolved and thus assists in the neutralization of the acid. It stops acid attack on cement binder (Biczok 1967, Stutterheim 1953, Huges and Guest 1978). Harrison (1987) and Huges and Guest (1978) stated that using limestone aggregate should be considered in some circumstances such as periodic acid attack or a very dilute acid attack. Fattuhi and Huges (1988) suggested that limestone could be used as an admixture in environments with sulfuric acid concentration less than 0.02%. In this research, aggregate is not taken as a variable since no concrete will be studied.

Attogbe and Rizkalla tried to limit this compound by using sulfate resisting cement. The result turned out less expansive in the first period since no  $C_3A$  reacted with gypsum. But in the long run the corrosion still occurred because the hydrogen ion reacted with free lime and CSH gel.

Use of high alumina cement (HAC) has also been found to give a better resistance to sewer corrosion but attack still occurs. HAC is prohibited for use in some countries due to the use of high water cement ratio causing failure of the lumber of structural units (Perkins 1977, George 1975). The supersulfate cement has been considered instead. Its corrosive resistance is at least comparable to that of HVC.

Recently, the most acceptable method for the prevention crown corrosion is the ocrate process which uses  $SiF_4$  gas. This cement consists of water glass, sodium silicofluoride, active silicic acid, and acid-resisting aggregate (quartzite). For brickwork in manholes acid resistant cements of the silicofluoride-silicate-filler type have long been successfully used. The disadvantage of this cement is its vulnerability

to bases and fats (Biczok 1967).

Many trials have been made on protective treatments to concrete. Coating by inert filler is one of protection methods. These materials are acid-resisting mortars, tar-based paint, resin lacquers and etc.. Even though they are widely used but they still have some disadvantages. Since these material may crack, it will let corrosive substance to corrode concrete inside.

Each method has particular application. Sometimes, various methods can be combined. This research emphasizes in passive method by developing high corrosion resistant mortar. If it performs well, it could be used to repair the damaged surfaces of concrete sewer pipe and perhaps, it could be made into concrete pipes.

### **2.3 Mechanism of Sulfuric Acid Attack on Concrete**

To improve corrosion resistance of concrete, the mechanism of corrosion should be thoroughly investigated. This section will classify the corrosion, describe the corrosion process, identify the affected zones of sewer and pinpoint the vulnerable parts of sewer system.

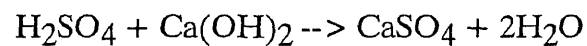
Corrosion on concrete has been classified into five categories by Plum and Hammersley (1984).

1. Leaching of free lime.
2. Aggressive chemical reacts with cement compound and forms compounds that may leach from or deposited in non binding form.
3. Same reaction as type two but the secondary product is the crystal compound that builds the internal stress resulting in gradual disruption.
4. Similar reaction as type two but the first product, crystallization of salt, causes disruption instead of secondary product.
5. Corrosion of steel reinforcement.

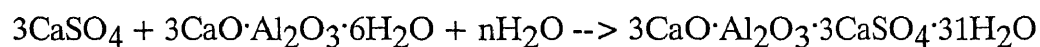
Practically, more than one type of attack occur since there are many chemicals involved in the reaction. In this paper, sulfuric acid is the concerning corrosive solution because it is the only chemical formed on the concrete surface.

Sulfuric acid is particularly corrosive since it has two corrosive ions,  $\text{SO}_4^{2-}$  and  $\text{H}^+$ . Numerous researchers have shown that its presence produces additional dissolution of hydrogen ion with a commensurate drop in pH (Attigbe and Rizkalla 1988, Mindess, Sydney and Young 1981, and Wenger 1958).

Sulfate attack can be classified as type 3 since its second product destroys concrete. Sulfate ion reacts with free lime to form gypsum by the following equation.



Then, gypsum reacts with calcium aluminate hydrate to form ettringite. This product is very expansive. It produces high internal stresses resulting in spalling, cracking and general strength loss of concrete strength. The following equation presents this reaction.



Another corrosive ion is  $\text{H}^+$ . Its process is classified as type 2.  $\text{H}^+$  reacts with all hydration products which are binding materials of concrete.,  $\text{Ca}(\text{OH})_2$ , CSH-I, CSH-II and so on. The products of this reaction are nonbinding materials which can leach out easily. As a result, the corrosion caused from  $\text{H}^+$  is severe and rapid. Nevill (1983) reported that, within the pH range of 3 to 6, deterioration progresses at a rate approximately proportional to the square root of time.

To investigate corrosion site, Attigbe and Rizkalla (1988) used a Scanning Electron Microscope (SEM) photographing reaction products. Their results showed

that the deterioration progressed inward and the corrosion rate increased along the penetration depth. Plum and Hammersley (1984) enumerated damage zones from surface layer to a study point as follows.

1. Exposed cement paste completely disintegrated.
2. Calcium hydroxide depleted and gypsum deposited.
3. Ettringite formed.
4. Unattack zone, rich of free lime.

This information hints out chemical and physical ways to inhibit acid attack. For chemical, methods used to inhibit acid attack are lowering major vulnerable compound,  $\text{Ca}(\text{OH})_2$ , using an admixture to bind free lime, lowering cement content, using limestone aggregate and so on. Physically, acid resistant coatings and the minimizing of concrete porosity are also proposed.

The most vulnerable compound in concrete is free lime. Results from previous researches agreed that the deterioration rate will decrease as the cement content decreases (Fattuhi and Huges 1988). Many attempts were made to lower the cement content. Fattuhi and Huges (1988) minimized the cement content by using high range water reducing agents. Since other durabilities tended to be lower, additional admixtures were introduced to compensate.

#### **2.4 Use of Fly Ash to Improve Sulfuric Acid Resistance of Mortar**

Pozzolan, as defined by ASTM C-593 (1990), is a high siliceous or aluminous compound which, in finely divided form, chemically reacts with alkali and alkaline earth hydroxide to form or assist to form cementitious compounds. It is an potential material to be used for corrosive resistance because of its chemical and physical property.

For chemical, it is believed that pozzolanic compound of fly ash will tie up free lime resulting in no vulnerable product to react with  $\text{SO}_4^{2-}$ . This pozzolanic

action has been investigated by many scientists.

Cao, Ho, and Guirguis (1990) observed that there was a two layered shell of hydration product around fly ash. At first, the fly ash particle acted as nucleation of hydration. As a result, the hydration products, free lime or CSH, covered the fly ash as an outer shell. The pozzolanic action between hydration products and siliceous or aluminous compounds of fly ash occurred later and formed an inner shell. Alexander, Wardlaw and Ivanusec (1984) reported that there were two stages in the pozzolanic process. The first stage was associated with bond formation and the second, with the formation of cementitious compounds.

However, this advantage of fly ash will work if it has high reaction rate. If it can tie up free lime before sulfate does, it can inhibit the corrosion. This rate can be measured by determining amount of free lime in the mix. Tazawa, Yonekura, Kawai, Kohata, and Teramoto (1992) observed this amount in mortar with fly ash, silica fume, meta kaolin and neofume. They found that pozzolanic reaction of fly ash seemed hardly to occur up to about 21 days. On the other hand, no change of  $\text{Ca(OH)}_2$  of the mix occurred. Meanwhile, the products of calcium hydroxide in silica fume, meta kaolin and neofume mortar decreased with time between 7 to 14 days. This can be explained by the property of silicon dioxide. Generic fly ash has siliceous in crystalline form which is not ready to react. But silica fume, meta kaolin and neofume have noncrystalline silicon dioxide.

Berry and Malhotra (1980) stated another evidence that the reaction rate between siliceous or aluminous compound with calcium hydroxide was very much slower than that of portland cement. Also, Fattuhi and Huges (1988) reported that as the age of specimen increased, the deterioration was reduced. Thus, the pozzolanic action is believed to occur at later age.

Another important factor affecting the rate of pozzolanic activity is the fly ash cement ratio. Gopalan (1993) reported that the time needed for the completion of

the pozzolanic reaction in fly ash concrete depended on this ratio. He stated that if high volume of fly ash was used, the rate of pozzolanic activity decreased. This research will use several ratios of fly ash to study its affect on the acid resistant property of fly ash mortar.

Although the pozzolanic reaction in fly ash concrete or mortar occurs later, previous studies have shown superior sulfate resistance even at early age. Therefore, there must be other protection mechanisms to account for this.

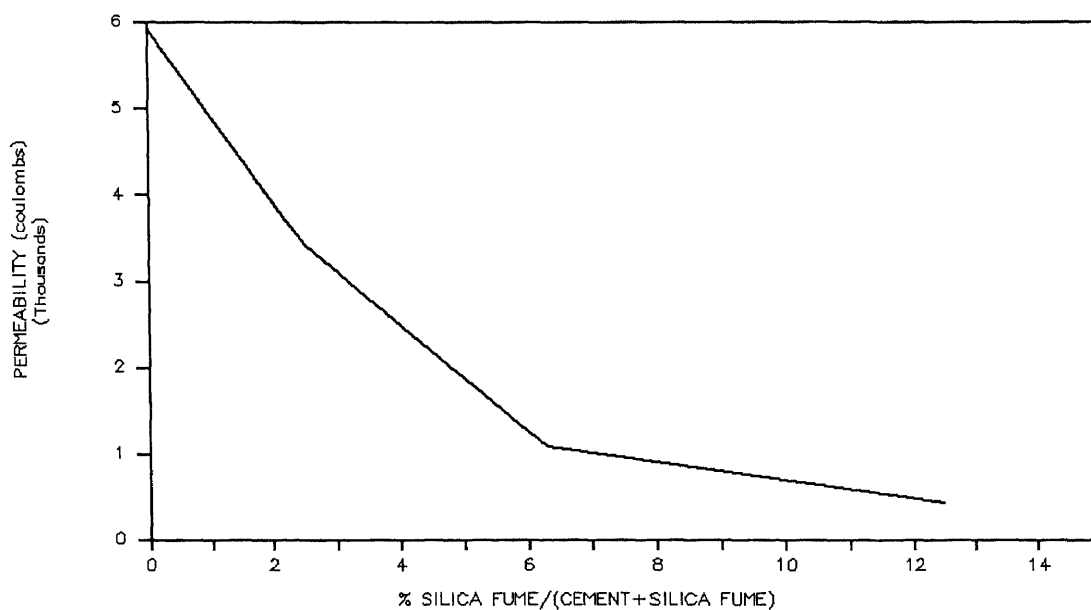
Most recent researches explained that the corrosive resistant property of fly ash concrete or mortar mostly resulted from its physical role. Marusin (1992) found that the concrete containing class F fly ash were less permeable than portland cement concrete. Sheu, Quo, and Kuo (1990) studied the corrosion resistant property of fly ash mortar with different particle sizes of fly ash in sodium sulfate solution. They concluded that the mortar with finer particle size of fly ash had greater resistance to sulfate attack than the control sample. Furthermore, the previous studies indicated that the fine particles of fly ash closed the pore space between aggregates and cement products. Therefore this research will study the role of fineness in the corrosion resistant of mortar by comparing mortar made with the finest particle fly ash and the original feed fly ash.

Many researchers reported the highly effective sulfate resistant property of fly ash mortar and concrete (Erdogan, Tokyay, and Ramyar 1992 and Freeman and Carrasquillo 1992) while no research used fly ash to improve acid resistance. Nassar and Lai (1990) and Irassar and Batic (1989) suggested that class F fly ash would be a good source of pozzolan which can improve resistance of concrete to sulfuric acid attack. Fattuhi and Huges (1988) used pulverized fuel ash to replace cement in concrete subjected to sulfuric acid. Their attempt was not successful nor were other researchers that used pozzolan to prevent chemical attack (Halstead 1954, Miller and Manson, and Fattuhi and Huges 1983).

More questions have been raised about its durabilities; strength, corrosion resistance and so on. The weakness of using fly ash in concrete is its low strength at early age. Additionally, if fly ash mortar can resist sulfate, can it resist acid?. The hydrogen ion may react with other compounds since it is the most corrosive ion. Thus, this research will investigate more on acid resistance behavior of fly ash mortar.

### 2.5 Use of Microsilica to Improve Sulfuric Acid Resistance and Compressive Strength of Fly Ash Mortar

Another material which can be used to replace cement is microsilica. This corrosion resistance of microsilica is affected by its physical and chemical microstructures. In term of physical protection, Bentur and Cohen (1987) reported that it reduced porosity by filling the gap between cement paste and aggregate. It can be seen from Figure 2.1 that the permeability of microsilica mortar decreases as microsilica percentage increases.



**Figure 2.1** Rapid Chloride Permeability Test Results of Silica Fume Concrete Obtained from Berke, Neal S., Pgeifer, Donald W., and Weil, T.G., 1988.



In term of chemical protection, Bache (1981) stated that microsilica appeared to be effective for corrosion resistant once due to its fine particles and highly active, non crystalline form. This high reactivity is also effective in filling the space between cement particles. Berke, Scali, Regan, and Shen (1992) investigated the presence of unreacted microsilica of 10% microsilica concrete and found that it was used up within 180 days.. Meanwhile, a considerable number of fly ash remain unreacted even after three years. In a study where silica fume and fly ash were used, it was concluded that the effect of fly ash on concrete permeability was overshadowed by the effect of silica fume (Berk, N.S., Pfeifer, D.W., and Weil, T.G., 1988).

But the potential benefits of using high percentages of silica fume is limited by the adverse effects of silica fume on fresh concrete. It decreases workability, placeability, flowability and finishability and it increases cohesiveness and stickiness. Thus, some engineering standards limit silica fume content to 10 percent by mass of cementitious materials. Moreover, Fattuhi and Huges (1986) reported that microsilica concrete had unacceptable weight loss if subjected to a sulfuric acid solution. In this study, microsilica will be mixed with fly ash mortar as a replacement to improve initial strength and sulfuric acid resistance of fly ash mortar.

## **2.6 Use of Admixture to Improve Sulfuric Acid Resistance of Mortar**

Different kinds of admixtures have been used to improve acid resistance (Fattuhi and Huges 1983, Fattuhi and Huges 1986, Fattuhi and Huges 1988). Some of them reacted with vulnerable constituents resulting in a reduction in the deterioration rate. For example, one superplasticizer consumed  $C_3A$  resulting in less vulnerable compound to  $SO_4^{2-}$  left in the mix and less deterioration occurred. Styrene butadiene latex and polyvinyl alcohol (PVA) have been used successfully as admixtures. PVA gave low rate of deterioration by keeping the matrix together but it resulted in high expansion.

Generally, admixture is used to minimize porosity of concrete or mortar. Addition to lowering the cement content, it reduces air voids caused by evaporation of water from concrete. These admixtures are considered to be used not only because of fewer air voids but also because it provides a more uniform, well compacted, and denser concrete or mortar (Wider and Spears 1981).

However, some of admixtures cannot be used because of their reaction with acid as it can be seen from many articles. Thus, care should be taken before using each kind of admixture. Superplasticizer is added to the fly ash mortar to investigate the acid resistance and to enhance initial strength of fly ash mortar.

Fully effective method for prevention of sulfuric acid attack has not been proposed to date. Brown and Baker (1979), Debuy (1975), Raju (1987) and Seki (1975) suggested equations to estimate the design life of concrete instead of developing means to resist sulfuric acid attack.

## CHAPTER 3

### MATERIALS AND EXPERIMENTAL METHODS

#### 3.1 Experimental Program

In this chapter, the experimental programs for studying the influence of parameters affecting the corrosion resistant of acid. Percentage of fly ash, type of fly ash, and admixture were selected as main parameters. The study also includes the investigation on the corrosion resistance of the fractionated fly ash mortar.

The standard ASTM 2"x2"x2" cube specimens for study the corrosion resistant and compressive strengths of mortar were used, respectively. All compressive strength tests were performed on a MTS closed-loop servo hydraulic testing machine. Details of these test programs are planned as follows:

#### 3.2 Materials

Materials used in this study consist of standard portland cement type I, siliceous sand (river sand), fly ash, silica fume, superplasticizer, sulfuric acid, and water.

**Cement**-A standard portland cement type I.

**Sand**-Local siliceous sand (river sand) passing through sieve No. 4 (opening size 4.75 mm) was used for casting mortar.

**Fly Ash**-Three different kinds of dry fly ash were selected in this experiment:

- 1) Finest fractionated dry bottom fly ash from Hudson, US.
- 2) Original feed dry bottom fly ash from Hudson, US.
- 3) Original feed dry bottom fly ash from Mae Moh, Thailand

All fly ashes were generated from power plants. Dry fly ash is the type of fly ash coming out from the precipitators and is usually stored in the hopper or in the silo for immediate delivery if the demand exists. All fly ashes used are classified as

Class F, low calcium, fly ash according to ASTM C-618 (1990).

**Silica Fume**-Silica fume is a by-product from the silicon metal industry. It often comes in very fine particle of size less than 1 micron. Normally consists of 96-98% of reactive SiO<sub>2</sub>. Silica fume used in this study is in powder form. The addition of silica fume was intended to produce high strength concrete.

**Superplasticizer**-Superplasticizer is sulfonate naphthalene formaldehyde based, high range water reducing admixture. It meets the requirements of ASTM C 494 Type A, F or G. It is normally used to lower the water-cement ratio in concrete. The process is often employed to produce high strength concrete.

**Sulfuric Acid**- Sulfuric acid 10 ml/l was prepared by using sulfuric reagent (96.6% concentration) 10% by volume mixed with deionized water 90% by volume.

**Water**-Two kinds of water were used in this program. Tap water was used as a mixing water of all types of mortar. Deionized water was mixed in acid solution.

### 3.3 Test Program

#### 3.3.1 Effect of Fly Ash Characteristics on Sulfuric Acid Resistance of Fly Ash Mortar

Fractionated fly ashes, 3F, the original feed of dry bottom fly ash, and MM fly ash (from Thailand) were mixed with cement to form fly ash cement mortar. Standard 2-inch cubes were cast and cured in saturated lime water for 28 days before being put into the acid pond. The mix proportions used are shown in Table 3.1. The percentages of fly ash used in the mixes were 35 and 50 percent by weight of cementitious (cement+fly ash) materials. Fly ash was used as cement replacement. The water to cementitious materials ratio of all mixes was kept constant at 0.5. Fly ash cement mortar samples and the control samples (no fly ash) were then immersed in the H<sub>2</sub>SO<sub>4</sub> acid solution with a concentration of 100 ml/l. All samples were kept under the same corrosive environment until the date of testing. To evaluate the

extent of the damage caused by acid attack, the samples were removed from the acid pond and washed with tap water. The samples were then weighed and tested compressive strength at the saturated surface dry condition. The data of specimens were then determined and recorded.

### **3.3.2 Effect of Fractionated Fly Ash on Sulfuric Acid Resistance of Fly Ash Mortar**

Fractionated fly ashes, 3F, the original feed of dry bottom fly ash was mixed with cement to form fly ash cement mortar. Standard 2-inch cubes were cast and cured in saturated lime water for 28 days before being put into the acid pond. The mix proportions used are shown in Table 3.1. The percentages of fly ash used in the mixes were 35, 45, 50 and 60 percent by weight of cementitious (cement+fly ash) materials. Fly ash was used as cement replacement. The water to cementitious materials ratio of all mixes was kept constant at 0.5. Fly ash cement mortar samples and the control samples (no fly ash) were then immersed in the  $H_2SO_4$  acid solution with a concentration of 100 ml/l. All samples were kept under the same corrosive environment until the date of testing. To evaluate the extent of the damage caused by acid attack, the samples were removed from the acid pond and washed with tap water. The samples were then weighed and tested compressive strength at the saturated surface dry condition. The data of specimens were then determined and recorded.

### **3.3.3 Effect of Microsilica on Sulfuric Acid Resistance and Compressive Strength of Fly Ash Mortar**

In this test program, two batches of mortar are mixed. Microsilica mortar were mixed with 5%, 10% and 50% cement replacement. The water cement ratio of MS5 and MS10 were 0.5 but that of MS50 was 0.7. Another batch is microsilica and fly ash mortar. Microsilica was mixed with 3F fly ash, cement and sand. The

replacement of a portion of portland cement by microsilica was 5% and by fly ash was 30% by weight of cementitious (cement + fly ash + microsilica) materials. The water cement ratio of microsilica fly ash mortar was 0.5. The mix proportion is presented in Table 3.1. All specimens were mixed and cast in accordance with ASTM C-109 (1990). Then they were cured in saturated lime water prior to put into 100 ml/l of sulfuric acid solution. After that they were removed from the pond and weighed and tested compressive strength at the saturated dry condition.

#### **3.3.4 Effect of Superplasticizer on Sulfuric Acid Resistance and Compressive Strength of Fly Ash Mortar**

Superplasticizer was mixed with 3F, OR, MM fly ash, cement and sand. The replacement of a portion of portland cement by fly ash was 35% and 50% by weight of cementitious materials. The mix proportion is presented in Table 3.1. All specimens were mixed and cast in accordance with ASTM C-109 (1990). Then they were cured in saturated lime water prior to put into 100 ml/l of sulfuric acid solution. After that they were removed from the pond and weighed and tested compressive strength at the saturated dry condition.

#### **3.3.5 Optimum Mix of Fractionated Fly Ash Mortar**

Attempts are made to relate the corrosion resistance and compressive strength of fly ash. The results of fractionated fly ash mortar with different mix proportions and admixtures were used to find the optimum mix of fly ash mortar.

#### **3.3.6 Strength Contribution of Fly Ash Mortar**

3F fly ash and river sand were used as cement replacement in mortar. The replacement of a portion of portland cement by fly ash and sand were varied, 0%, 15%, 25% and 50% by weight of cementitious materials. The cementitious material

means a summation of cement and fly ash for fly ash mortar. For sand mortar, it means a summation of cement and sand used as replacement. The mix proportion is presented in Table 3.1. All specimens were mixed and cast in accordance with ASTM C-109 (1990). Then they were cured in saturated lime water and tested compressive strength at the age of 1, 3, 7, 14, and 28 days.

Table 3.1 Mix Proportion

Sample	Cementitious Material (C)			Sand	W/C	P
	CEM	FA	MS			
MC	100%	0%	--	2.75	0.5	--
M3F35	65%	35%	--	2.75	0.5	--
MOR35	65%	35%	--	2.75	0.5	--
MM35	65%	35%	--	2.75	0.5	--
M3F50	50%	50%	--	2.75	0.5	--
MOR50	50%	50%	--	2.75	0.5	--
MM50	50%	50%	--	2.75	0.5	--
PM3F35	65%	35%	--	2.75	0.35	10
PMOR35	65%	35%	--	2.75	0.35	10
PMM35	65%	35%	--	2.75	0.35	10
PMS53F30	65%	30%	5%	2.75	0.5	10
PM3F50	50%	50%	--	2.75	0.35	10
PMOR50	50%	50%	--	2.75	0.35	10
PMM50	50%	50%	--	2.75	0.35	10
MS5	95%		5%	2.75	0.5	--
MS10	90%		10%	2.75	0.5	--
MS50	50%		50%	2.75	0.7	--
MS53F30	65%	30%	5%	2.75	0.5	--
M3F15	85%	15%	--	2.75	0.5	--
M3F25	75%	25%	--	2.75	0.5	--
M3F45	55%	45%	--	2.75	0.5	--
M3F50	50%	50%	--	2.75	0.5	--
M3F60	40%	60%	--	2.75	0.5	--
S15	85%	--	--	3.41	0.5	--
S25	75%	--	--	4.00	0.5	--
S50	50%	--	--	6.50	0.5	--

## Notes:

C - Cementitious Materials (Cement+Fly Ash+Microsilica)

W - Water

P - Superplasticizer

F - Fly Ash

3F- Finest Fractionated Fly Ash

OR- Original Dry Feed Fly Ash from Hudson, USA

MM- Original Dry Feed Fly Ash From Mae Moe, Thailand

MS- Microsilica

S - River sand

CEM-Ordinary portland cement type I



## CHAPTER 4

### RESULTS AND DISCUSSIONS

#### 4.1 Effect of Fly Ash Characteristics to Sulfuric Acid Resistance of Fly Ash Mortar

Different type of fly ash are mixed with cement to form the fly ash cement mortars. Standard 2-inch cubes were cast and cured in saturated lime water about 28 days before being put into the acid pond. The mix proportions used are tabulated in Table 3.1. The percentage of fly ash used in the mixed was 35 and 50 percent by weight of cementitious materials. The water to cementitious material ratio of all mixes was kept constant at 0.50. No other admixtures were used in this program. Fly ash cement-mortar samples and the control samples (no fly ash) were then immersed in the sulfuric acid solution with a concentration of 100 ml/l. All samples were kept under the same corrosion environment until the day of testing. To evaluate the extent of the damage caused by acid attack, the samples were removed from the acid pond and washed with tap water. The samples were then weighed at the saturated surface dry condition. After weighing, the sample will then be determined compressive strength. Sample designated "MC" is the control mix which contains only ordinary portland cement type I (no fly ash in the mix), "3F" is the mix which contains the finest fractionated fly ash, "OR" is the mix which contains original fly ash from US., and "MM" is the mix which contains original fly ash from Mae Moh, Thailand. The number "35" and "50" stand for the percentage of cement replaced by fly ash.

Table 4.1 lists the weight of fly ash mortar immersed in  $H_2SO_4$  100 ml/l at different immersed time when using different type of fly ash, 3F, OR, and MM with 35% and 50% cement replacement. Table 4.2 lists their compressive strength when immersed in  $H_2SO_4$  100 ml/l at different immersed time. Figure 4.1 and 4.2 show

the weight of 3F, OR, MM and the normal cement control mortar after different immersion times in 100 ml/l  $H_2SO_4$  with 35% cement replacement. Figure 4.3 and 4.4 show those of the 50% fly ash mortar. Clear illustrations of weight and compressive strength of each sample are shown in Figure A 1, A 3 to A 10 and Figure A 2, A 11 to A 18 in Appendix A, respectively.

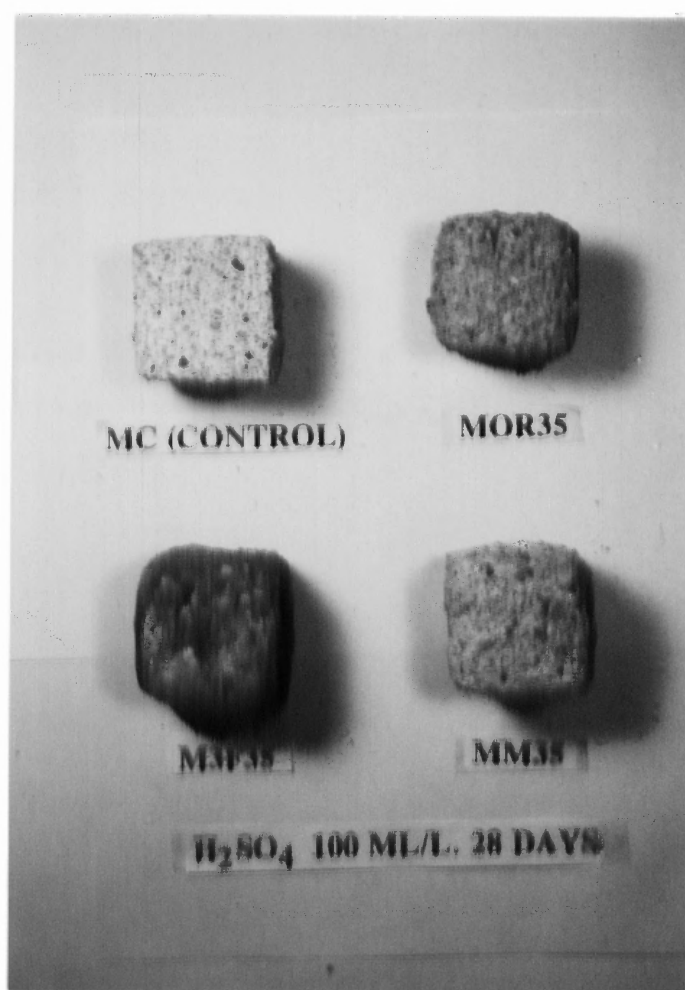
**Table 4.1** Weight of Fly Ash Mortar in  $H_2SO_4$  100 ml/l at Different Immersed Time (35% and 50% Cement Replacement)

Sample No.	Weight at Different Ages (g)						Comp psi.
	0-day	1-day	3-day	7-day	14-day	28-day	
MC	298.1	288.0	265.2	217.6	173.1	129.3	8178.3
M3F35	298.4	299.5	298.5	262.5	217.0	194.6	5925.0
MOR35	297.6	292.4	279.4	241.2	203.2	140.0	5680.8
MM35	292.5	280.9	272.2	235.9	182.6	162.3	5413.3
M3F50	296.9	297.0	292.9	293.6	293.2	288.6	4695.0
MOR50	295.5	293.3	291.7	285.3	284.1	270.7	4647.5
MM50	289.2	286.1	284.6	268.9	253.9	243.8	4540.0

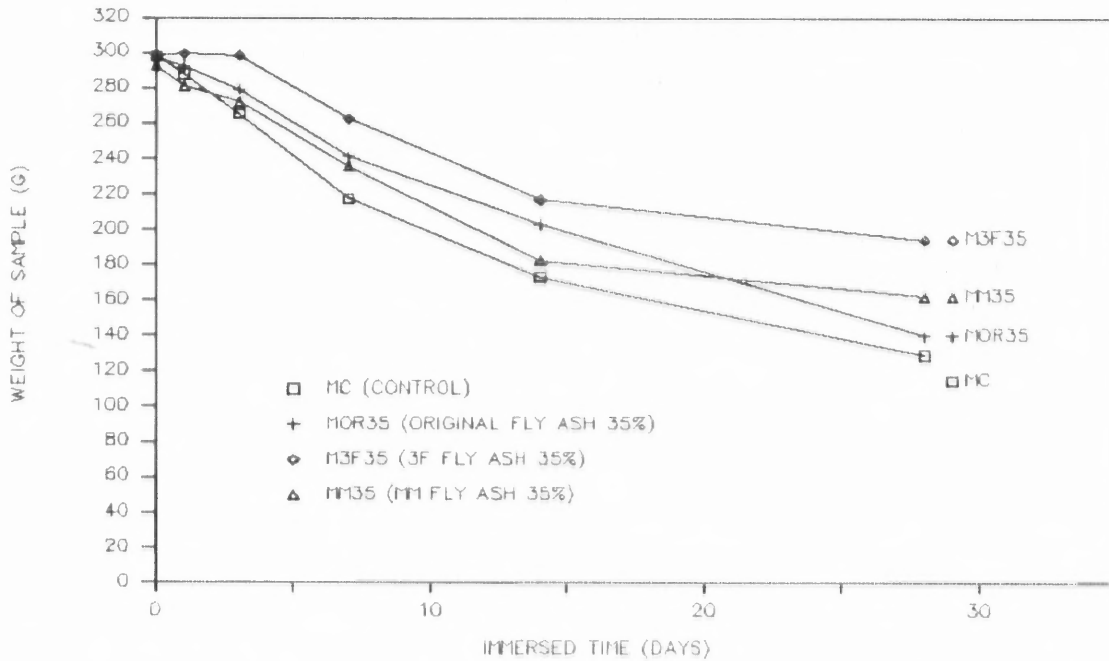
**Table 4.2** Compressive Strength of Fly Ash Mortar in  $H_2SO_4$  100 ml/l at Different Immersed Time (35% and 50% Cement Replacement)

Sample No.	Strength at Different Ages (psi)					
	0-day	1-day	3-day	7-day	14-day	28-day
MC	8178.5	6012.5	5005.8	4619.2	2475.6	1879.1
M3F35	5925.0	5775.8	5931.3	5037.3	3836.7	2266.1
MOR35	5680.8	4534.2	4975.0	4739.1	3850.0	2027.5
MM35	5413.3	5262.5	5487.5	4311.7	2179.4	2745.7
M3F50	4695.0	4747.5	5022.5	5104.2	3540.8	2950.0
MOR50	4647.5	5045.0	4570.0	4256.7	3019.2	2415.8
MM50	4540.0	4643.3	4915.0	3291.7	2395.1	2226.1

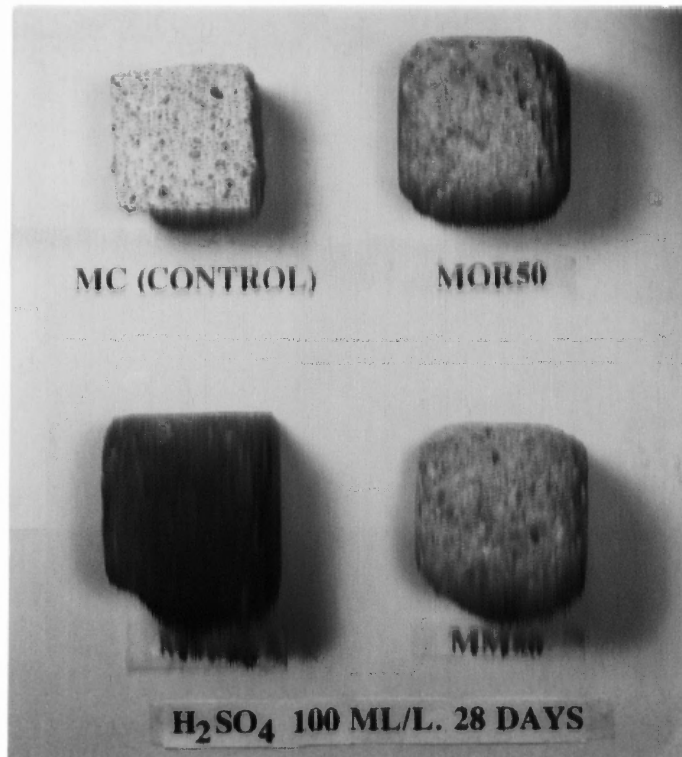
It can be seen from Figure 4.1, 4.2, 4.3 and 4.4 that fly ash mortar can resist sulfuric acid more than normal cement mortar, either 35% or 50% cement replacement. The 3F fly ash mortar is the most sulfuric acid resistance mix out of those two mixes, OR and MM. The MM fly ash mortar is the most vulnerable mix compared between fly ash mortar.



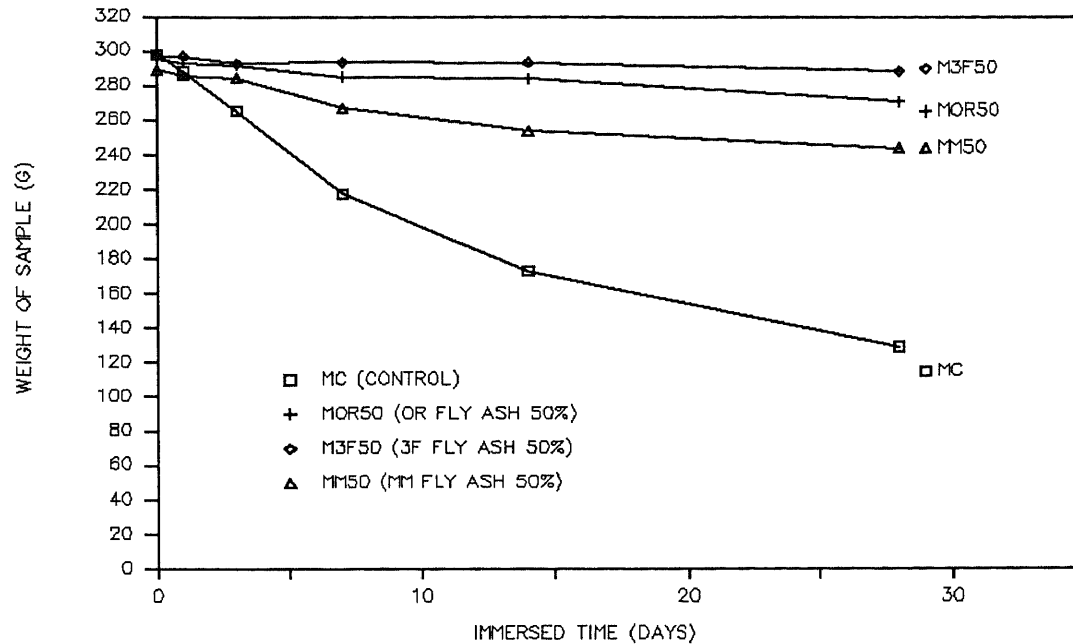
**Figure 4.1** Different Type of 35% Fly Ash Mortar After Immersed in  $H_2SO_4$  100 ml/l for 28 Days



**Figure 4.2** Relationship between Weight of Fly Ash Mortar Samples in  $H_2SO_4$  100 ml/l and Immersed Time When Using Fly Ash 35% as Cement Replacement



**Figure 4.3** Different Type of 50% Fly Ash Mortar After Immersed in  $H_2SO_4$  100 ml/l for 28 Days



**Figure 4.4** Relationship between Weight of Fly Ash Mortar Samples in  $H_2SO_4$  100 ml/l and Immersed Time When Using Fly Ash 50% as Cement Replacement

#### 4.1.1 Effect of Chemical Composition of Fly Ash and Cement to Sulfuric Acid Resistance of Fly Ash Mortar

**Table 4.3** Chemical Composition of Fly Ashes and Cement

		Chemical Composition (%)							
Sam	LOI	SO <sub>3</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	K <sub>2</sub> O	MgO	NaO
CEM	0.73	2.53	20.07	8.84	1.41	60.14	0.86	2.49	0.28
3F	4.97	1.69	49.89	26.94	5.43	2.99	1.76	0.99	0.33
OR	2.75	0.98	52.25	26.72	5.43	2.41	1.67	0.69	0.28
MM	0.40	2.36	40.39	22.75	16.06	12.65	2.37	2.06	0.52

Table 4.3 shows the chemical composition of fly ashes, 3F, OR, and MM. Sample CEM is the ordinary portland cement type I used in this study. The major

concerning in chemical compositions of these fly ashes are in the  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{CaO}$ .

The hypothesis is that  $\text{SiO}_2$  in the fly ash will bind with  $\text{Ca}(\text{OH})_2$ , thus protecting mortar from attack. It can be seen from Table 4.3 that  $\text{SiO}_2$  content of the OR fly ash mortar is the highest and that of the MM fly ash mortar is the lowest. The expectation is that the OR should be the most effective imparting acid resistant and MM should be the least effective.

It can be seen from Table 4.3 that  $\text{Al}_2\text{O}_3$  content in cement is rather high. ASTM C 150-90 recommends that  $\text{Al}_2\text{O}_3$  content of sulfate resisting cement should not be more than 5%. Therefore, the type of cement used in this test, ordinary portland cement type I, is vulnerable to sulfate attack. Although the fly ashes have quite high percentages of  $\text{Al}_2\text{O}_3$ , it does not react with  $\text{CaSO}_4$  to form the expansive compound, ettringite. This is because the  $\text{Al}_2\text{O}_3$  of fly ash is not amorphous and is therefore unavailable to react with  $\text{CaSO}_4$ .

Another important chemical compound to consider is  $\text{CaO}$ . The calcium content of coal has a significant effect on fly ash mineralogy and reactivity. Diamond and Lopez (1981) and McCarthy, Swanson, Keller, and Blatter (1984) stated that fly ashes which have less than 10%  $\text{CaO}$  consist mostly of aluminosilicate-type glass. Meanwhile, fly ashes which have more than 15%  $\text{CaO}$  consist mostly of reactive calcium aluminosilicate-type glass along with crystalline compounds,  $\text{C}_3\text{A}$ ,  $\text{CS}$  and  $\text{CaO}$ . Samples of 3F and OR have low  $\text{CaO}$  content. Therefore, they do not add vulnerable products to the mortar structure. Since the MM fly ash which has a high 16.06%  $\text{CaO}$  content. Its mortar is more corroded easily than the other two mixes, as shown in Figure 4.1, 4.2, 4.3 and 4.4.

Given its chemical compound, the OR fly ash mortar is expected to be the most acid resistant mix of this experimental series. Meanwhile, the MM fly ash mortar is expected to be the most vulnerable one. The result shows that MM fly ash

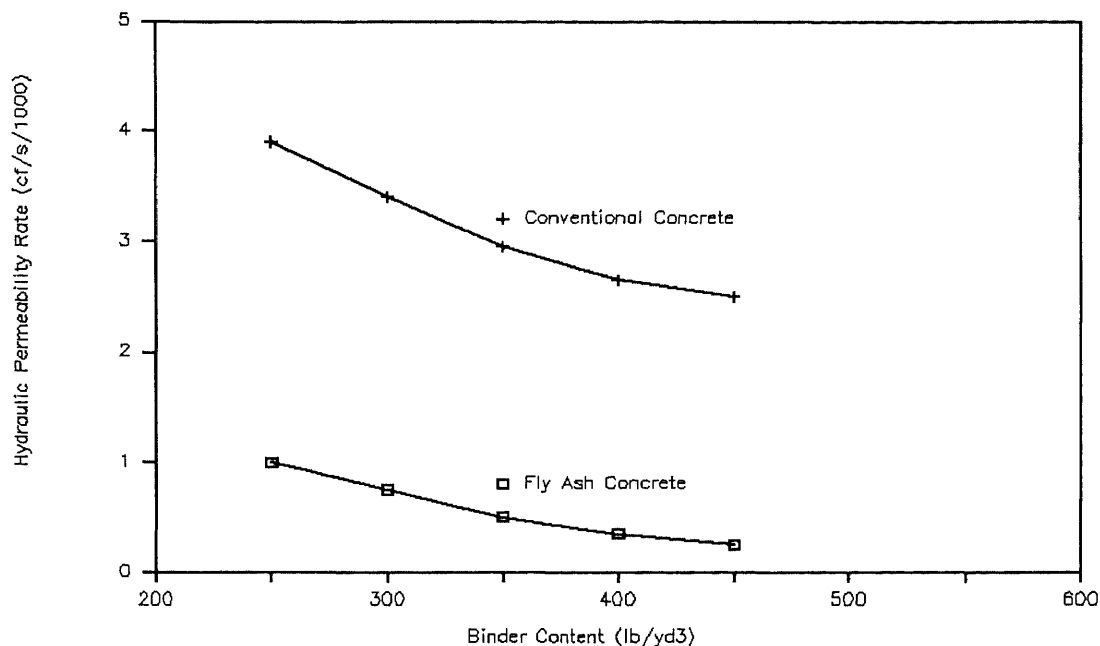
mortar is the most vulnerable mortar compared to those fly ash mixes. It can be seen that instead of OR, 3F fly ash mortar is the most acid resistant at either 35% or 50% cement replacement. Therefore, there must be some other characteristics of fly ash contributing to its acid resistance.

#### **4.1.2 Effect of Permeability of Fly Ash Mortar to Sulfuric Acid Resistance**

The permeability is another important characteristic of mortar inhibiting the acid attack. Many researchers have found that much of an improvement of pozzolana at first stage is attributed to this physical action (Bache, H.H. 1981) Thus, many attempts tried to lower the permeability of mortar.

Many researchers observed the porosity and permeability of ordinary portland cement concrete and fly ash concrete. Using Scanning Electron Microscopy, Berke, N.S., Scali, M.J., Regan, J.C., and Shen, D.F. (1992) found that ordinary portland cement concrete was more porous than concrete with the mineral admixtures silica fume and fly ash. In addition, Torii, K., and Kawamura, M. (1992) concluded after a study using the rapid chloride permeability test that normal cement concrete was more permeable than concrete containing mineral admixtures

Adding fly ash to mortar significantly reduces its permeability as shown in Figure 4.5. This may be due to their fineness and their particle size distribution. Berke, N.S., Scali, M.J., Regan, J.C., and Shen, D.F. (1992) reported that the small particle of pozzolans fill up the capillary and gel pore spaces forming a tighter, less porous paste structure. Bayasi, Z. (1992) found that the decrease in volume of permeable void when fly ash is used can be attributed to void refining due to better gradation of fine particles. Therefore, it retarded the acid to diffuse into the inner structure.



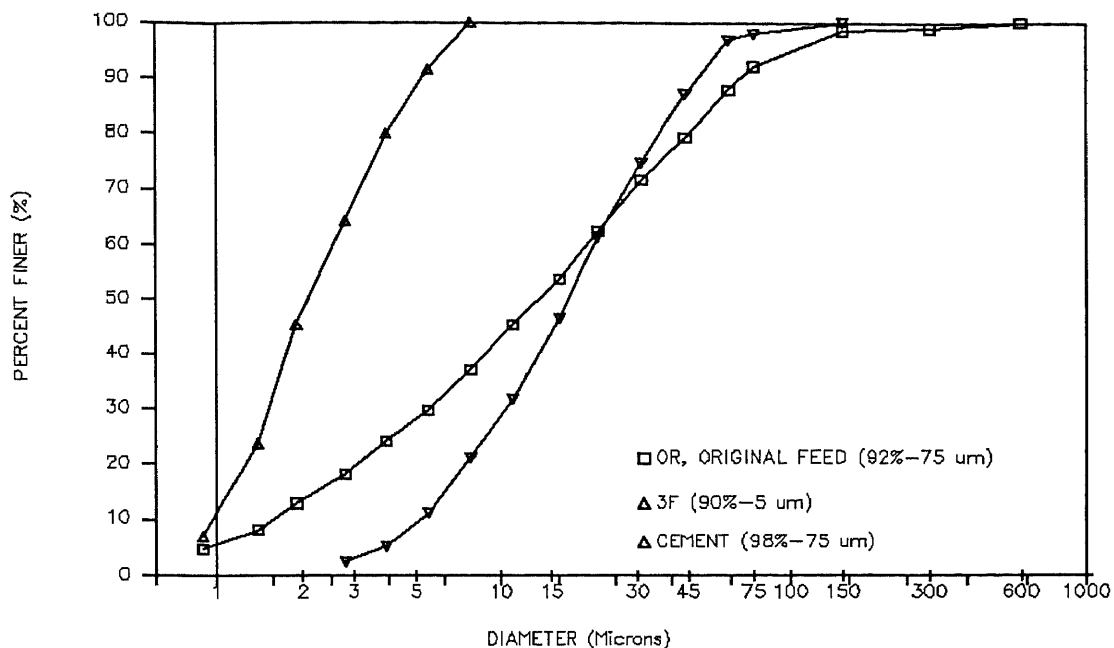
**Figure 4.5** Typical Effects of Fly Ash on the Hydraulic Permeability of Concrete Obtained from Federal Highways Administration, U.S Department of Transportation, 1986.

It can be seen from Table 4.4 and Figure 4.6 that most of particle of 3F fly ash is finer than that of OR fly ash. The small particle of 3F fly ash is able to fit into the small spaces between the cement and aggregate thereby diminishing the size and extent of the pore spaces. Being less permeable the 3F mortar is less vulnerable to attack and is corroded less than the plain or OR mortar although the 3F fly ash has less  $\text{SiO}_2$  than the OR fly ash. Thus, it can be concluded that the fineness of fly ash has higher effect to acid resistance than chemical characteristic.

**Table 4.4** Fineness of Fly Ashes and Cement

Sample No.	Specific Gravity (g/cm <sup>3</sup> )	Fineness		Mean Diameter (um)
		Retained 45 um (%)	Blaine (cm <sup>2</sup> /g)	
CEM	3.12		3815	-
3F	2.54	0	7844	2.11
OR	2.34	20.0	3235	13.73





**Figure 4.6** Particle Size Distribution of 3F, OR, MM Fly Ash and Cement

#### 4.2 Effect of Fractionated Fly Ash on Sulfuric Acid Resistance of Fly Ash Mortar

Fractionated fly ash, 3F, is mixed with cement to form the fly ash cement mortar. Standard 2-inch cubes were cast and cured in saturated lime water 28 days before being put into the acid pond. The mix proportions used are tabulated in Table 3.1. The percentage of fly ash used in the mixes was 35, 45, 50 and 60 percent by weight of cementitious materials. The water to cementitious material ratio for all mixes was kept constant at 0.50. No other admixtures were used. Fly ash cement-mortar samples and the control samples (no fly ash) were then immersed in a 100 ml/l sulfuric acid solution. All samples were kept under the same corrosion environment until the day of testing. To evaluate the extent of the damage caused by the acid attack, the samples were removed from the acid pond and washed with tap water. The samples were then weighed in the saturated surface dry condition. After weighing, the samples were tested for their compressive strength. Sample designated

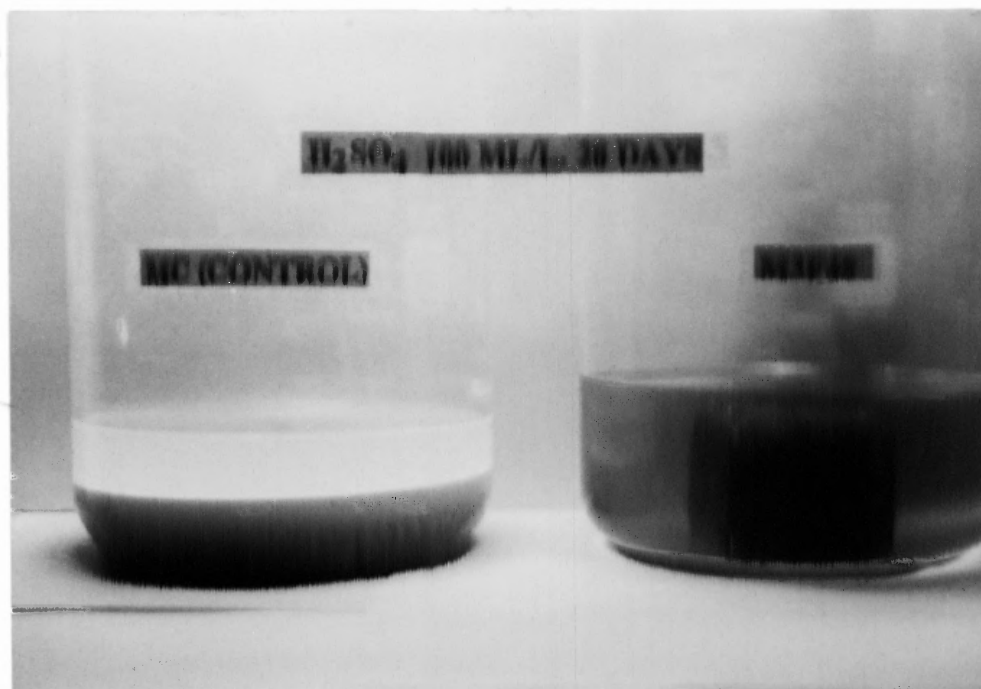
"MC" is the control mix which contained no fly ash, "3F" are the mixes which contained 3F fly ash. The numbers "35", "45", "50" and "60" are the percentage of cement replaced by fly ash.

**Table 4.5** Weight of 3F Fly Ash Mortar in H<sub>2</sub>SO<sub>4</sub> 100 ml/l at Different Immersed Time (35%, 45%, 50%, and 60% Cement Replacement)

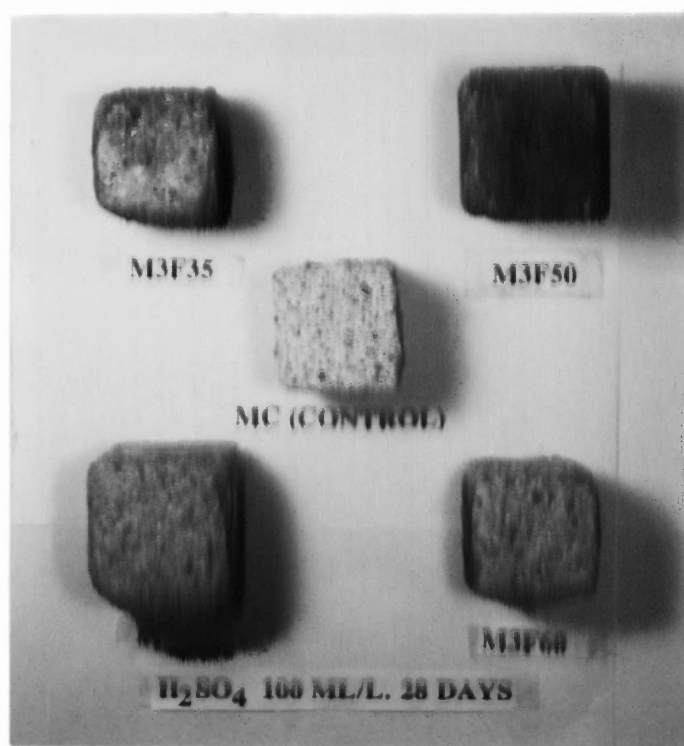
Sample No.	Weight at Different Ages (g)						Comp psi.
	0-day	1-day	3-day	7-day	14-day	28-day	
MC	298.1	288.0	265.2	217.6	173.1	129.3	8178.3
M3F35	298.4	299.5	298.5	262.5	217.0	194.6	5925.0
M3F45	296.7	293.5	293.5	286.2	279.3	268.9	4797.5
M3F50	296.9	297.0	292.9	293.6	293.2	288.6	4695
M3F60	298.8	292.4	288.7	267.6	251.3	194.0	4459.2

Table 4.5 shows the weights of the samples at different times after being submerged in the concentrated 100 ml/l of H<sub>2</sub>SO<sub>4</sub> and their 28-day compressive strength. Clear illustration of weight of each sample is shown in Figure A 3 to A 6 in Appendix A. The normal mortar sample is obviously much more corroded than the fly ash mortars.

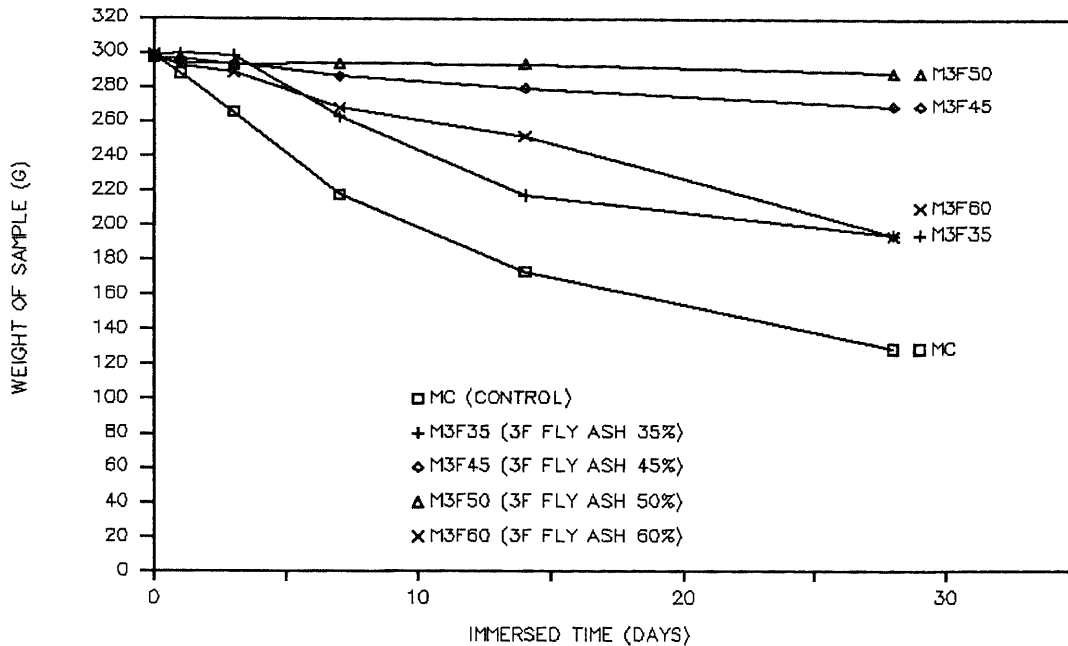
The normal cement mortar lost 56.6 % of its weight in 28 days. It seems that every compound of control sample is vulnerable to sulfuric acid attack. Two corrosion processes occur simultaneously. For sulfate attack, the SO<sub>4</sub><sup>2-</sup> reacts with Ca(OH)<sub>2</sub> to form gypsum which produces expansive compound. For acid attack, hydrogen ion reacts directly with CSH, the hydration product, to form silica gel. These by-products cannot be held in the normal mortar structure. They leach out resulting in smaller specimen. It can be seen from Figure 4.7 that there is much silica gel in solution submerging normal cement mortar and the specimen does not spall. Meanwhile, the 45% 3F fly ash mortar does not have silica gel surrounding it.



**Figure 4.7** Normal Cement Mortar and 45 % 3F Fly Ash Mortar and Their Products in  $H_2SO_4$  100 ml/l for 28 Days



**Figure 4.8** 3F Fly Ash Mortar at different percentage of fly ash and Normal Cement Mortar After Immersed in  $H_2SO_4$  100 ml/l for 28 Days



**Figure 4.9** Relation between Weight of 3F Fly Ash Mortar Sample in  $H_2SO_4$  100 ml/l at Different Percentage of Fly Ash and Immersed Time

The deterioration of mortar was reduced, as expected, using fly ash as a cement replacement. Clear illustrations of the sulfuric acid resistance of fly ash mortar are shown in Figure 4.8 and 4.9. The more details on the nature of this resistance follow.

It can be seen from Figure 4.9 that the deterioration of normal cement mortar, MC, is always highest. Fly ash mortars, on the other hand, up to 7 days have almost only small weight losses. It is believed that the deterioration at this early stage is controlled by permeability of specimen since it occurs mostly at surface. If the mortar is more porous, the acid will penetrate further to react with vulnerable compounds. Therefore, the normal cement mortar which has the highest permeability has more deterioration than the others. That the effect of fly ash on mortar at early ages is fly ash's contributions to the so called "Packing effect" rather than its pozzolanic action.

In the second stage, 7 to 14 days, fly ash's major contribution to sulfuric acid resistance is still to lower the permeability of mortar. But the deterioration rate for each fly ash mortar can be distinguished more clearly than the very early stage. It can be seen from Figure 4.9 that the fly ash mortar with the lowest percentage of fly ash and the normal cement mortar have higher deterioration rates. But no change the rates for M3F45 and M3F50 show.

Those higher deteriorations result from greater surface area for reaction. The acid penetrates through the pores into the sample where it can react in three rather than only two dimensions (Islander, Devinsky, Mansfeld, Postyn, and Shih 1991). Thus, vulnerable compounds are more easily attacked by the acid. But, for mortar with a high percentage of fly ash, the acid is impeded because of its lowered permeability. The contribution of fly ash's pozzolanic action is developing but still has no effect on the acid resistance.

In the final period, the deterioration of fly ash mortar tends to be constant. In addition to blocking acid penetration with its fine particles, its pozzolanic activity buttresses the corrosion inhibition. Silicious compounds react with free lime to form more stable products that further fill the pore structure. The evidence supporting this idea was obtained from Feldman, R.F. (1992). He found that the open channels in concrete with mineral admixture at higher age were blocked by pozzolanic reaction products.

Even though the hydrogen ion can still react with the hydration products, but these products are buried inside as stable compounds. The whole structure is held together and tightly sealed so that no further weight loss is experienced. The evidence for this conclusion is in Figure 4.8 where no silica gel or gypsum leaching out of high fly ash mortar is observed.

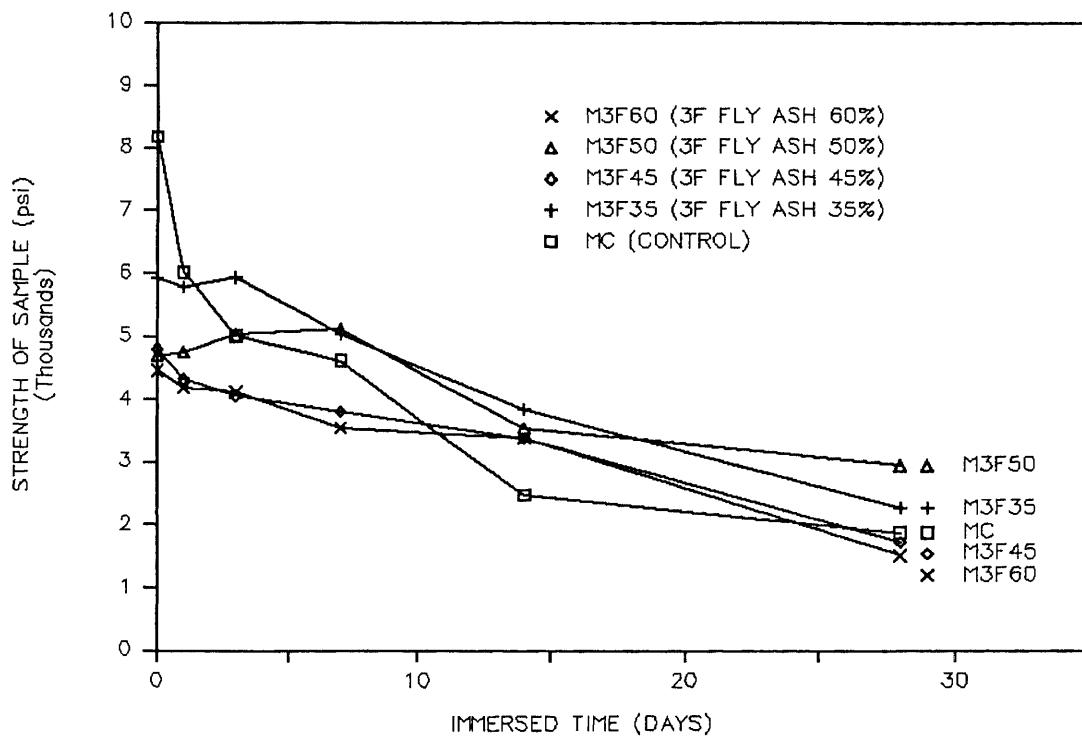
It can be seen from Figure 4.9 that there is a limitation on the use of fly ash. Up to 50% cement replacement, the more fly ash consisting in the mix, the higher

the sulfuric acid resistance. But above this level the acid resistance is suppress. As seen from Figure 4.9, the 60 % fly ash mortar loses more weight, at all times, than the 50% fly ash mortar. Therefore, the most effective sulfuric acid resistance, resulting from both physical and chemical mechanisms, occurs with 50% cement replacement with fly ash.

The limitation for using fly ash may be the result of a lower percentage of calcium hydroxide available. When there is less  $\text{Ca(OH)}_2$  there will be less formation of bonding compounds to secure the structure thereby allowing the acid to penetrate. Costa and Massazza (1983) found that the reaction between fly ash and lime released by hydrolysis of clinker silicates not only depends on the specific surface area of fly ash but also on the calcium hydroxide availability. Additionally, Gopalan (1993) reported that if the fly ash-cementitious ratio increased, the contribution of the pozzolanic reaction would decrease. This is explained as the combined effect of reduction in free lime liberated and increase in chemisorption by greater amounts of fly ash.

**Table 4.6** Compressive Strength of 3F Fly Ash Mortar in  $\text{H}_2\text{SO}_4$  100 ml/l (35%, 45%, 50%, and 60% Cement Replacement)

Sample No.	Strength at Different Ages (psi)					
	0-day	1-day	3-day	7-day	14-day	28-day
MC	8178.5	6012.5	5005.8	4619.2	2475.6	1879.1
M3F35	5925.0	5775.8	5931.3	5037.3	3836.7	2266.1
M3F45	4797.5	4320.8	4055.5	3799.2	3377.5	1730.0
M3F50	4695.0	4747.5	5022.5	5104.2	3540.8	2950.0
M3F60	4459.2	4192.5	4115.3	3583.2	3373.9	1515.4



**Figure 4.10** Relationship between Compressive Strength of 3F Fly Ash Mortar Samples in H<sub>2</sub>SO<sub>4</sub> 100 ml/l at Different Percentage of Fly Ash and Immersed Time

Table 4.6 presents the compressive strength with respect to time of 3F fly ash mortar after immersed in H<sub>2</sub>SO<sub>4</sub> 100 ml/l. Complete illustrations of relationship between compressive strength of each fly ash mortar and immersed time are presented in Figures A 11 to A 14 in Appendix A. It can be seen from the Figure 4.10 that there are the differences in initial strength as expected. This property varies depending on the percentage of fly ash. If the higher percentage is used, the lower the 28-days strength will be obtained.

It is usually expected that a mortar will increase in strength with age. However all fly ash sample had less and less strength after longer immersed time. Even the high fly ash mortar, M3F50, which almost has no weight loss has less strength. However, it cannot conclude from the Figure 4.10 that the high percentage

fly ash has more acid resistance since there is no apparent relation between the percentage of fly ash used in the mix and its compressive strength.

**Table 4.7** Compressive Strength and Weight of 3F Fly Ash Mortar in  $H_2SO_4$  100 ml/l at 28 Days Immersed Time (35%, 45%, 50% and 60% Cement Replacement)

Sample	56-d Strength		St. Loss %	56-d Weight		Wt. Loss %
	curing	$H_2SO_4$		curing	$H_2SO_4$	
MC	9872	1879	80.97	298.1	43.8	85.31
M3F35	9833	2266	77.00	298.4	103.8	34.79
M3F45	5993	1730	71.13	296.7	268.9	9.37
M3F50	6062	2950	51.34	296.6	288.6	2.80
M3F60	5773	1515	73.75	298.9	194.0	35.10

Table 4.7 presents strength and weight of different fly ash mortar in  $H_2SO_4$  at 28 days immersed time. The percentage of strength loss of each 3F fly ash mortar is obtained by comparing strength of sample immersed in  $H_2SO_4$  for 28 days with 56-days strength (not immersed in  $H_2SO_4$ ). The percentage of weight loss of each sample is obtained by comparing weight before immersed and after immersed in sulfuric acid.

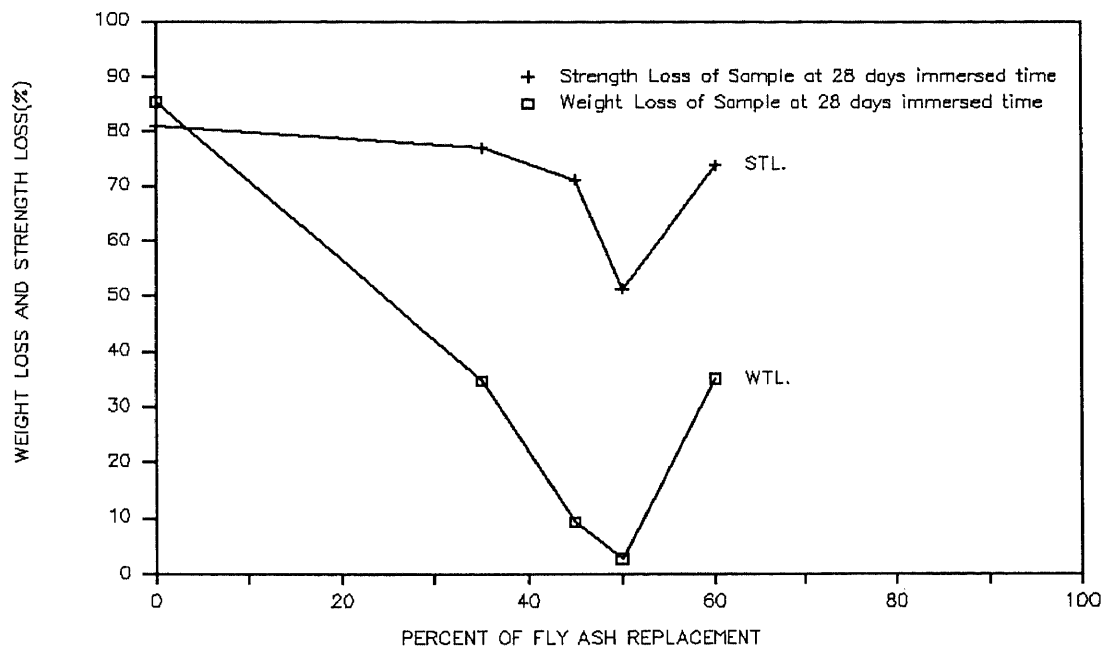
It can be seen from Figure 4.11 that the strength loss of each percentage of fly ash mortar (not more than limitation percent) is higher than the weight loss. This confirms that the reaction still occurs inside and changes the chemical and physical properties of sample.

It is explained that the hydrogen ion penetrated into the inner structure of fly ash mortar and reacted with free lime and CSH. Due to the low porosity of specimen, the products are buried inside. Therefore, less weight loss gains but strength reduces significantly in fly ash mortar. The evidence supporting this idea is that there are more white materials dispersing inside the normal cement mortar than



the fly ash mortar.

It can be concluded from the results that fly ash can be used to improve sulfuric acid resistance of mortar. Up to the limited value, the higher the volume of fly ash used in the mix, the higher the sulfuric acid resistance it provides. The percentage of fly ash should be proportional to the cement content of that mix to give the highest durability. However, compressive strength of fly ash mortar should be enhanced so it can be used in practical.



**Figure 4.11** Relationship between Compressive Strength and Weight of 3F Fly Ash Mortar Sample in  $H_2SO_4$  100 ml/l 28 Days Immersed Time and Different Percentage of Fly Ash

#### 4.3 Effect of Microsilica on Sulfuric Acid Resistance and Compressive Strength of Fly Ash Mortar

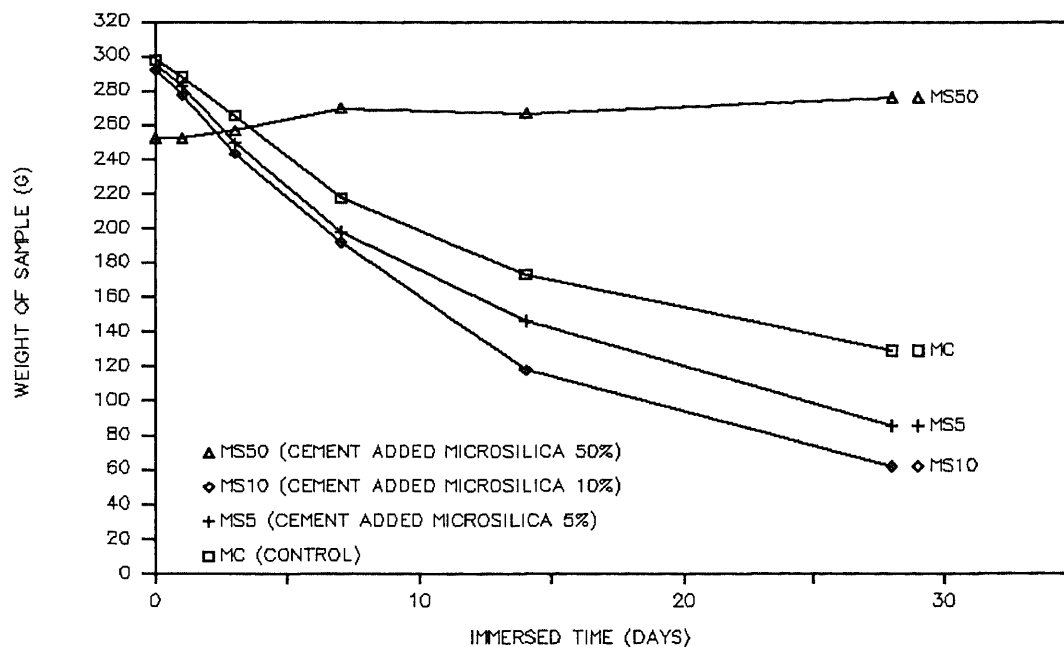
In this section, testing is made to improve the sulfuric acid resistance and compressive strength of fly ash mortar. Previous study showed that fly ash mortar has lower initial compressive strength than ordinary portland mortar. In addition,

although fly ash mortar has low weight loss in corrosive environment but it still has low compressive strength. Thus, the other materials are used to improve its durabilities. The concerned material for compensating the low strength is microsilica.

Microsilica in the powder form 5%, 10% and 50% are used as cement replacement in mortar. Also, the 5% microsilica is mixed with the 30% 3F fly ash as cement replacement in mortar. "MS" stands for microsilica and "3F" stands for finest fly ash. The water cement ratio of MS5, MS10, and MS53F30 were kept constant 0.50 but that of MS50 was 0.70. The mix proportion is shown in Table 3.1. All mixes were cast, cured and tested as the same process as fly ash mortar. Complete illustrations of relationship between weight samples and immersed time are shown in Figure A 19 to A 22 in Appendix A. The illustrations of relationship between compressive strength of sample are shown in Figure A 23 to A 26 in Appendix A.

**Table 4.8** Weight of Microsilica Mortar in H<sub>2</sub>SO<sub>4</sub> 100 ml/l at Different Immersed Time

Sample No.	Weight at Different Ages (psi)						Comp psi.
	0-day	1-day	3-day	7-day	14-day	28-day	
MC	298.1	288.0	265.2	217.6	173.1	129.3	8178.3
MS5	295.6	282.9	249.4	198.0	146.1	85.9	7905.0
MS10	292.1	277.4	243.0	192.0	118.0	62.1	7883.3
MS50	252.5	252.2	256.7	269.7	266.8	276.2	2431.3



**Figure 4.12** Relationship between Weight of Microsilica Mortar in  $H_2SO_4$  100 ml/l and Immersed Time

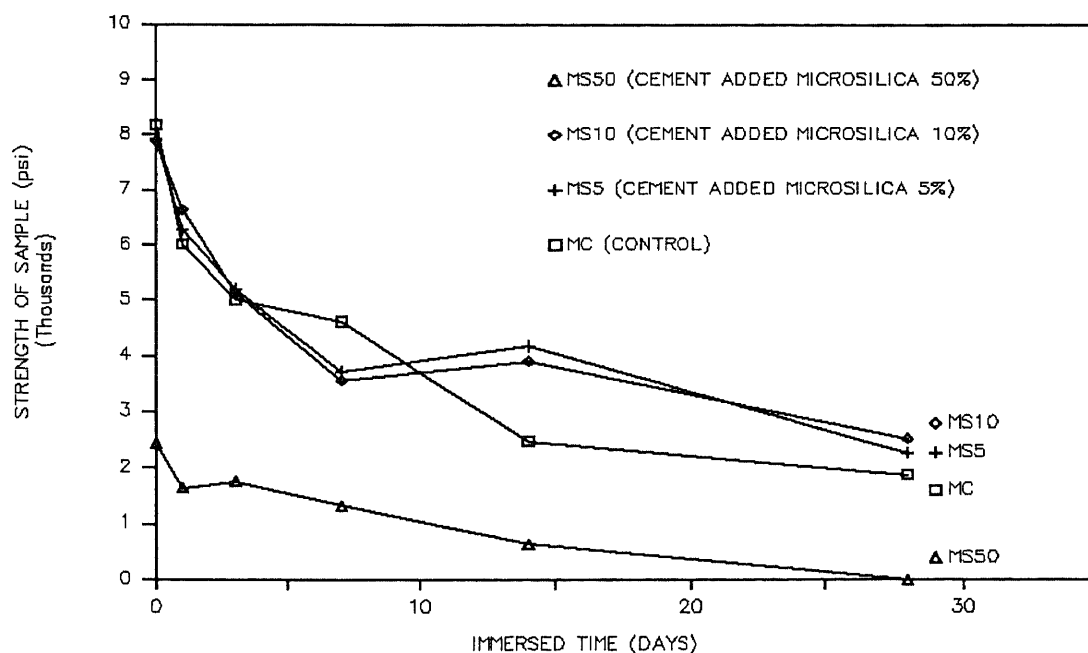
Table 4.8 shows the weight of microsilica mortar. Figure 4.12 is the relationship between the weight of those mixes and age. It can be seen from Figure 4.12 that there are two ranges of using microsilica, the low percentage, 5% and 10% and the high percentage of microsilica 50%. The low percentage of microsilica has a lower sulfuric acid resistance than high percentage as expected. This can be explained that small particle size of microsilica fills the void between cement and aggregate and inhibits acid from reacting with vulnerable compounds. Moreover, at later age, the pozzolanic action also tied up these cement with aggregate resulting in a lower deterioration.

By comparing within low percentage of microsilica, 5% and 10%, it can be seen that the 5% microsilica mortar has higher corrosive resistance than the 10% microsilica mortar. And these two mixes are more vulnerable to sulfuric acid than normal cement mortar. These results are opposite to the assumption that the

microsilica contributes acid resistance. The microsilica with 5% and 10% should have higher acid resistance than normal cement mortar. Also, the 5% microsilica mortar should have lower acid resistance than the 10% microsilica mortar. More parameters should be investigated to explain this phenomena.

**Table 4.9** Compressive Strength of Microsilica Mortar in  $H_2SO_4$  100 ml/l at Different Immersed Time

Sample No.	Compressive Strength at Different Ages (psi)					
	0-day	1-day	3-day	7-day	14-day	28-day
MC	8178.3	6012.5	5005.8	4619.2	2475.6	1879.1
MS5	7905.0	6269.2	5195.0	3719.7	4120.0	2277.0
MS10	7883.3	6632.5	5114.2	3559.2	3911.5	2520.2
MS50	2431.3	1646.3	1763.8	1337.5	639.2	0



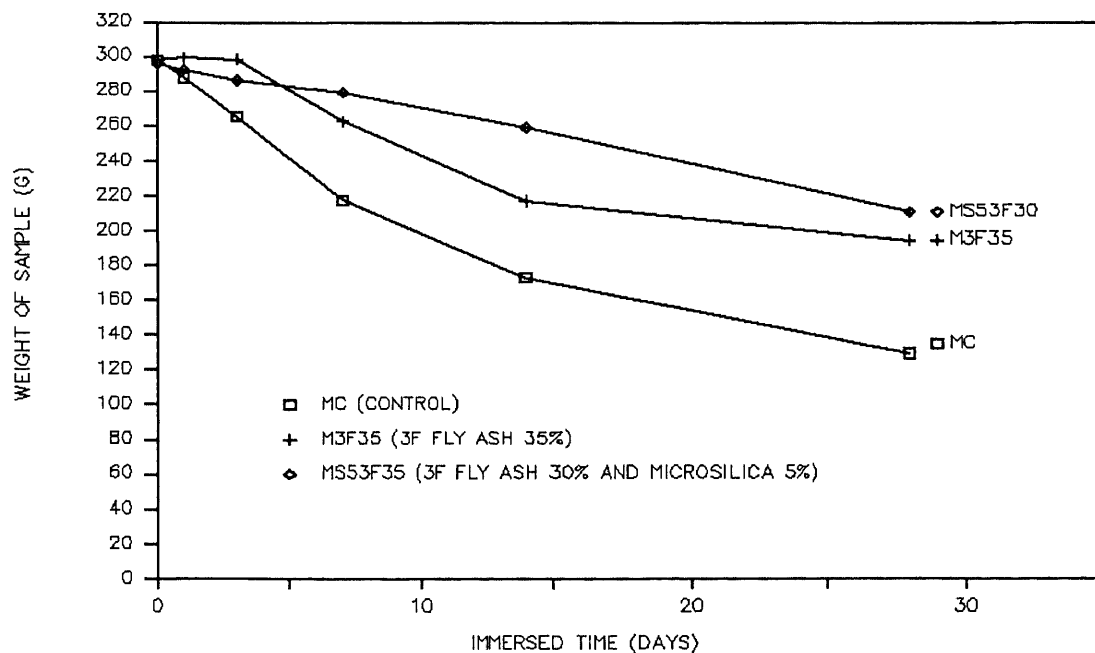
**Figure 4.13** Relationship between Compressive Strength of Microsilica Mortar in  $H_2SO_4$  100 ml/l at Different Percentage of Microsilica and Immersed Time

Table 4.9 presents their compressive strength. Figure 4.13 is the relationship between the compressive strength of all mixes and age. It can be seen from Figure

4.13 that the compressive strength of the low percentage microsilica mortar is higher than that of the high one. This can be explained by the lime available concept. Since the MS50 does not have enough  $\text{Ca(OH)}_2$  to react with  $\text{SiO}_2$  so there is less bond in it resulting in low compressive strength. Since this test program is made to improve compressive strength more than sulfuric resistance of fly ash mortar, the 5% microsilica is selected to mix with fly ash mortar.

**Table 4.10** Weight of Microsilica and Fly Ash Mortar in  $\text{H}_2\text{SO}_4$  100 ml/l at Different Immersed Time

Sample No.	Weight at Different Ages (psi)						Comp psi.
	0-day	1-day	3-day	7-day	14-day	28-day	
MC	298.1	288.0	265.2	217.6	173.1	129.3	8178.3
M3F35	298.4	299.5	298.5	262.5	217.0	194.6	5925.0
MS5F30	295.7	292.6	286.3	279.2	259.3	211.0	7975.0

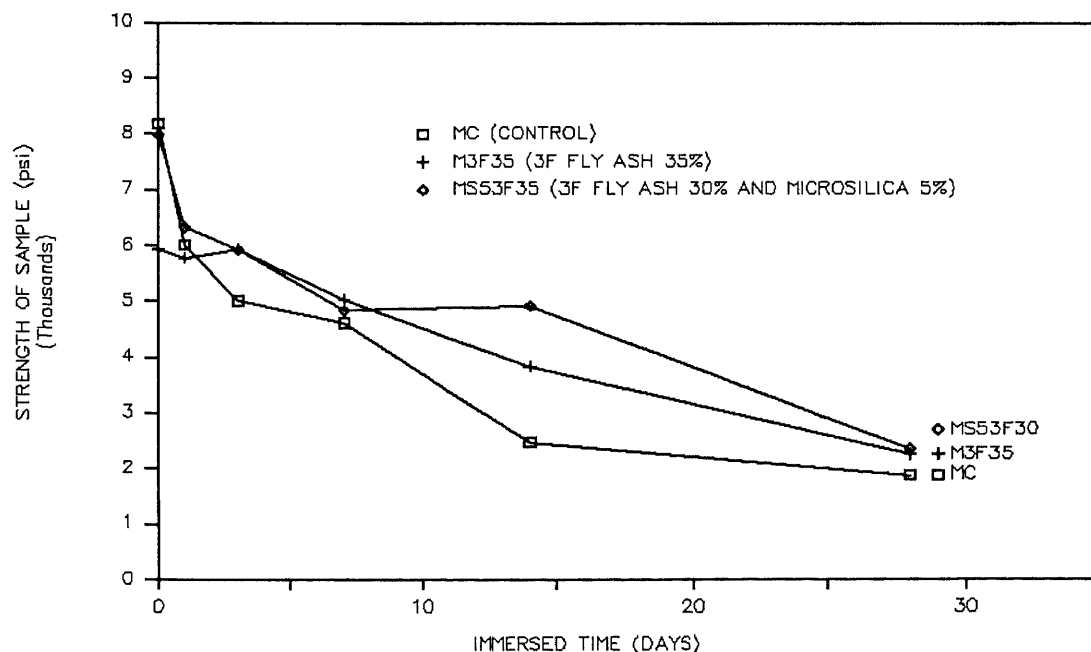


**Figure 4.14** Relationship between Weight of Microsilica Fly Ash Mortar and Fly Ash Mortar in  $\text{H}_2\text{SO}_4$  100 ml/l and Immersed Time When Using 35% Cement Replacement

Since the cementitious materials, sand, and water are constant, the properties of mortar depends on the characteristics of the cementitious materials, microsilica and fly ash. It can be seen from Figure 4.14 that MS53F30 has less weight loss than M3F35 almost every immersed time. At the same 35% cement replacement, weight loss of MS53F30 mortar at 28 days immersed time is 28.64% but that of M3F35 mortar is 35%. Therefore, using microsilica can improve the sulfuric acid resistance of fly ash mortar.

**Table 4.11** Compressive Strength of Microsilica and Fly Ash Mortar in  $H_2SO_4$  100 ml/l at Different Immersed Time

Sample No.	Compressive Strength at Different Ages (psi)					
	0-day	1-day	3-day	7-day	14-day	28-day
MC	8178.3	6012.5	5005.8	4619.2	2475.6	1879.1
M3F35	5925.0	5775.8	5931.3	5037.3	3836.7	2266.1
MS5F30	7975.0	6322.5	5928.8	4842.6	4923.9	2358.4



**Figure 4.15** Relationship between Compressive Strength of Microsilica Fly Ash Mortar and Fly Ash Mortar in  $H_2SO_4$  100 ml/l and Immersed Time When Using 35% Cement Replacement

Another benefit of microsilica is providing high initial strength mortar. It can be seen from Table 4.9 that 28-day strength of MS53F30 is higher than that of M3F35. But it does not reduce the strength reduction of fly ash mortar immersed in sulfuric acid as expected. It can be seen from Figure 4.15 that strength of microsilica fly ash mortar decreases substantially almost in the same rate as fly ash mortar, after immersed in sulfuric acid.

By using microsilica as cement replacement, it can be concluded that microsilica can improve both sulfuric acid resistance and initial compressive strength but it cannot reduce strength reduction of fly ash mortar immersed in sulfuric acid.

#### **4.4 Effect of Superplasticizer on Sulfuric Acid Resistance and Compressive Strength of Fly Ash Mortar**

Fly ash mortars with superplasticizer are mixed in parallel with fly ash mortars without superplasticizer. The selected mixes are PM3F35, PM3F50, PMOR35, PMOR50, PMM35, PMM50 and PMS53F35. The water cement ratio was 0.35. The mix proportion is shown in Table 3.1. All mixes were cast, cured and test as the same process as fly ash mortar. Comparison between weight of fly ash mortar with and without superplasticizer at different immersed time are shown in Figure A 27 to A 34 in Appendix A. Comparison between compressive strength of fly ash mortar with and without superplasticizer at different immersed time are shown in Figure A 35 to A 42 in Appendix A.

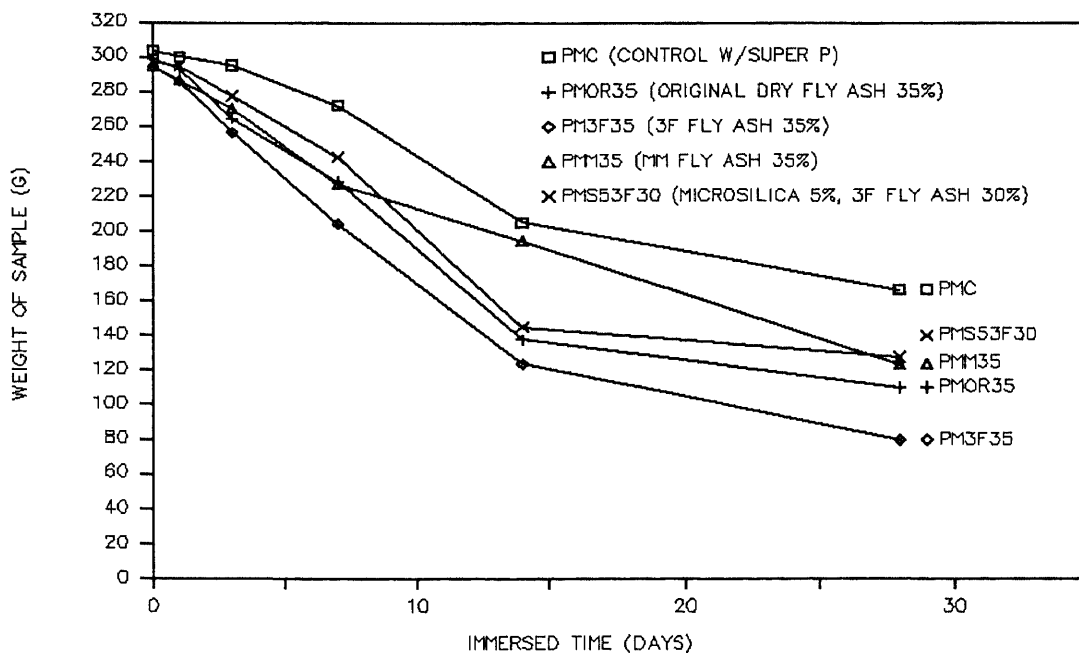
Superplasticizer, sulfonated naphthalene formaldehyde, is used in this test program. It is supposed to enhance compressive strength and reduce weight loss because it reduces water cement ratio to 0.35 causing a lower permeability. But the results show the adverse effect of superplasticizer. The weight and compressive strength of sample are lower than those of without superplasticizer. This may be resulted from the reaction between its chemical compounds and acid.

It can be seen that there is no pattern in Figure 4.16 and 4.17. It does not

show that using the finest fly ash or the high percentage of fly ash with superplasticizer can improve sulfuric acid resistance. The 3F fly ash mortar with 50% cement replacement which is the most sulfuric acid resistant mix in non superplasticizer serie has the high weight loss when it contains superplasticizer.

**Table 4.12** Weight of Fly Ash Mortar in  $H_2SO_4$  100 ml/l at Different Immersed Time with Superplasticizer (35% Cement Replacement)

Sample No.	Weight at Different Ages (psi)						Comp psi.
	0-day	1-day	3-day	7-day	14-day	28-day	
PMC	303.7	300.2	295.4	271.8	204.9	166.3	8786.7
PM3F35	294.6	285.8	256.7	204.1	123.9	79.9	7473.2
PMOR35	298.8	293.1	264.6	228.1	137.8	110.1	7842.5
PMM35	292.5	280.8	272.2	235.9	182.6	162.3	5413.3
PMS53F	297.4	294.9	277.5	242.3	144.9	127.8	8590.8

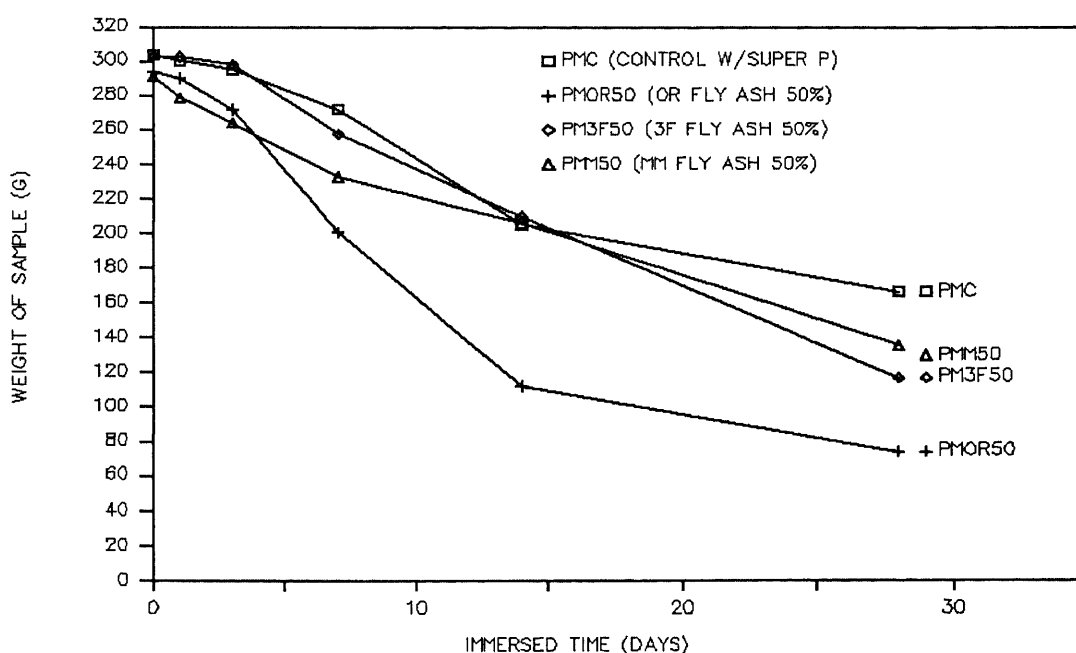


**Figure 4.16** Relationship between Weight of Fly Ash Mortar with Superplasticizer in  $H_2SO_4$  100 ml/l and Immersed Time When Using 35% Cement Replacement



**Table 4.13** Weight of Fly Ash Mortar in  $H_2SO_4$  100 ml/l at Different Immersed Time with Superplasticizer (50% Cement Replacement)

Sample No.	Weight at Different Ages (psi)						Comp psi.
	0-day	1-day	3-day	7-day	14-day	28-day	
PMC	303.7	300.2	295.4	271.8	204.9	166.3	8786.7
PM3F50	302.9	302.7	298.1	257.4	209.8	117.1	7264.2
PMOR50	293.8	290.2	271.6	200.7	112.3	74.1	6772.5
PMM50	291.2	278.9	264.0	232.7	205.9	135.6	6537.5



**Figure 4.17** Relationship between Weight of Fly Ash Mortar with Superplasticizer in  $H_2SO_4$  100 ml/l and Immersed Time When Using 50% Cement Replacement

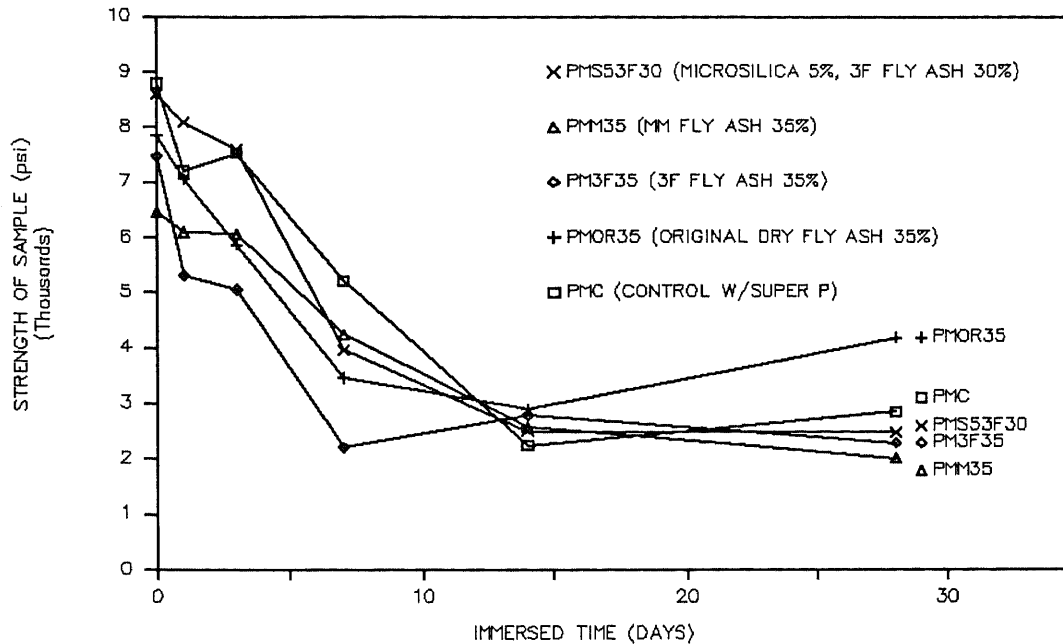
Moreover, the superplasticizer does not enhance the compressive strength of fly ash mortar in acid environment even though it has low water cement ratio, 0.35, both 35% and 50% cement replacement as shown in Figure 4.18, and 4.19, respectively. It reduces the strength even lower than normal cement mortar. It can be seen that the compressive strength of most of mortars at 28-day immersed time is

higher than those of 14-day immersed time. This may be because at 14 day the reaction forming non binding material occurred at the outer layer of mortar and it did not fall from the structure. At 28-day, these materials fell down and left the hard core giving the high strength with less surface area than the 14-day specimen. However, more investigation should be done to study the reaction of superplasticizer with sulfuric acid.

It can be concluded that this type of superplasticizer cannot enhance both of sulfuric acid resistance and compressive strength of fly ash mortar. Other types of superplasticizer should be investigated more to increase the compressive strength of the fly ash mortar in sulfuric acid.

**Table 4.14** Compressive Strength of Fly Ash Mortar in  $H_2SO_4$  100 ml/l at Different Immersed Time with Superplasticizer (35% Cement Replacement)

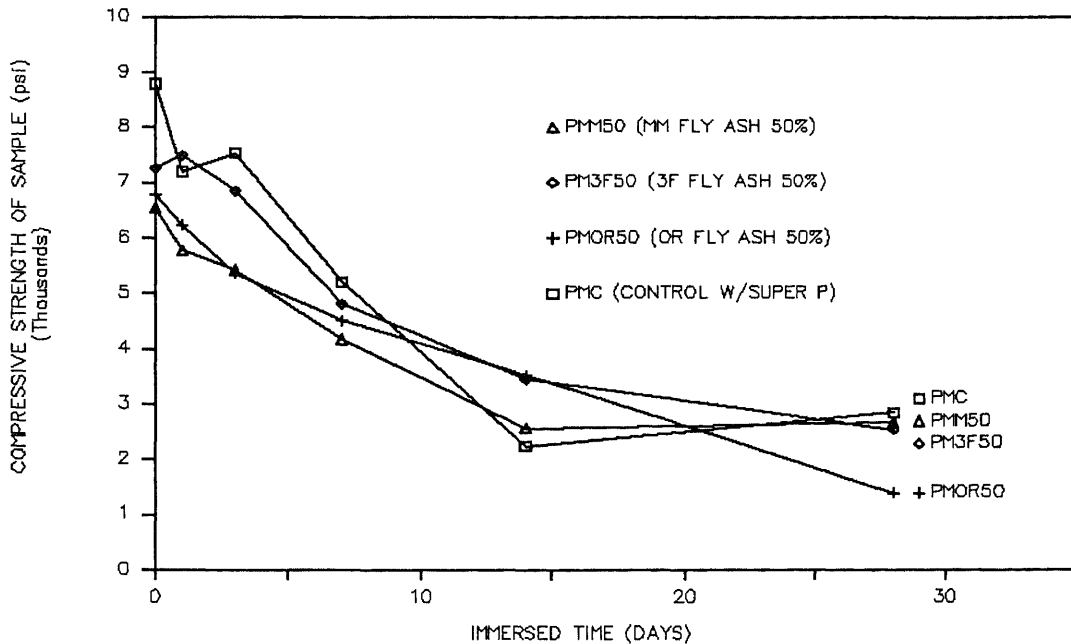
Sample No.	Compressive Strength at Different Ages (psi)					
	0-day	1-day	3-day	7-day	14-day	28-day
PMC	8178.3	6012.5	5005.8	4619.2	2475.6	1879.1
PM3F35	7473.2	5307.6	5053.8	2206.1	2789.8	2293.6
PMOR35	7842.5	7051.7	5862.5	3458.4	2903.4	4183.5
PMM35	6450.0	6091.7	6063.3	4242.0	2584.0	2018.7
PMS53F	8590.8	8081.7	7593.3	3966.7	2508.1	2485.9



**Figure 4.18** Relationship between Compressive Strength of Microsilica Fly Ash Mortar and Fly Ash Mortar with Superplasticizer in  $H_2SO_4$  100 ml/l and Immersed Time When Using 35% Cement Replacement

**Table 4.15** Compressive Strength of Fly Ash Mortar in  $H_2SO_4$  100 ml/l at Different Immersed Time with Superplasticizer (50% Cement Replacement)

Sample No.	Compressive Strength at Different Ages (psi)					
	0-day	1-day	3-day	7-day	14-day	28-day
PMC	8178.3	6012.5	5005.8	4619.2	2475.6	1879.1
PM3F50	7264.2	7491.7	6842.5	4815.1	3467.7	2542.5
PMOR50	6772.5	6237.5	5365.8	4515.6	3525.0	1401.1
PMM50	6537.5	5787.5	5420.8	4177.9	2574.3	2677.9

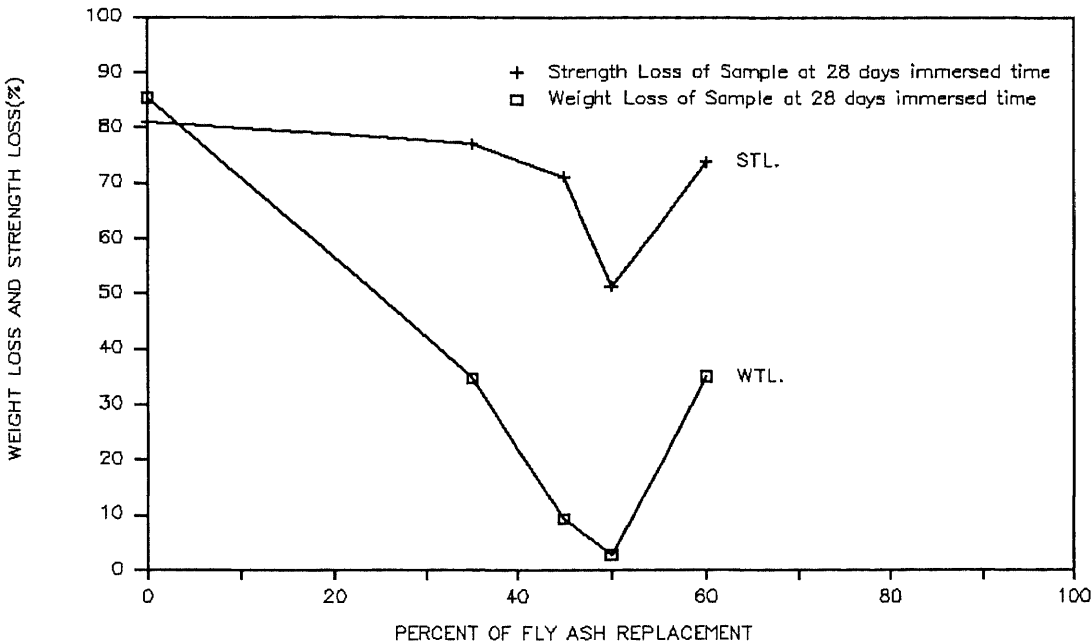


**Figure 4.19** Relationship between Compressive Strength of Microsilica Fly Ash Mortar and Fly Ash Mortar with Superplasticizer in  $H_2SO_4$  100 ml/l and Immersed Time When Using 50% Cement Replacement

#### 4.5 Optimum Mix of Fractionated Fly Ash Mortar .

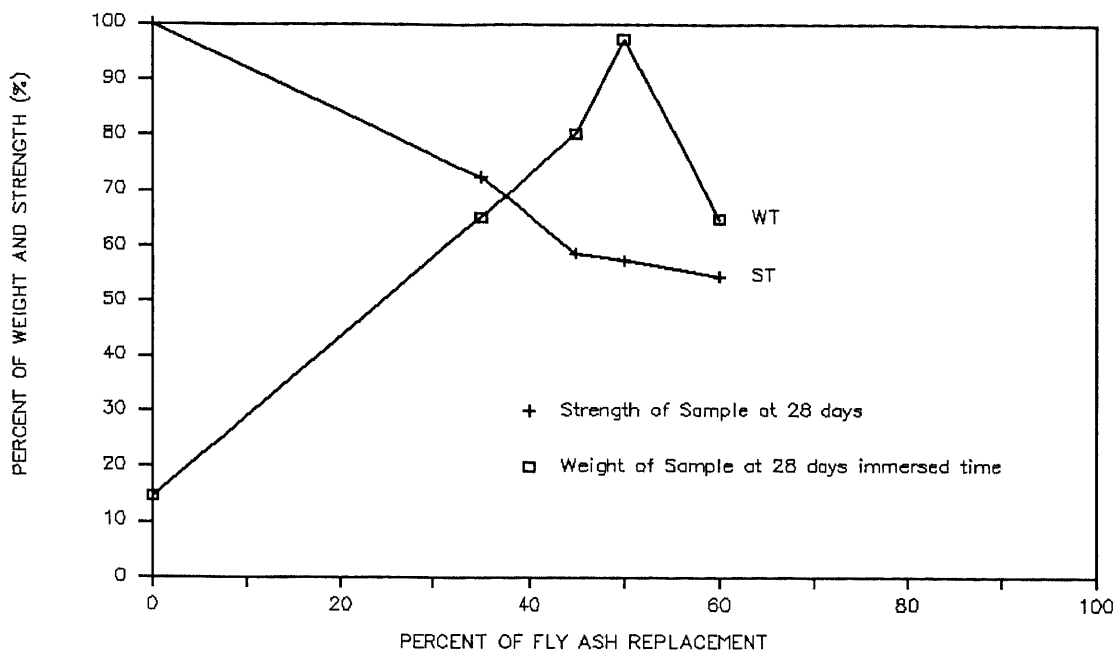
In this study, optimum fly ash mortar is proposed. The desired properties are acceptable compressive strength and well corrosive resistance. The corrosion resistant property of fly ash mortar can be obtained from either weight loss or strength loss. However, no standard of corrosion resistance is proposed. Thus this study obtained optimum value of these durability by comparing to other mortars.

It can be seen from Figure 4.20 that the weight loss and strength loss of the 50% fly ash mortar at 28 days immersed time is the lowest compared to the other mixes. Thus it is considered to be the optimum mix for using in high corrosive environment.

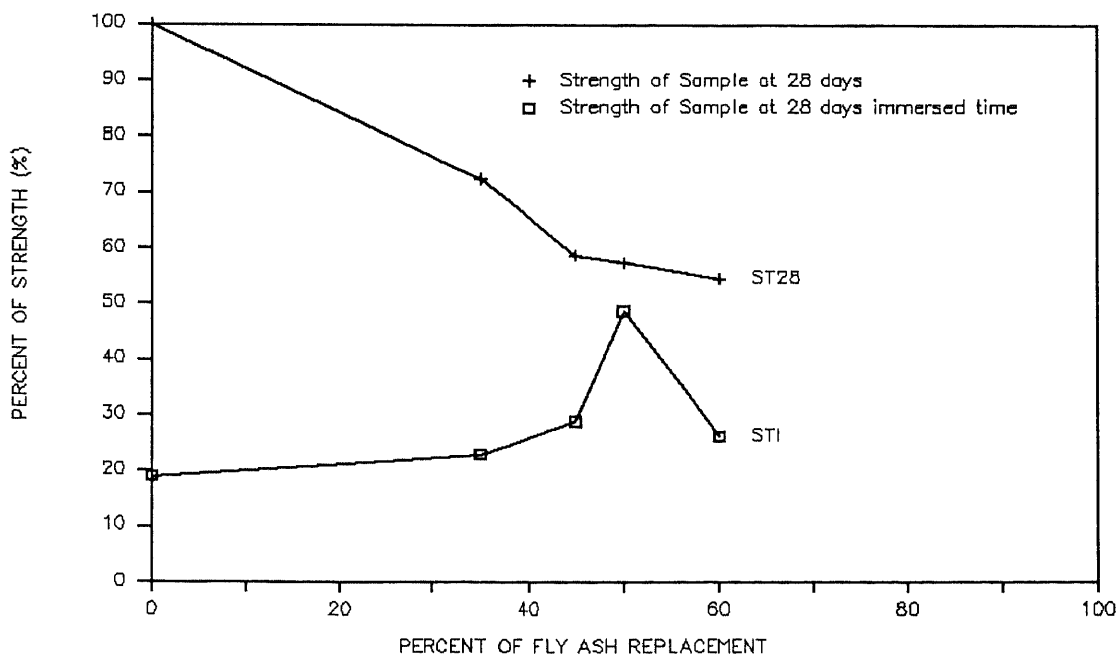


**Figure 4.20** Relation between Compressive Strength and Weight of 3F Fly Ash Mortar Sample in  $H_2SO_4$  100 ml/l 28 Days Immersed Time and Different Percentage of Fly Ash

Another concerned parameter is its compressive strength at 28 days. Although it can resist acid attack but if it has unacceptable strength, it could be destroyed by other ways. Also, It can be seen from Figure 4.22 that the strength of the 50% fly ash mortar at 28 days immersed time is the highest compared to the other mixes. However its 28-day compressive strength is quite low, 56% of the control mortar. It should be improved strength by using other admixtures. If the 28-day compressive strength of the 50% fly ash mortar is elevated and the strength loss is lowered, this percentage of fly ash will be used effectively in practical.



**Figure 4.21** Relationship between Compressive Strength at 28 Days and Weight of 3F Fly Ash Mortar Sample at 28 Days Immersed Time in  $H_2SO_4$  100 ml/l and Different Percentage of Fly Ash



**Figure 4.22** Relationship between Compressive Strength at 28 Days and Compressive Strength of 3F Fly Ash Mortar Sample at 28 Days Immersed Time in  $H_2SO_4$  100 ml/l at Different Percentage of Fly Ash

#### 4.6 Strength Contribution of Fly Ash Mortar

In addition to the study of strength contribution in fly ash mortar, river sand and fly ash are used as a cement replacement in this test program. These mortars were mixed in parallel. Standard 2-inch cubes were cast and cured in saturated lime water about 28 days. The mix proportions used are tabulated in Table 3.1. The percentage of sand and fly ash used in the mixed were 15, 25 and 50 percent by weight of cementitious materials. For mortar with sand replacement, the cementitious material means a summation of cement and replacing sand. The water to cementitious material ratio of all mixes was kept constant at 0.50. No other admixtures were used in this program. Sample designated "MC" is the control mix which contains 100% ordinary portland cement type I, "3F" is the mix which contains finest fractionated fly ash, "S" is the mix which sand was used as replacement. The number "15", "25" and "50" stand for the percentage of cement replaced by fly ash. The compressive strength of samples were tested at the age of 1, 3, 7, 14, and 28 days.

**Table 4.16** Compressive Strength of Mortar with Fly ash or River Sand as a Replacement at Different Age

Sample No.	Compressive Strength at Different Ages(psi)				
	1-day	3-day	7-day	14-day	28-day
MC	2850.0	5216.0	7006.0	7883.0	9095.0
M3F15	2666.0	5002.0	6771.0	7968.0	9415.0
M3F25	2106.0	4248.0	5988.0	7336.0	8882.0
M3F50	1322.3	2552.0	3545.0	4062.0	5127.0
S15	1475.8	3600.8	4187.5	4610.0	5316.7
S25	1217.5	2822.5	3700.8	3920.0	4263.8
S50	375.0	919.2	1483.3	1615.8	1629.2

**Table 4.17** Percentage of Compressive Strength of Mortar with Fly ash or River Sand as a Replacement at Different Age

Sample No.	Percentage Compressive Strength (%)				
	1-day	3-day	7-day	14-day	28-day
MC	100.0	100.0	100.0	100.0	100.0
M3F15	93.5	95.9	96.6	101.1	103.5
M3F25	73.9	81.4	85.5	93.1	97.7
M3F50	46.4	48.9	50.6	51.5	56.4
S15	51.8	69.0	59.8	58.5	58.5
S25	42.7	54.1	52.8	49.7	46.9
S50	13.2	17.6	21.2	20.5	17.9

The compressive strength of the fractionated fly ash mortar and sand mortar with different percentage replacement of cement are shown in Table 4.16. Table 4.17 lists the percentage compressive strength of fractionated fly ash mortar and sand mortar compared to the control mortar. The relationship between the compressive strength of fly ash mortar and sand mortar with 15%, 25%, and 50% replacement of cement and age are shown in Figure 4.23, 4.24, and 4.25, respectively. Complete illustrations of their compressive strengths are shown in Figure A 43 to A 49 in Appendix A.

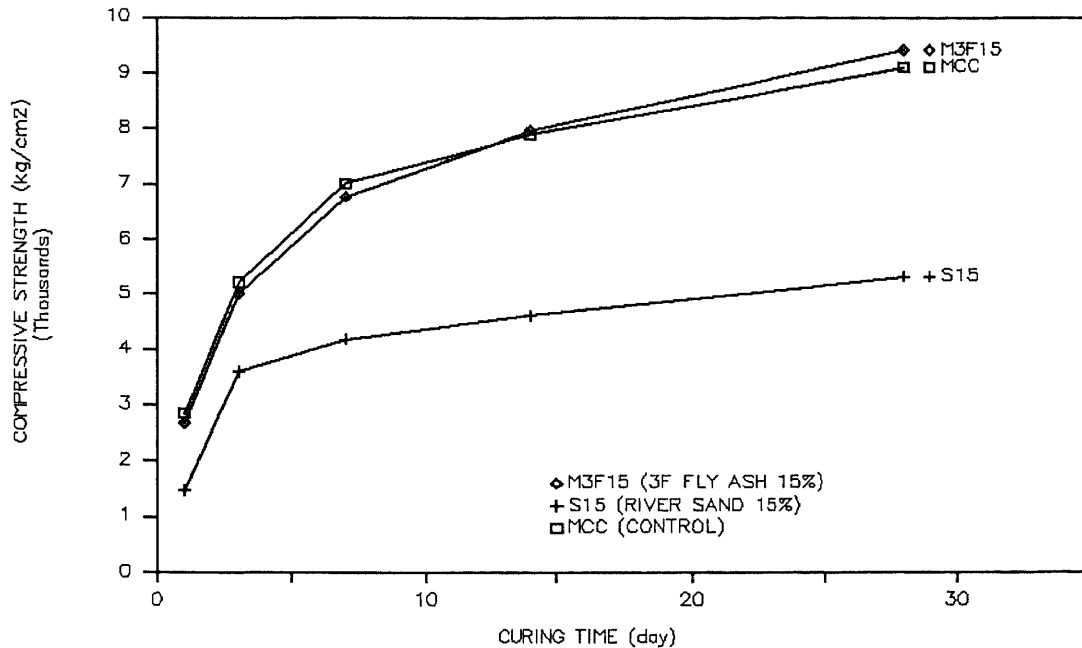
Compressive strength is contributed from physical and chemical mechanisms. For physical, it is already known that the finer the fineness of composition in sample, the higher the strength it gains. It provides high surface area for reaction and fills the space between particle.

Chemically, normal cement mortar gains strength from hydration reaction of cementitious compounds,  $C_3S$ ,  $C_2S$ , and so on. This reaction occurs mostly at early age of normal mortar. But for fly ash mortar, it occurs in slower rate than normal



cement mortar because it has high amount of nonreactive compound.

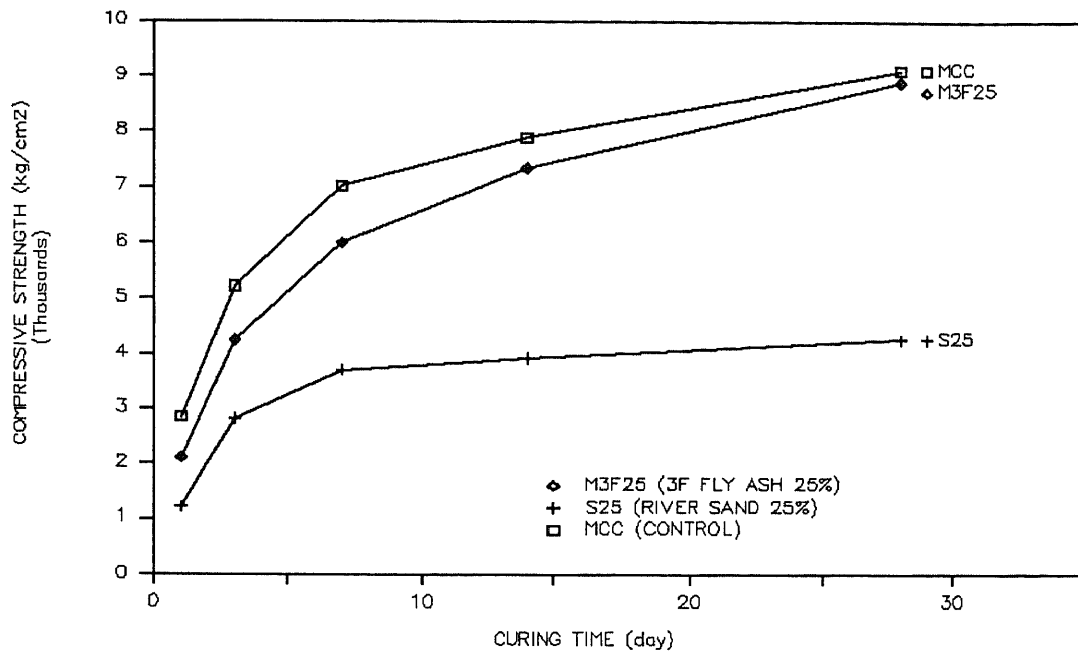
In this test program, the major strength contribution is assumed to gain from chemical mechanism. Thus, sand mortar gains strength from cement only. Fly ash mortar gains strength from both cement and fly ash. Therefore, the deduction between strength of fly ash mortar and sand mortar should be a contribution strength from fly ash. However this value is somewhat higher than expected due to the lower strength of sand mortar. This is because of the physical effect. The fineness of sand is lower than fly ash. Also, the hydration process is retarded by coarse sand.



**Figure 4.23** Relationship between Compressive Strength of Fly Ash Mortar and Sand Mortar and Age (15% Replacement)

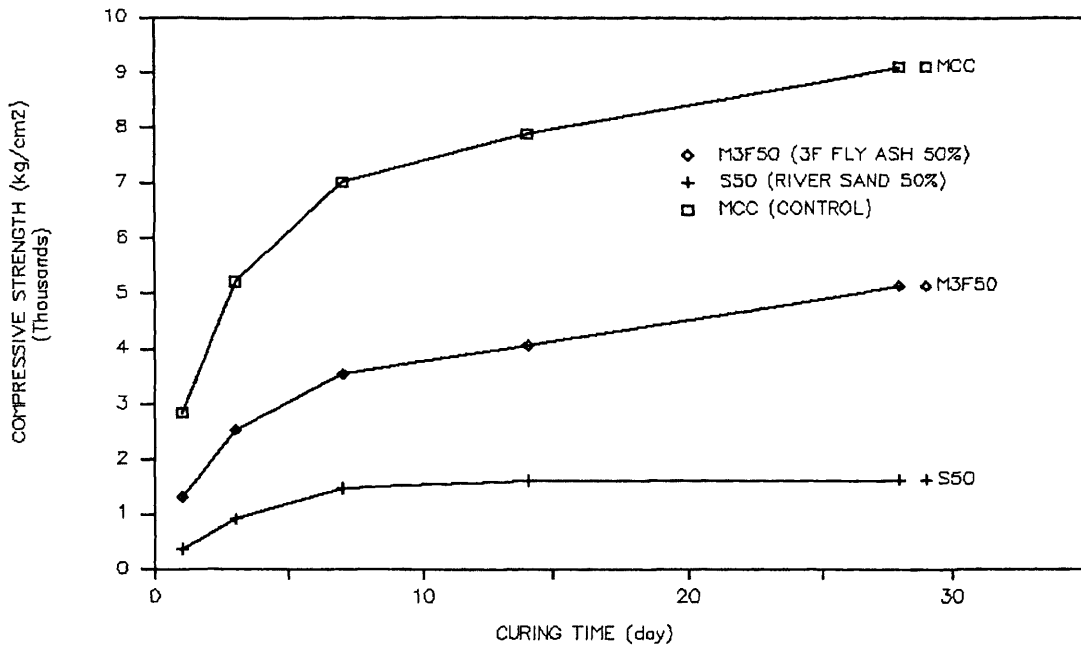
As expected the early age strength of the M3F15 mortar is lower than the control mortar. The compressive strength of fractionated fly ash mortar gradually increases with age. At 14 days, the compressive strength of sample M3F15 is equal to control sample and continues to higher after that. The strength contribution of

15% fly ash are 36.8% at 7 days and 45.0% at 28 days. Meanwhile, that of 85% cementitious compounds are 59.8% and 58.5% of control mortar at 7 days and 28 days, respectively. Thus, fly ash contributes strength to mortar in considerable value at first stage.



**Figure 4.24** Relationship between Compressive Strength of Fly Ash Mortar and Sand Mortar and Age (25% Replacement)

With a high percentage of fly ash in the mix the compressive strength of the M3F25 fly ash mortar is lower than that of M3F15. The compressive strength of M3F25 almost reaches that of the control mortar at 28 days, 97.7%. As stated earlier, the strength of this fly ash mortar comes from two source; cement and fly ash. The strength contribution from 75% cementitious compound is about 51.8% at 7 days and 46.9% at 28 days. And it does not give much high strength to the sample after this age. Fly ash, on the other hand, still contributes the strength in high rate after 7 days. The 25% fly ash gives 32.7% and 50.8% of control mortar at 7 days and 28 days, respectively.



**Figure 4.25** Relationship between Compressive Strength of Fly Ash Mortar and Sand Mortar and Age (50% Replacement)

It can be seen from Figure 4.25 that the strength of S50 reaches the maximum value at the age of 7 days. Then it tends to be constant. This is believed that the hydration of cement is ended up and gives strength at 21.2% of control mortar. Unlike M3F50 fly ash mortar, its strength tends to increase gradually from this point, 7 days, since the free lime which is absorbed by fly ash reacts with fly ash and gives strength. The rate of this reaction is not as high as the previous samples since there is lower amount of lime in the mix. The strength contribution of 50% fly ash are 29.4% and 38.5% of control mortar at 7 days and 28 days, respectively.

By comparing strength contribution of different percentage of fly ash, it can be concluded that the higher the percentage of fly ash is used, the lower the strength it contributes to the mortar. Also, its strength contribution progresses with time.

## CHAPTER 5

### CONCLUSIONS AND SUGGESTIONS

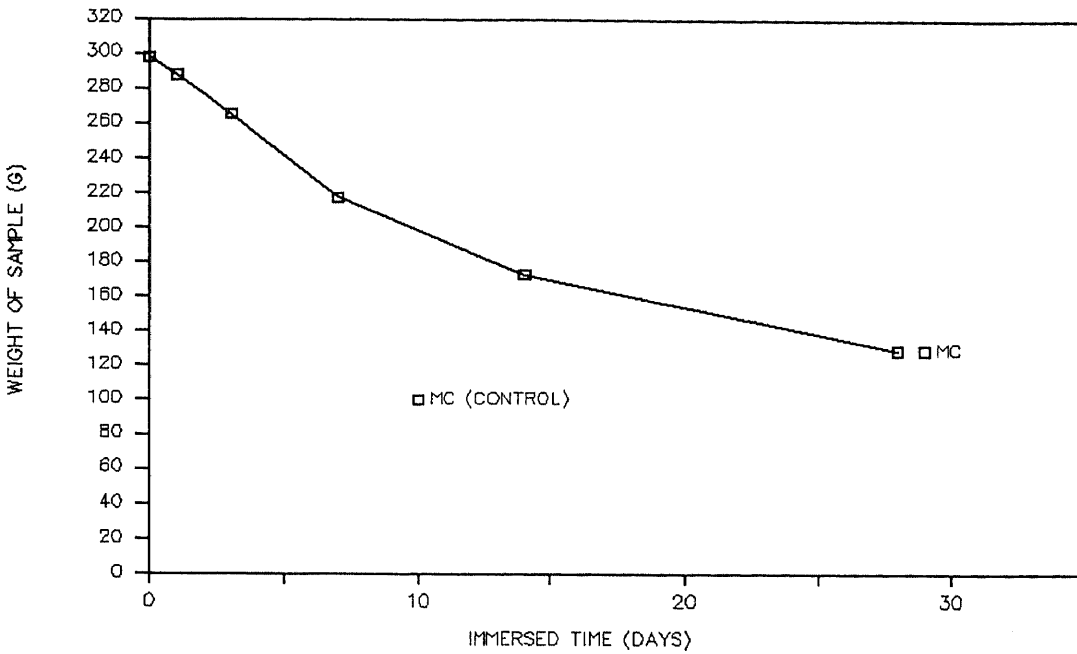
The results obtained through this investigation can lead to the following conclusions:

1. The chemical and physical properties of fly ash have significant effect on corrosion resistance against acid attack. The finest fly ash, 3F showed a better resistance than the original feed fly ash, OR. The Mae Moh fly ash mortars has less sulfuric acid resistance than the fly ash from the United States.
2. Fly ash can be used effectively to improve sulfuric acid resistance in mortar. Up to the limited value, the higher the volume of fly ash used in the mix, the higher the sulfuric acid resistance it provides. The percentage of fly ash should be proportional to the cement content of that mix to give the highest durability.
3. Microsilica can be used as a cement replacement to improve both corrosion resistance and initial compressive strength of fly ash mortar but it does not prevent the strength loss of fly ash mortar due to the attack from sulfuric acid.
4. Superplasticizer, sulfonated naphthalene formaldehyde, cannot be used as an admixture to improve sulfuric acid resistance of mortar because it increases the deterioration. Other superplasticizers which have different chemical composition should be investigated.
5. The amount of fly ash needed in the mix to provide for sulfuric acid resistance is about 50%. With these high volume content of fly ash in the mix, the fly ash mortar samples exhibits excellent sulfuric acid resistance. However, the strength reduction of this mix during immersed in sulfuric acid is still high.

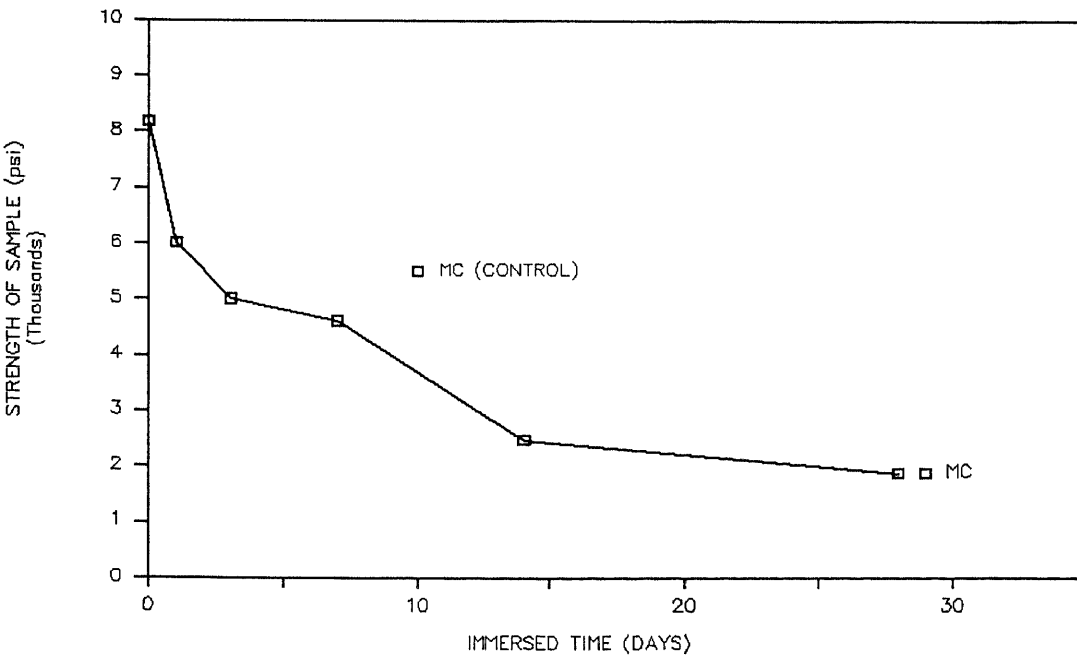
Thus other methods such as lowering water cement ratio should be investigated to improve its strength in acid.

6. It is founded that the higher percentage of fly ash is used as cement replacement, the lower it contributes compressive strength to the mix. It is also noted that for each fly ash percentage used, the higher the age of the mix is, the more compressive strength the fly ash provides.

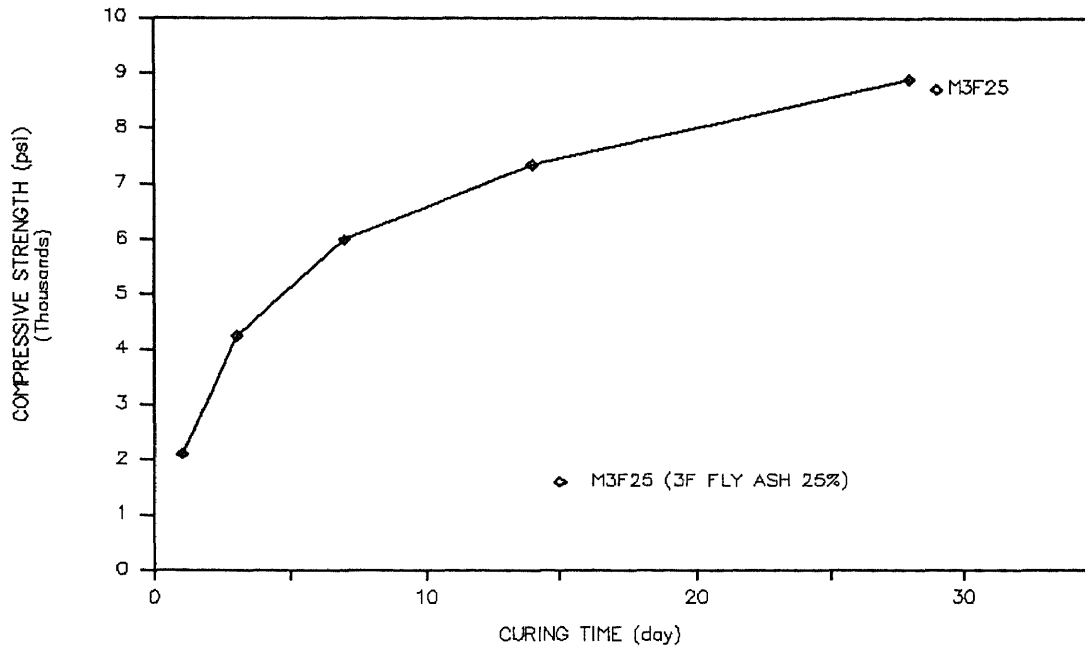
## APPENDIX A



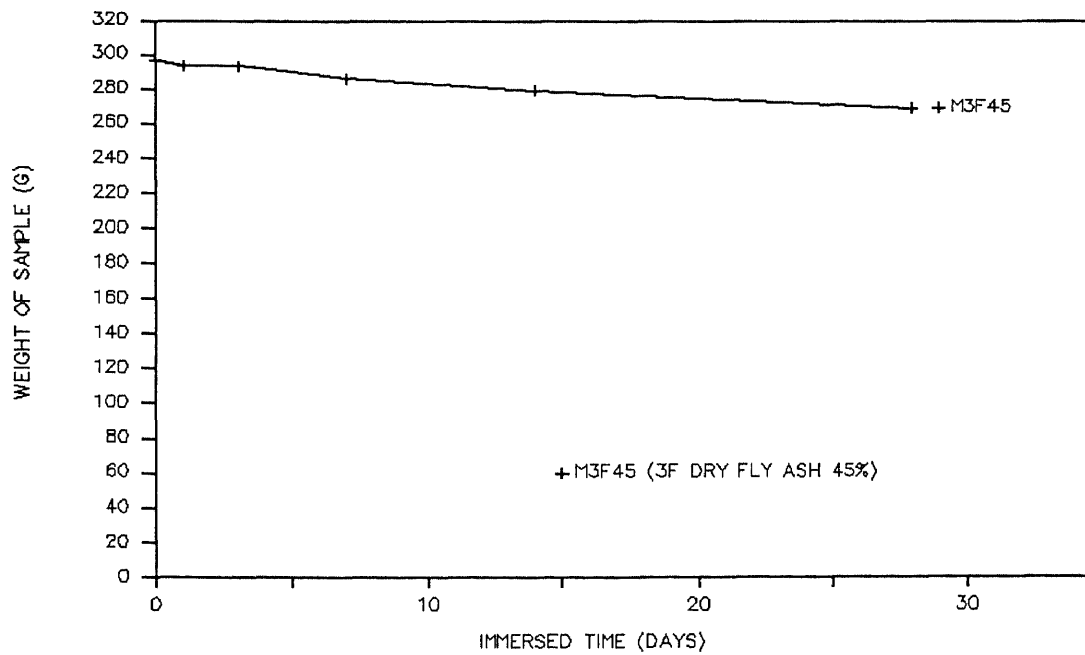
**Figure A 1** Weight of Normal Cement Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l



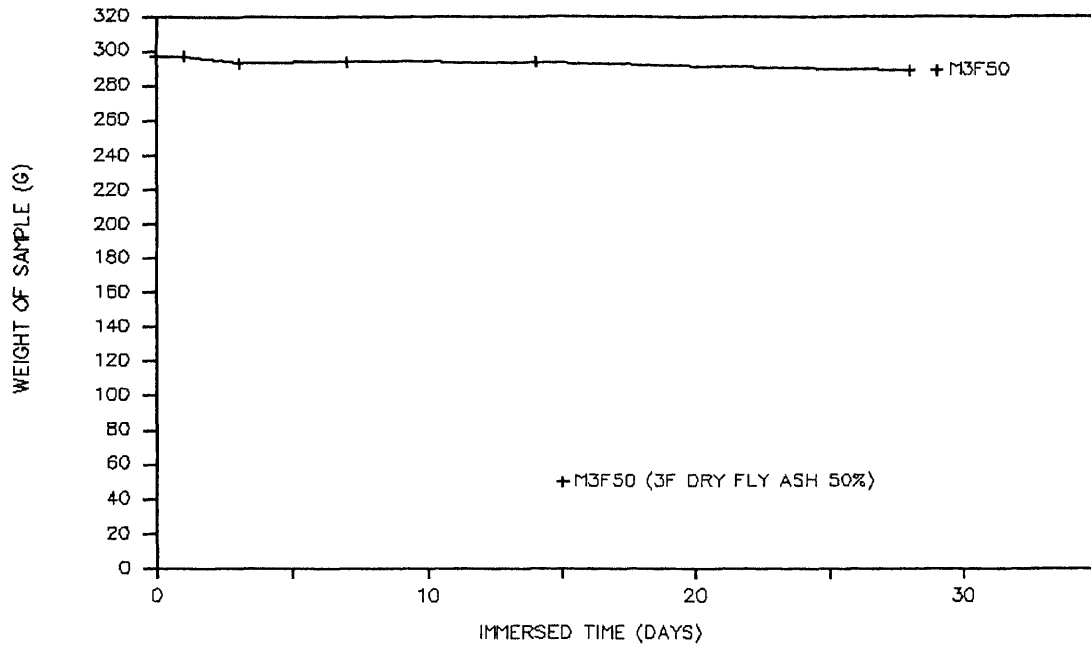
**Figure A 2** Compressive Strength of Normal Cement Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l



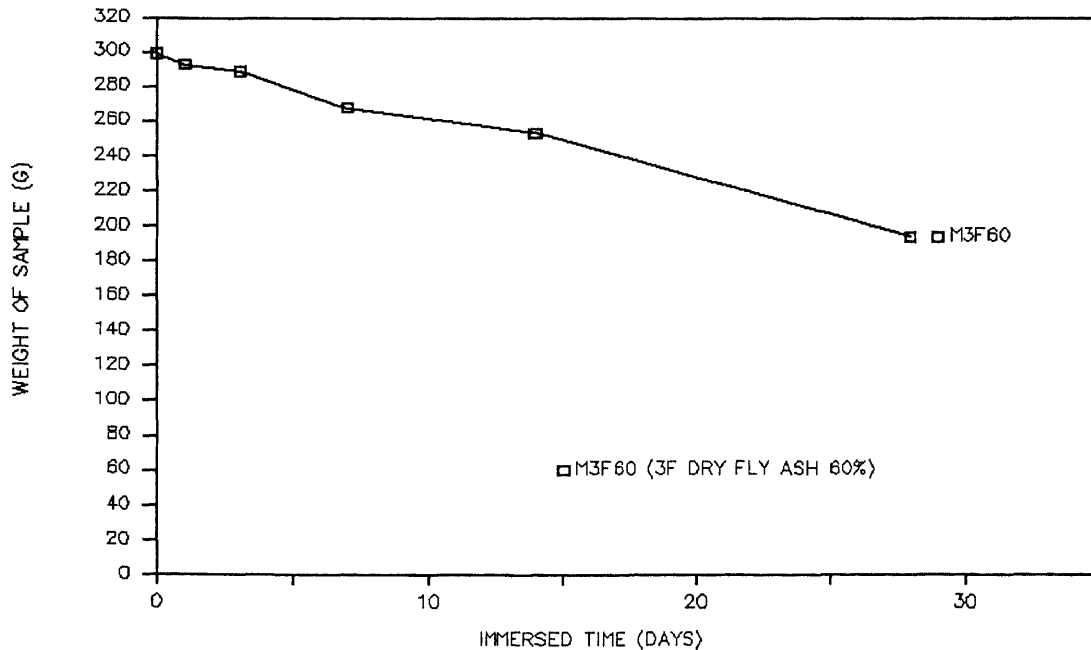
**Figure A 3** Weight of 3F Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (35% Replacement)



**Figure A 4** Weight of 3F Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (45% Replacement)

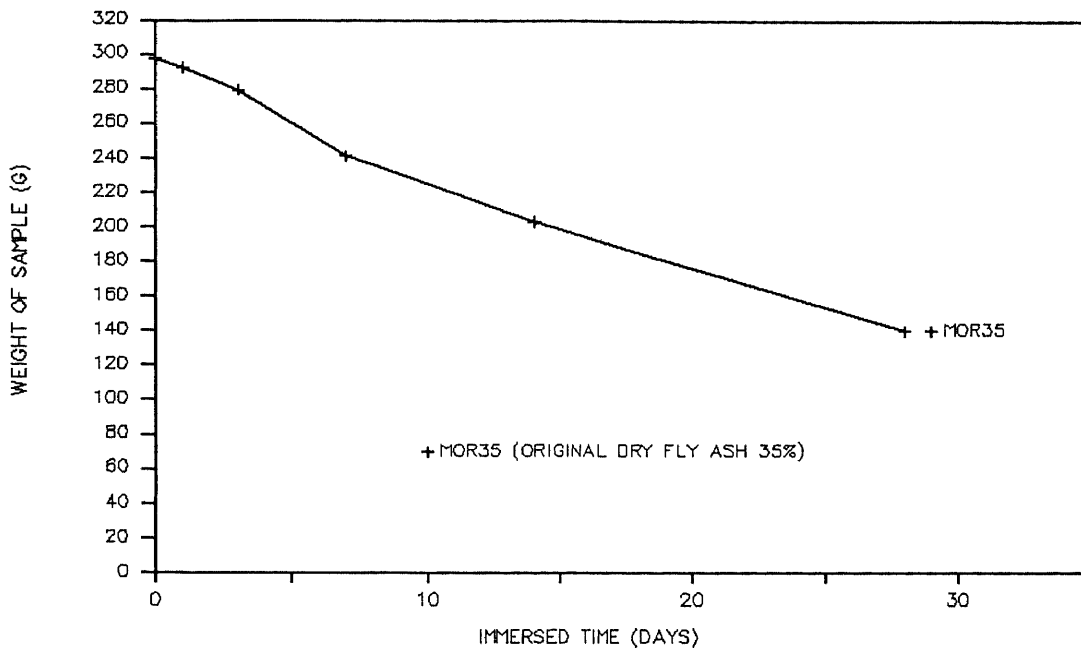


**Figure A 5** Weight of 3F Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l (50% Replacement)

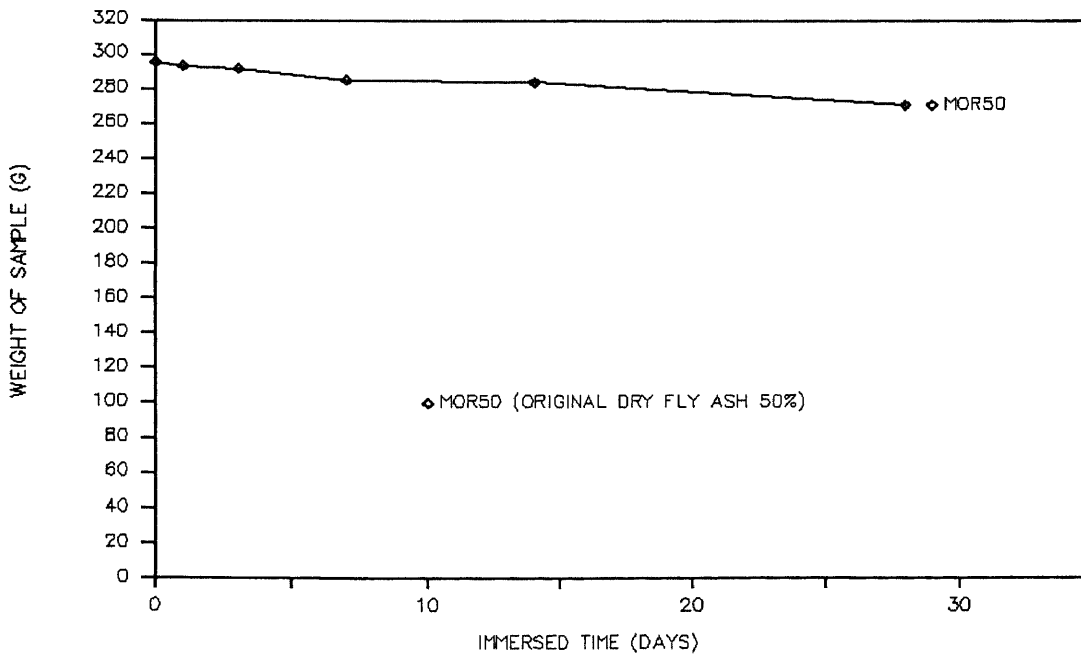


**Figure A 6** Weight of 3F Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l (60% Replacement)

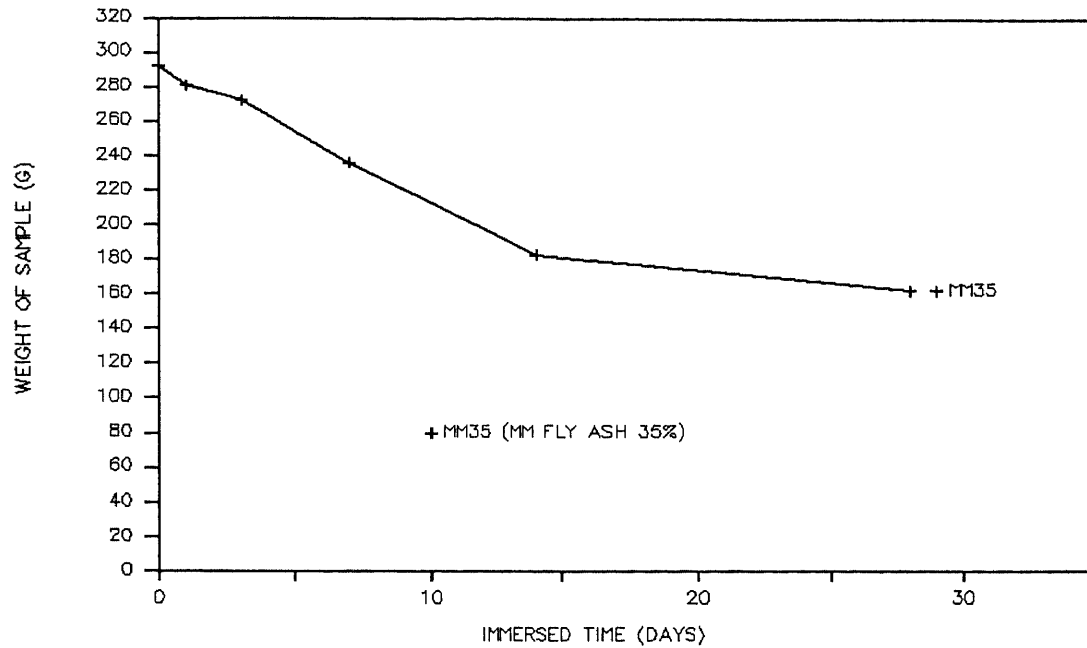




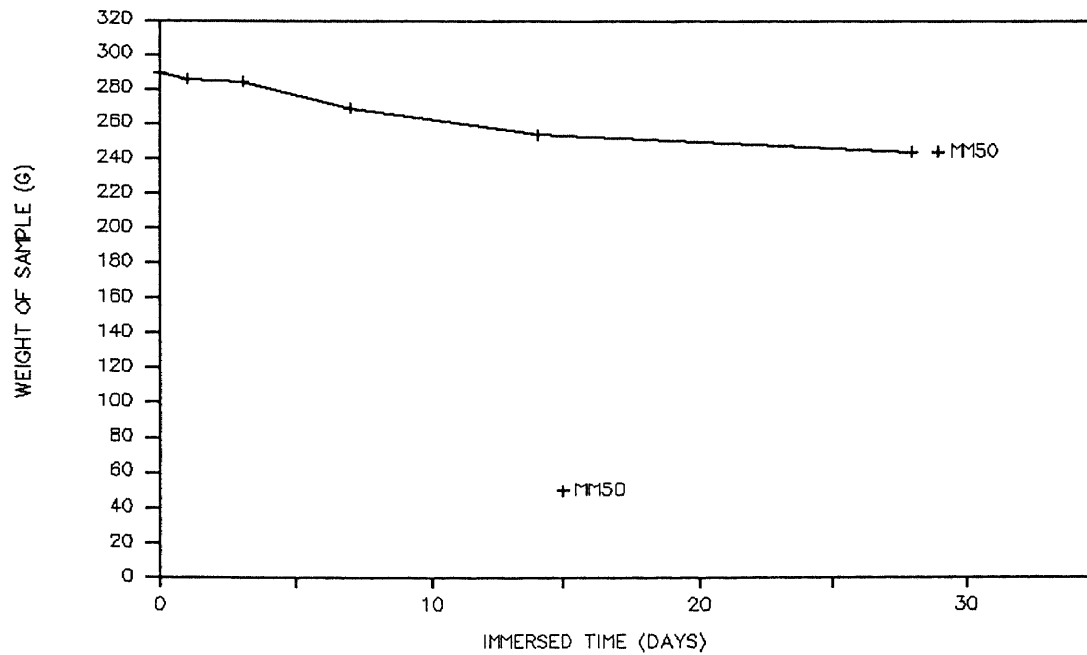
**Figure A 7** Weight of OR Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (35% Replacement)



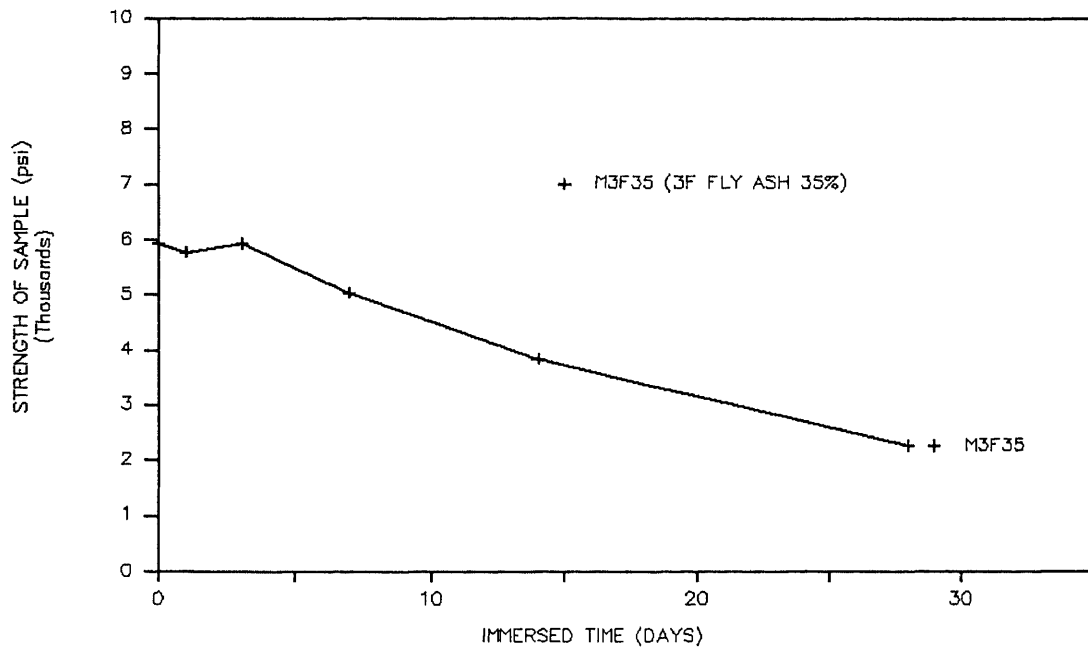
**Figure A 8** Weight of OR Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (50% Replacement)



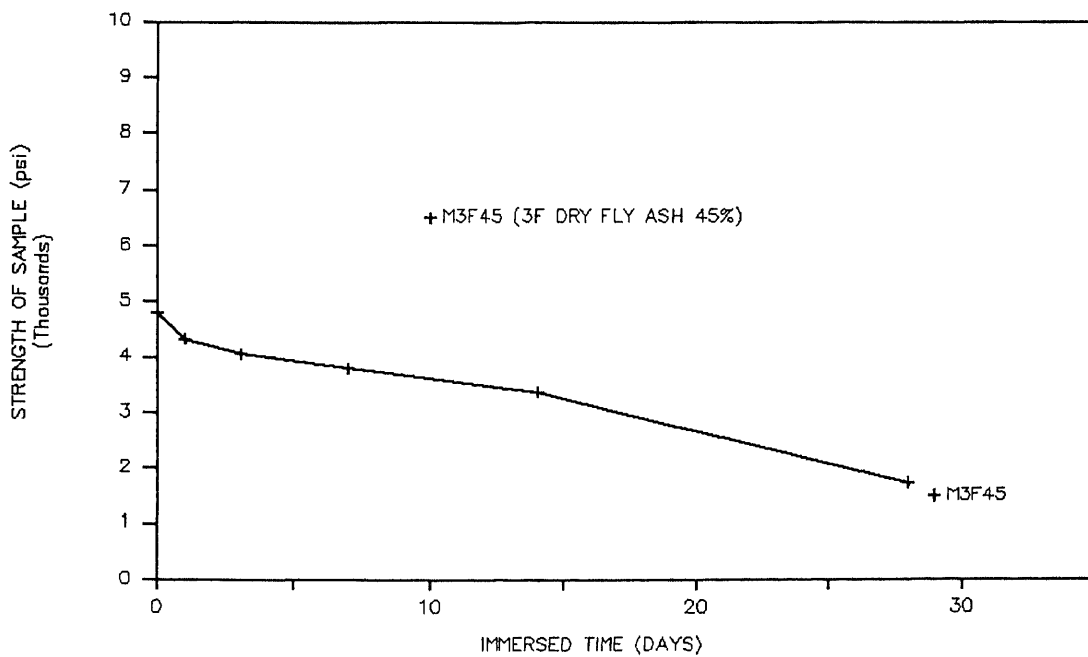
**Figure A 9** Weight of MM Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l (35% Replacement)



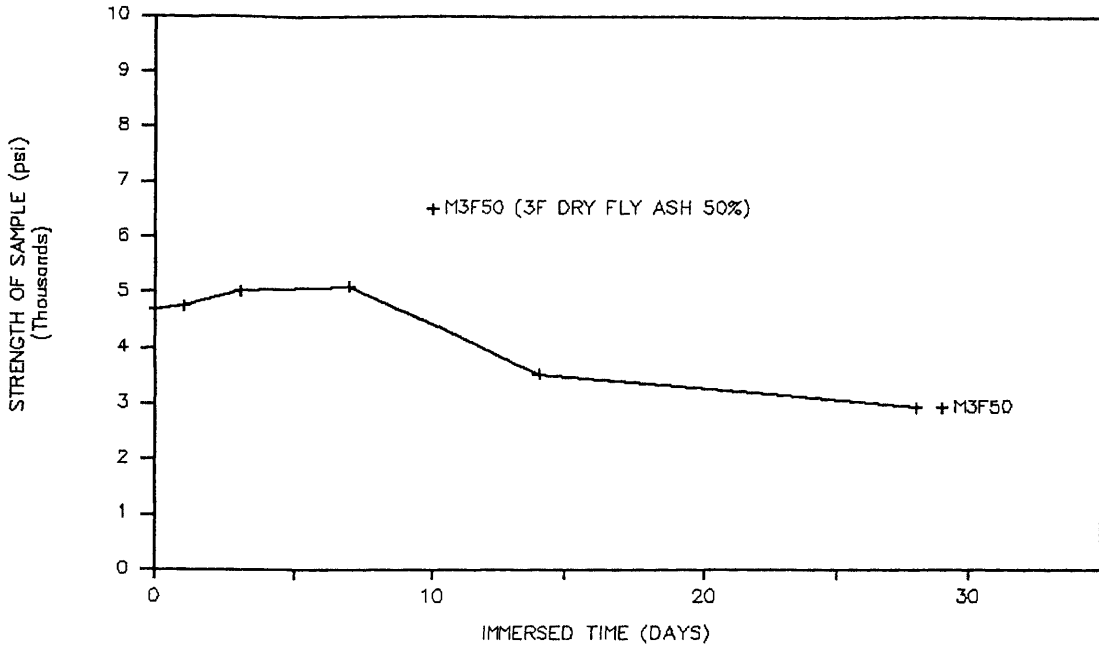
**Figure A 10** Weight of MM Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l (50% Replacement)



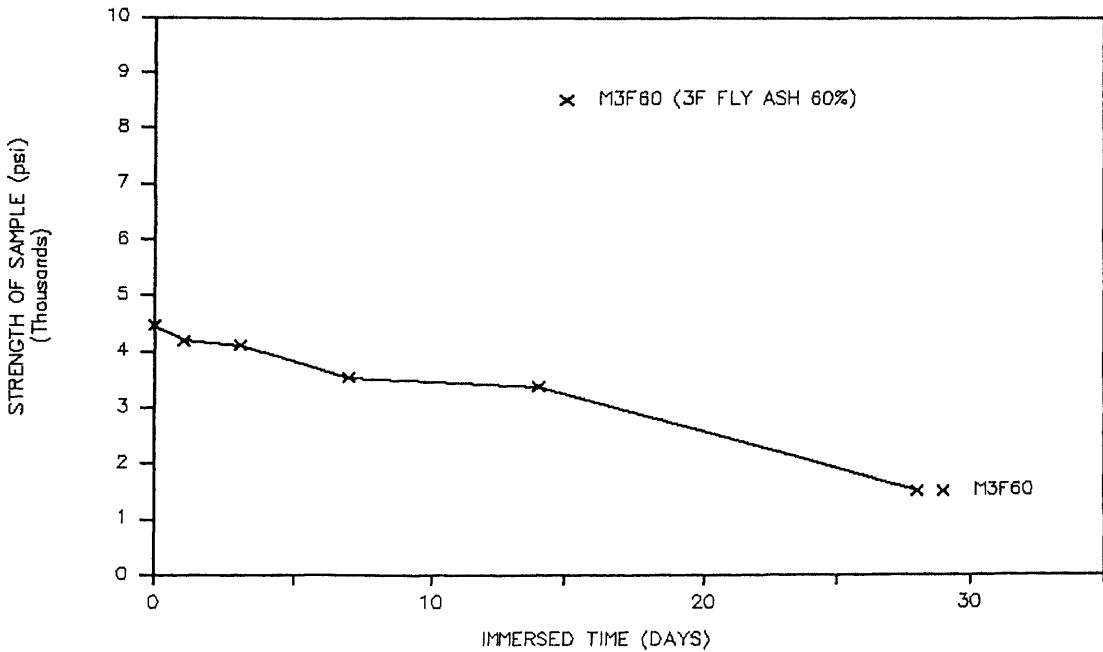
**Figure A 11** Compressive Strength of 3F Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l (35% Replacement)



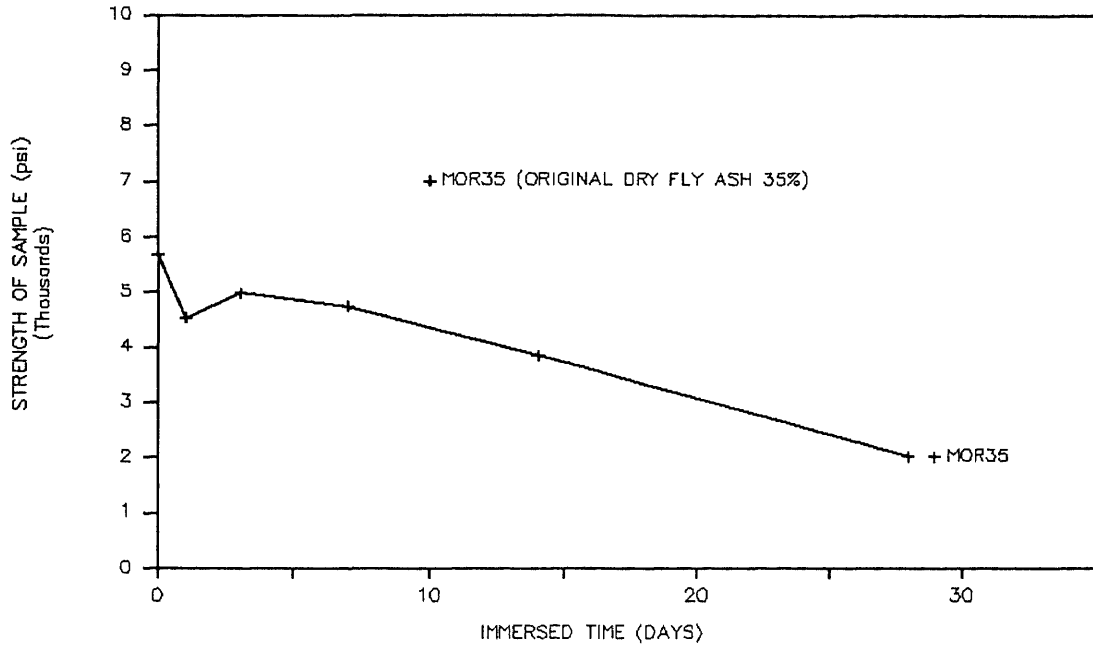
**Figure A 12** Compressive Strength of 3F Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l (45% Replacement)



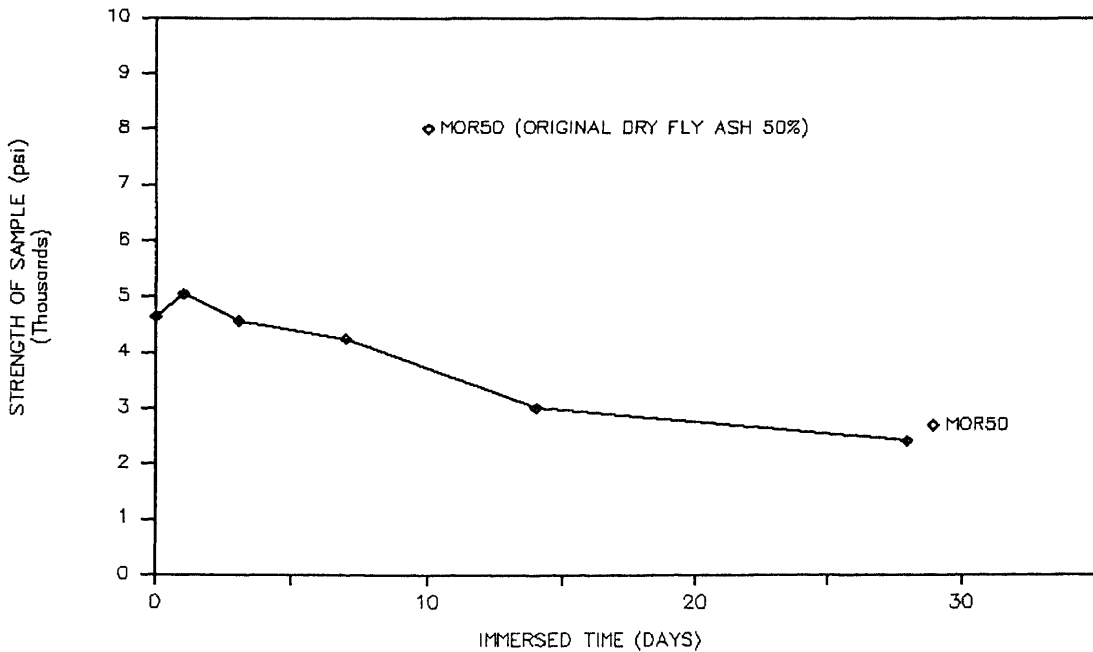
**Figure A 13** Compressive Strength of 3F Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (50% Replacement)



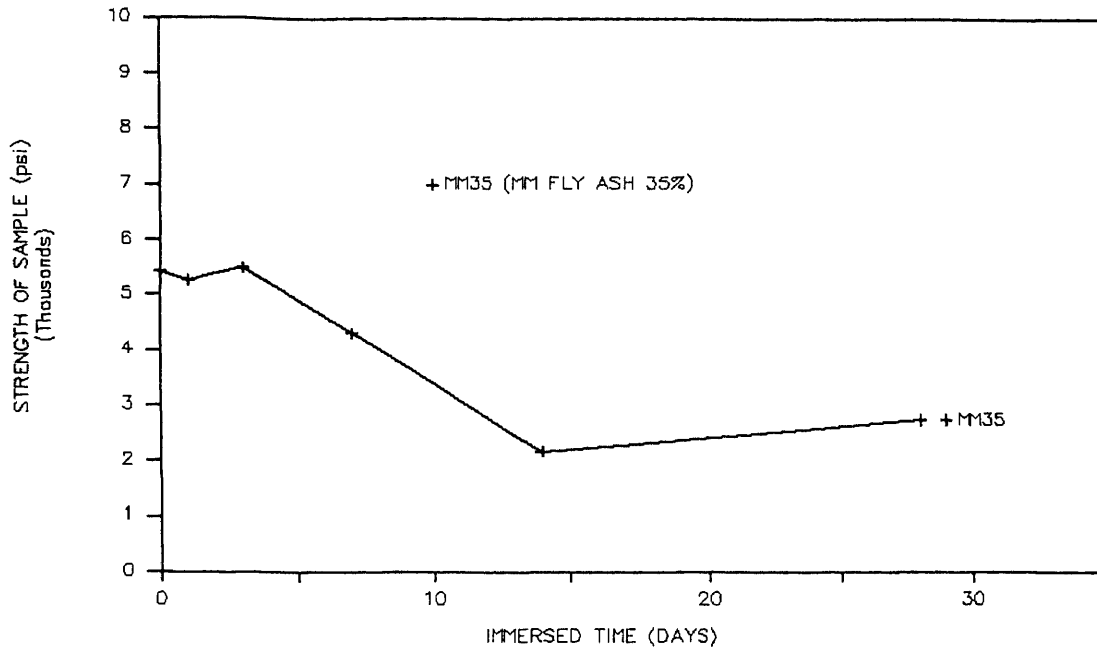
**Figure A 14** Compressive Strength of 3F Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (60% Replacement)



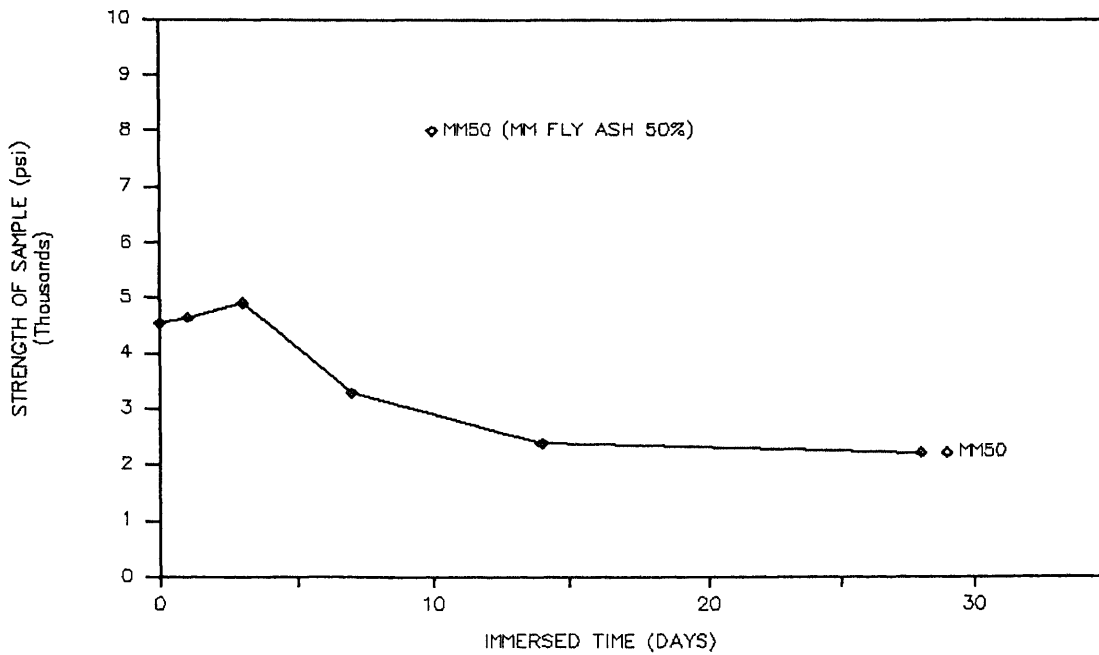
**Figure A 15** Compressive Strength of OR Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (35% Replacement)



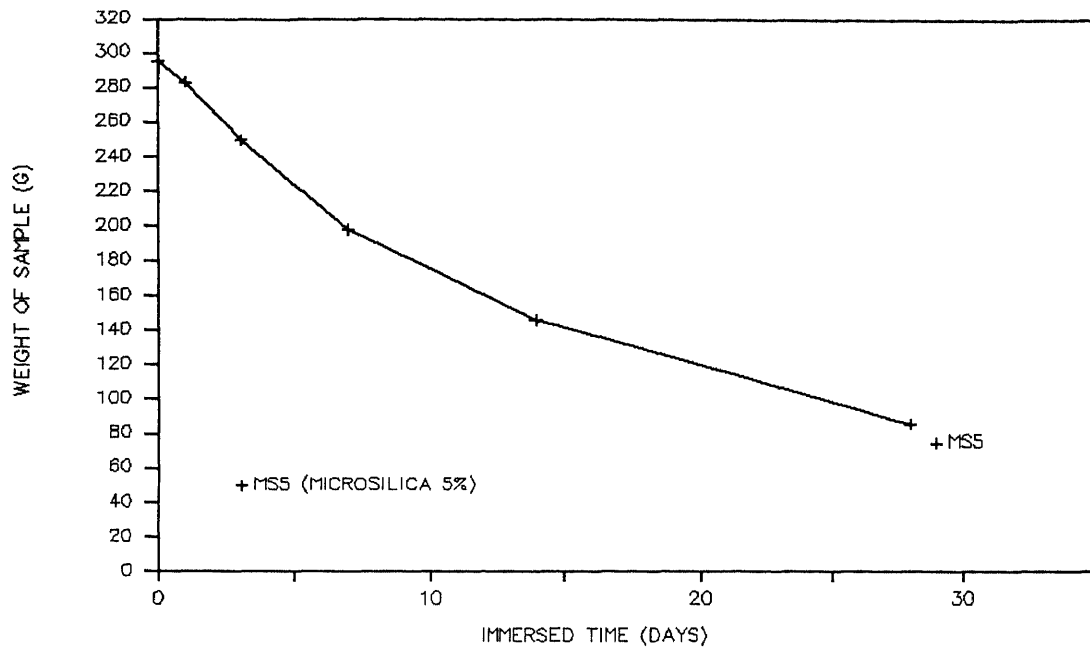
**Figure A 16** Compressive Strength of OR Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (50% Replacement)



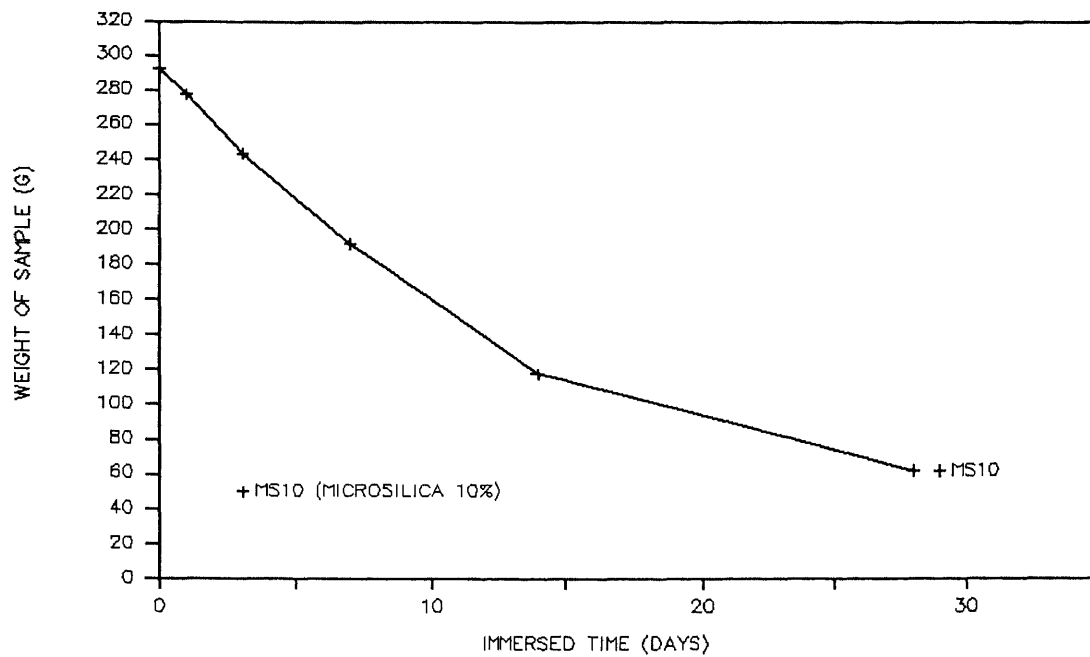
**Figure A 17** Compressive Strength of MM Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l (35% Replacement)



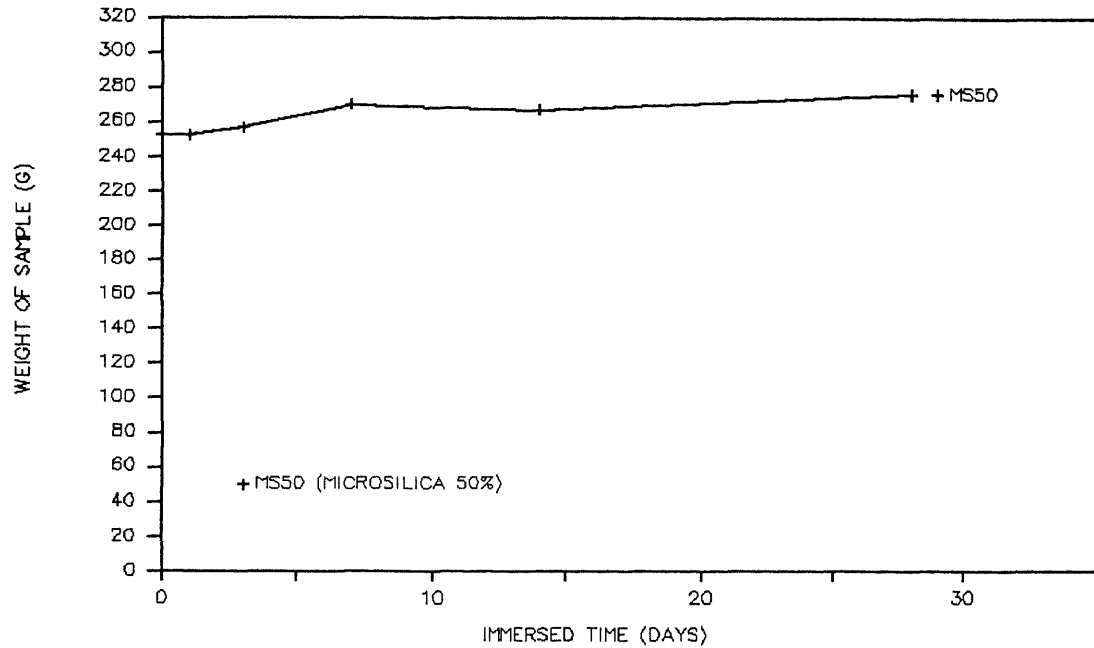
**Figure A 18** Compressive Strength of MM Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l (50% Replacement)



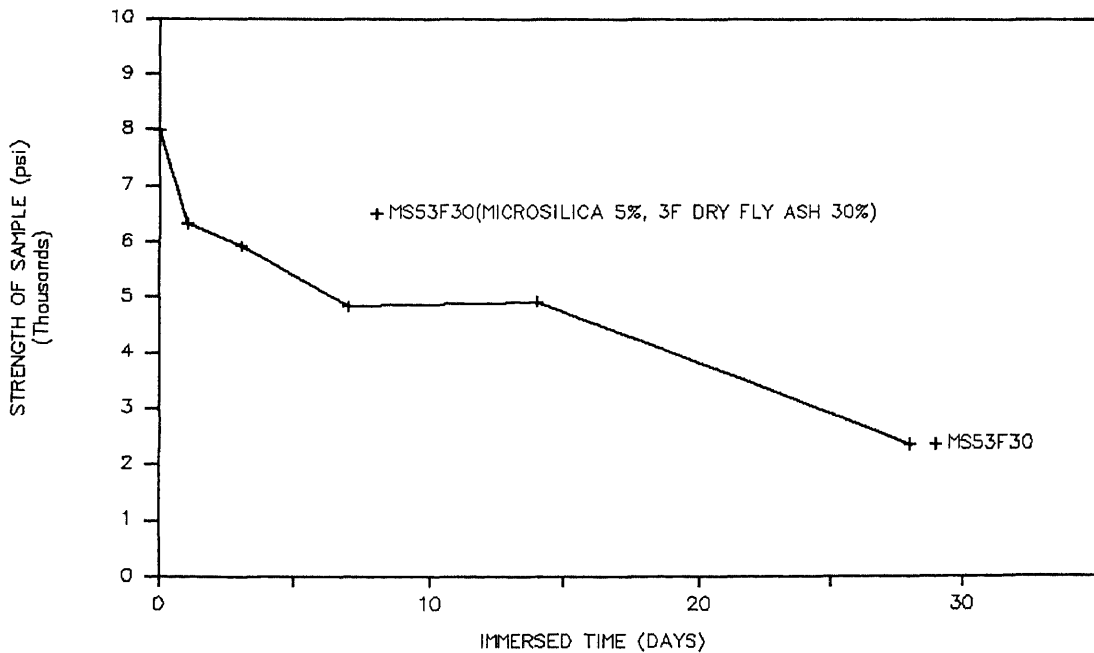
**Figure A 19** Weight of Microsilica Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (5% Replacement)



**Figure A 20** Weight of Microsilica Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (10% Replacement)

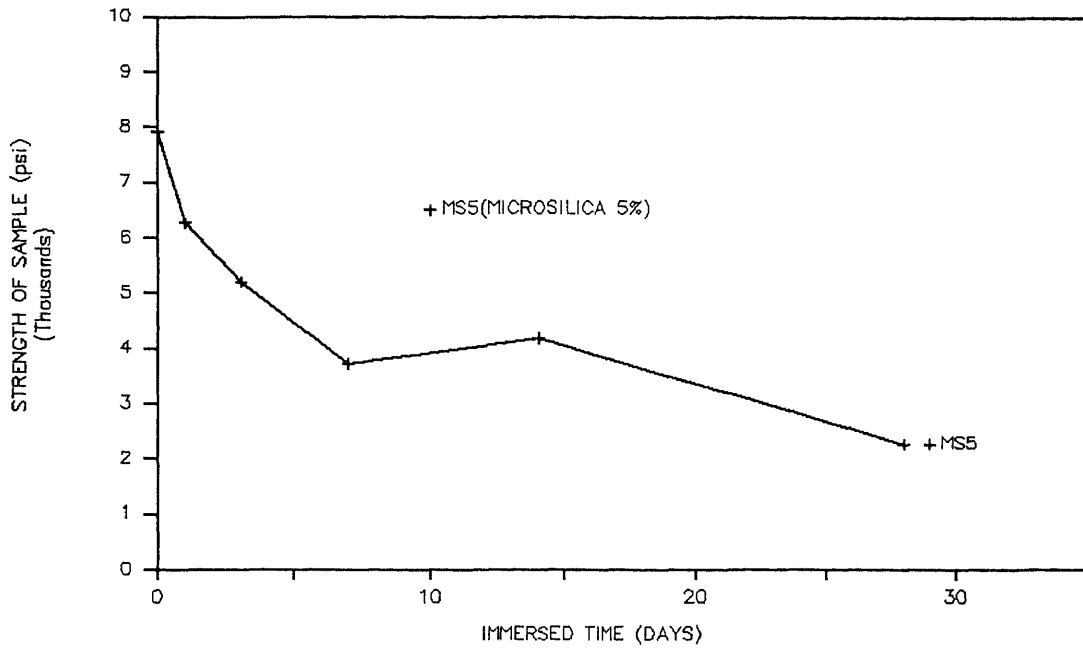


**Figure A 21** Weight of Microsilica Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (50% Replacement)

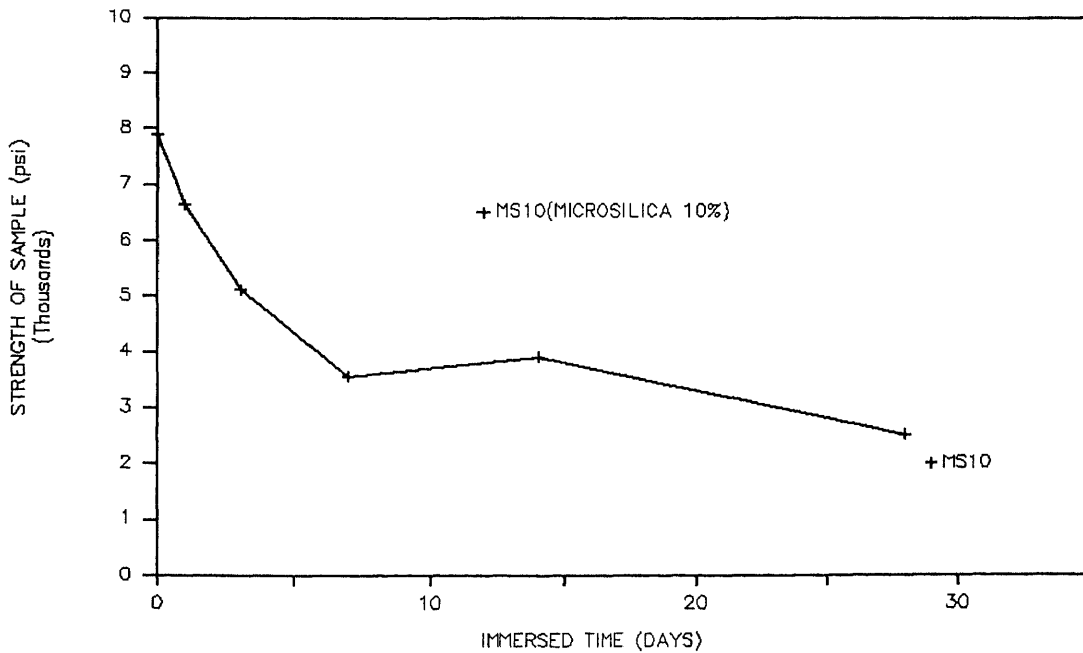


**Figure A 22** Weight of Microsilica and 3F Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (35% Replacement)

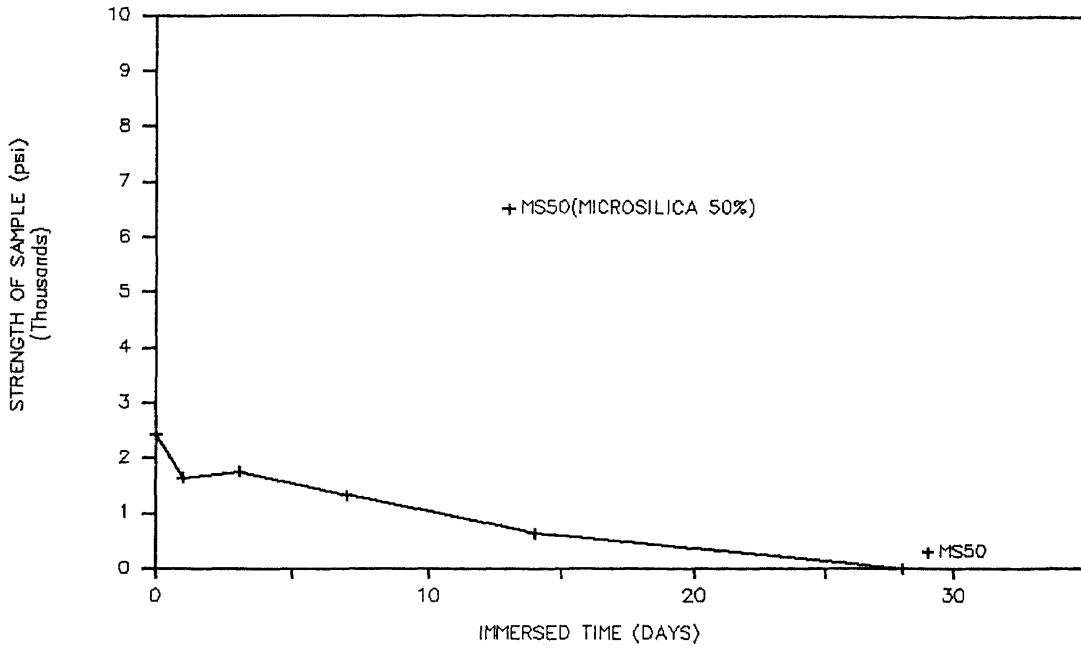




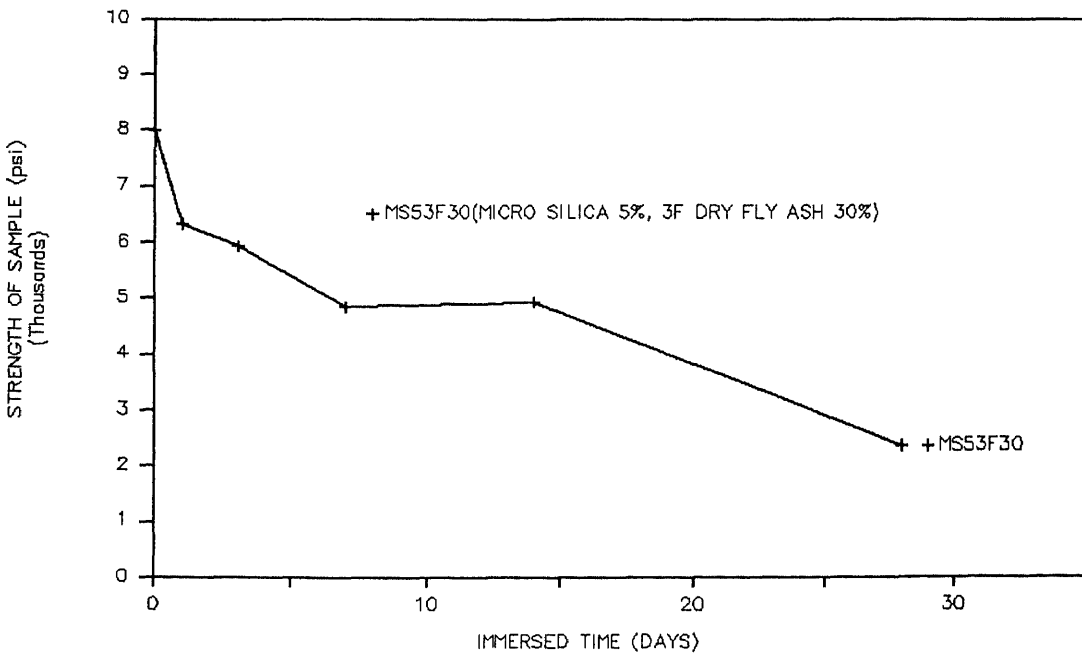
**Figure A 23** Compressive Strength of Microsilica Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (5% Replacement)



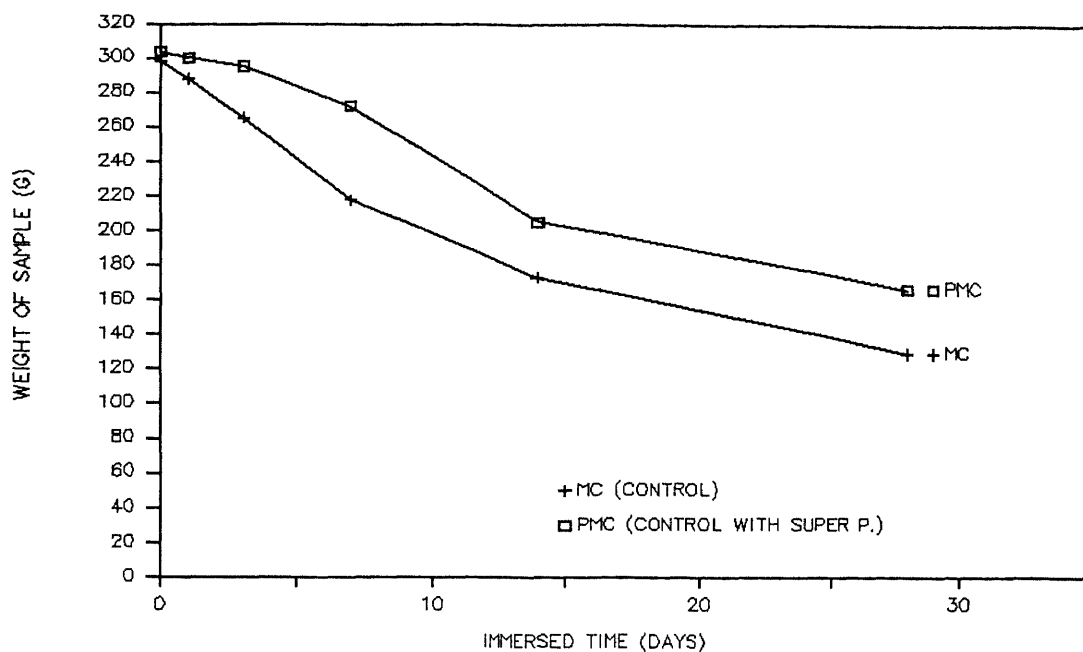
**Figure A 24** Compressive Strength of Microsilica Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (10% Replacement)



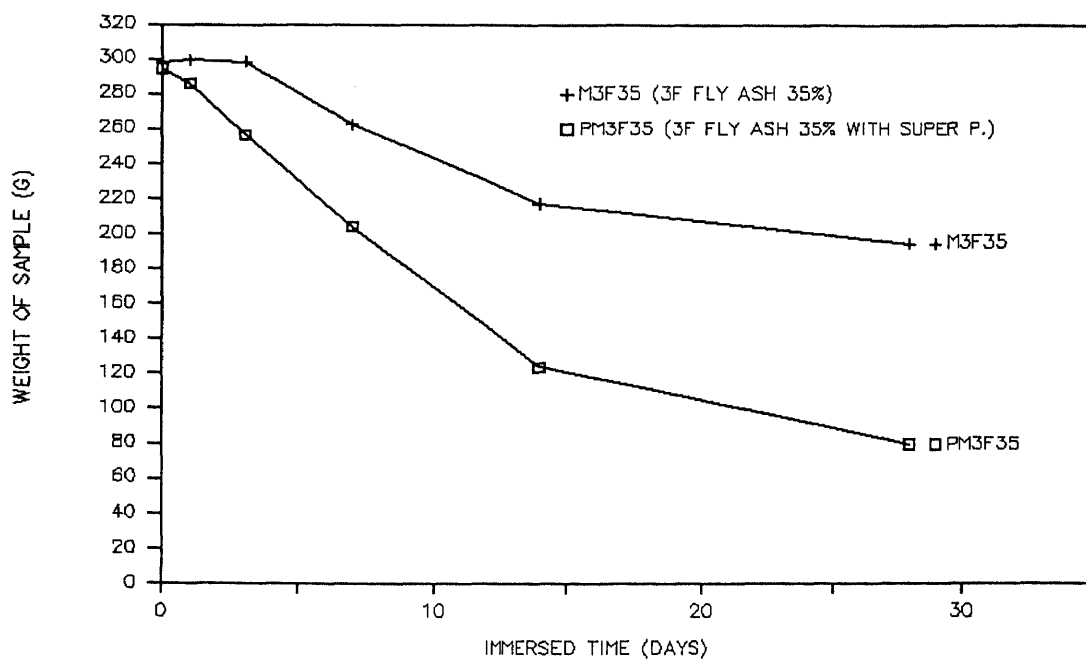
**Figure A 25** Compressive Strength of Microsilica Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (50% Replacement)



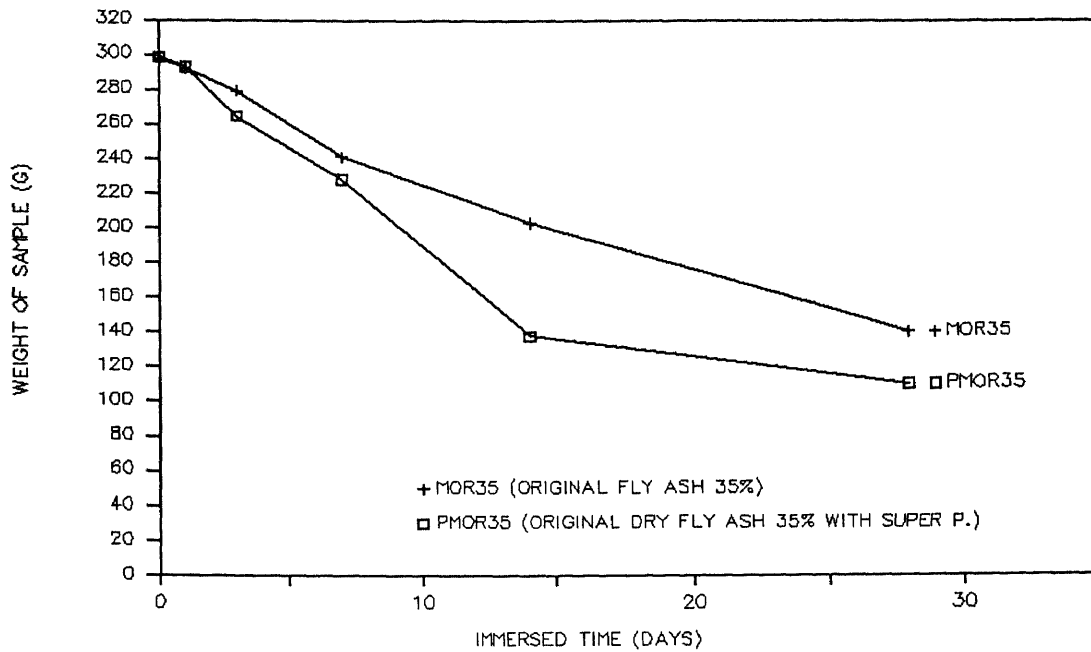
**Figure A 26** Compressive Strength of Microsilica and 3F Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l (35% Replacement)



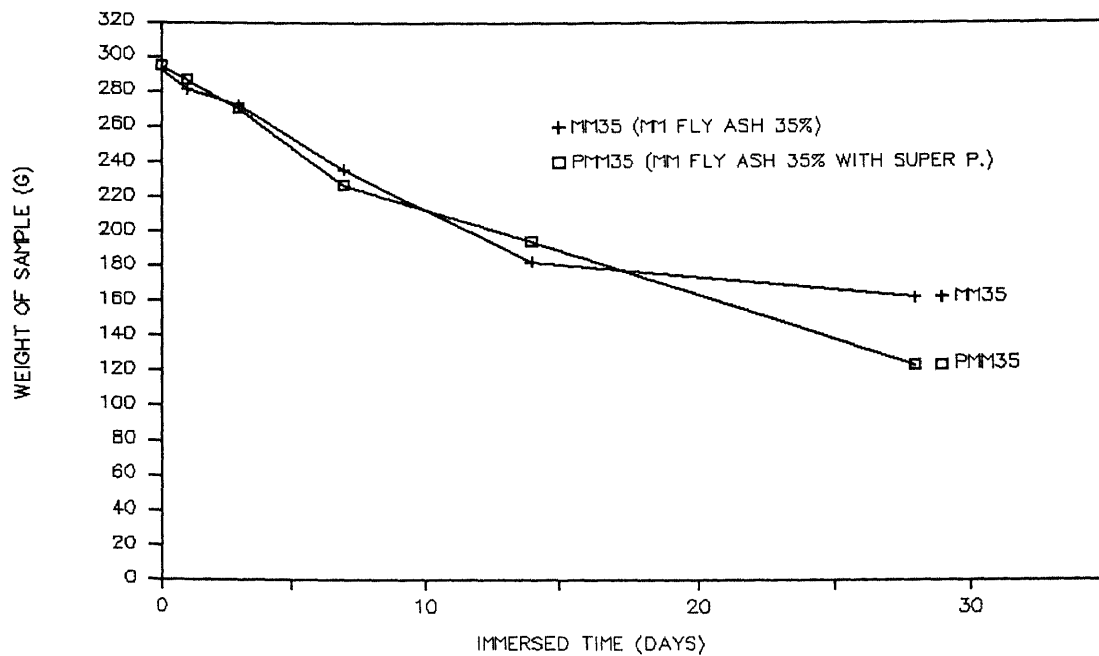
**Figure A 27** Weight of Normal Cement Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l with and without Superplasticizer



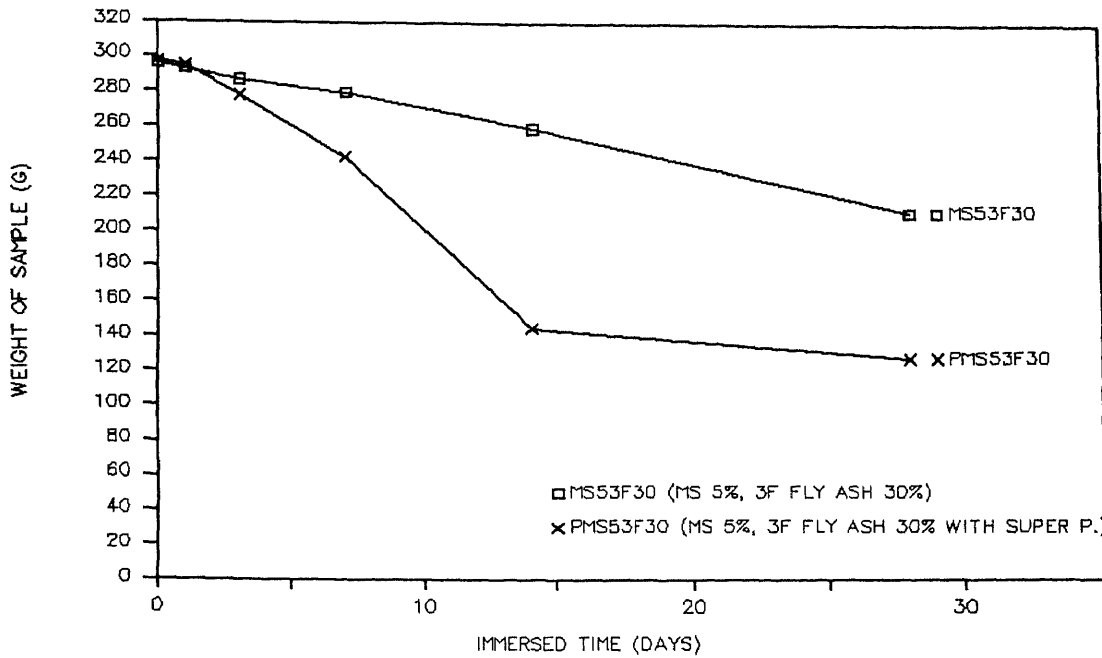
**Figure A 28** Weight of 3F Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l with and without Superplasticizer (35% Replacement)



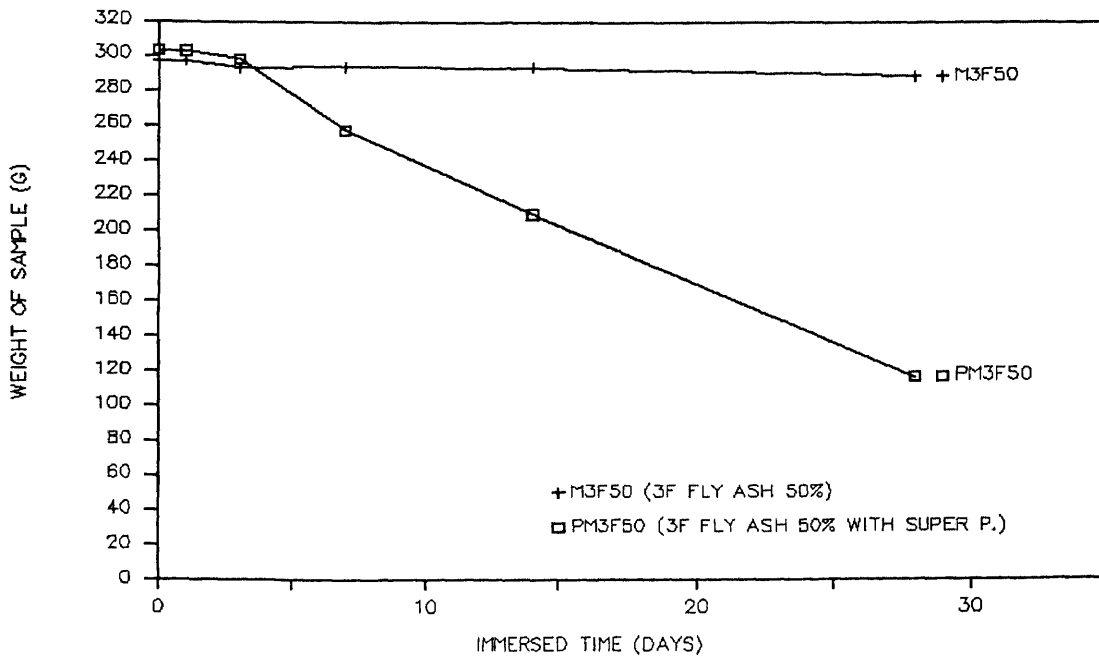
**Figure A 29** Weight of OR Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (35% Replacement)



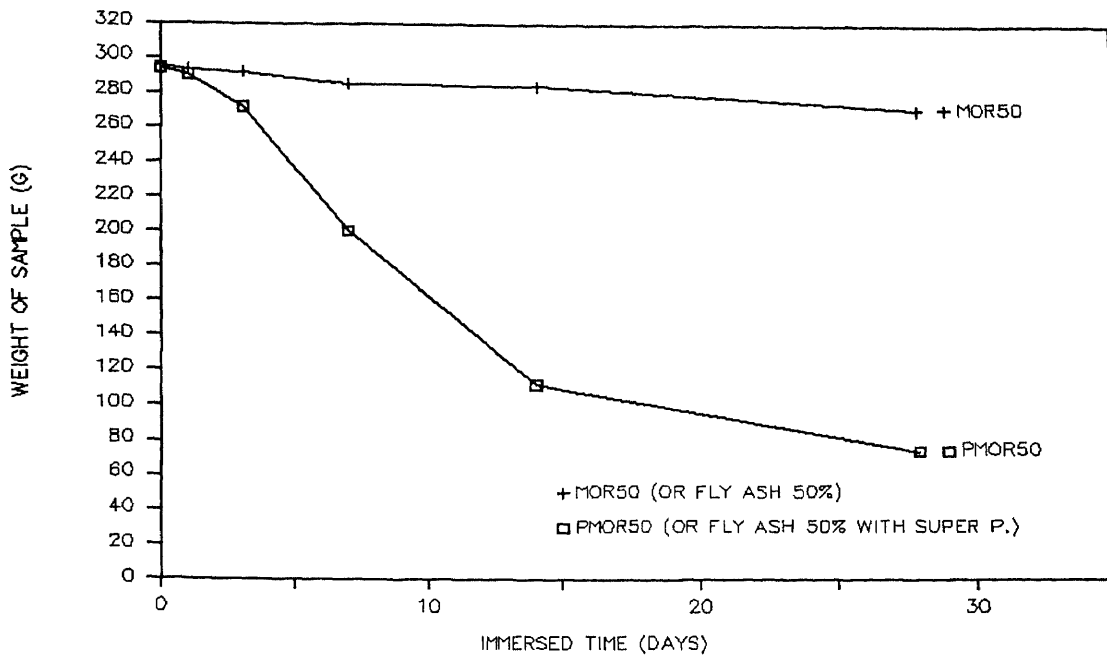
**Figure A 30** Weight of MM Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (35% Replacement)



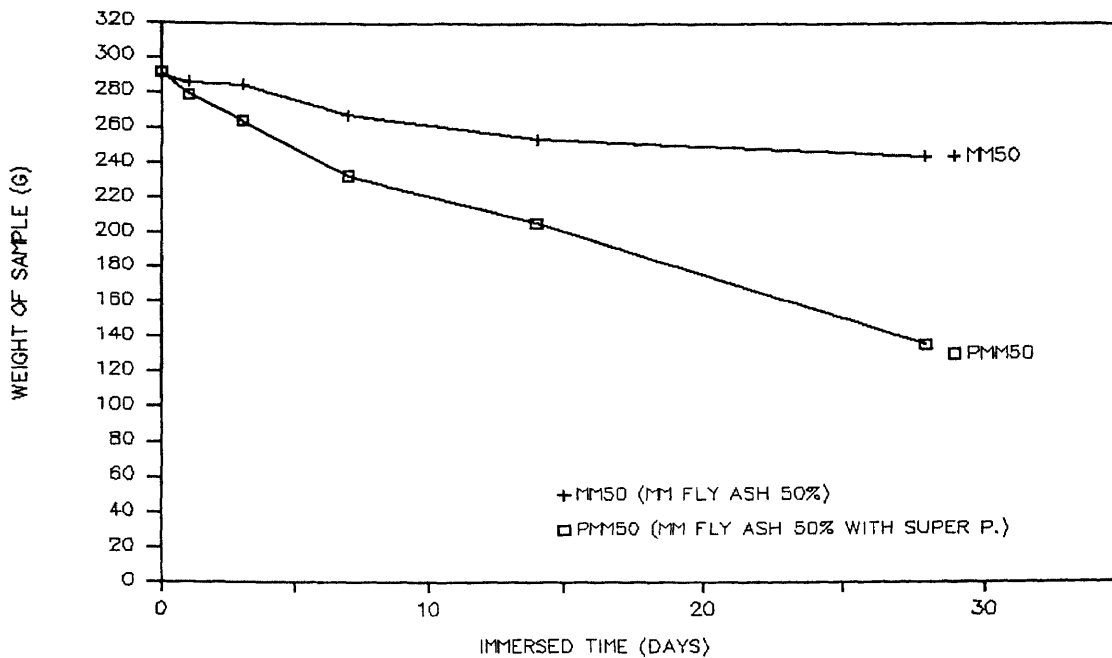
**Figure A 31** Weight of Microsilica and 3F Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (35% Replacement)



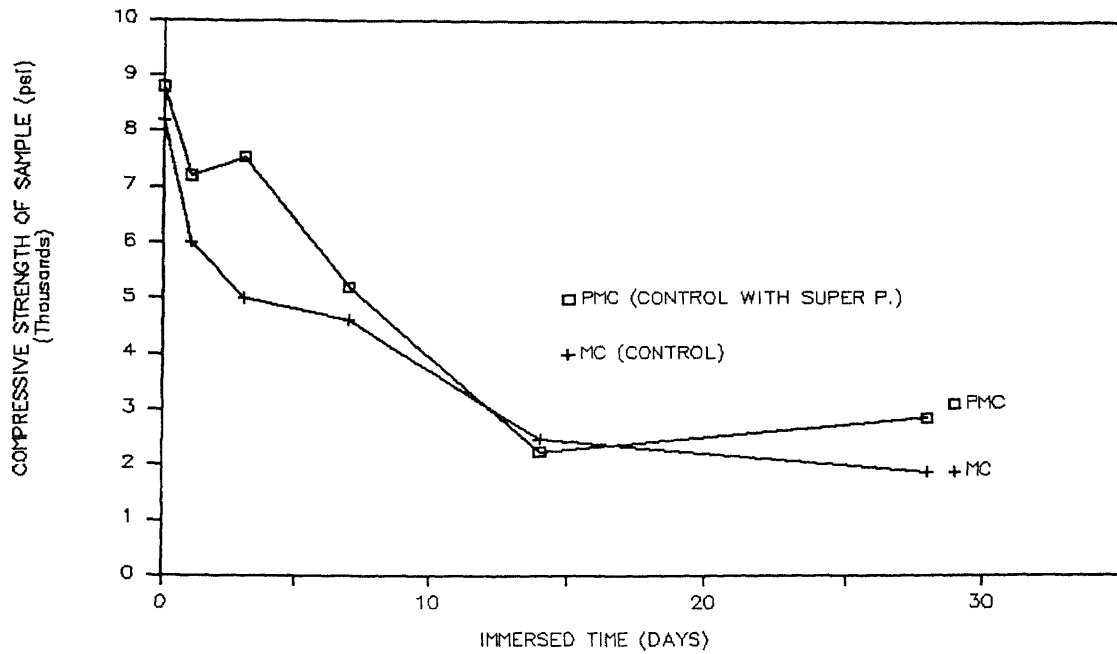
**Figure A 32** Weight of 3F Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (50% Replacement)



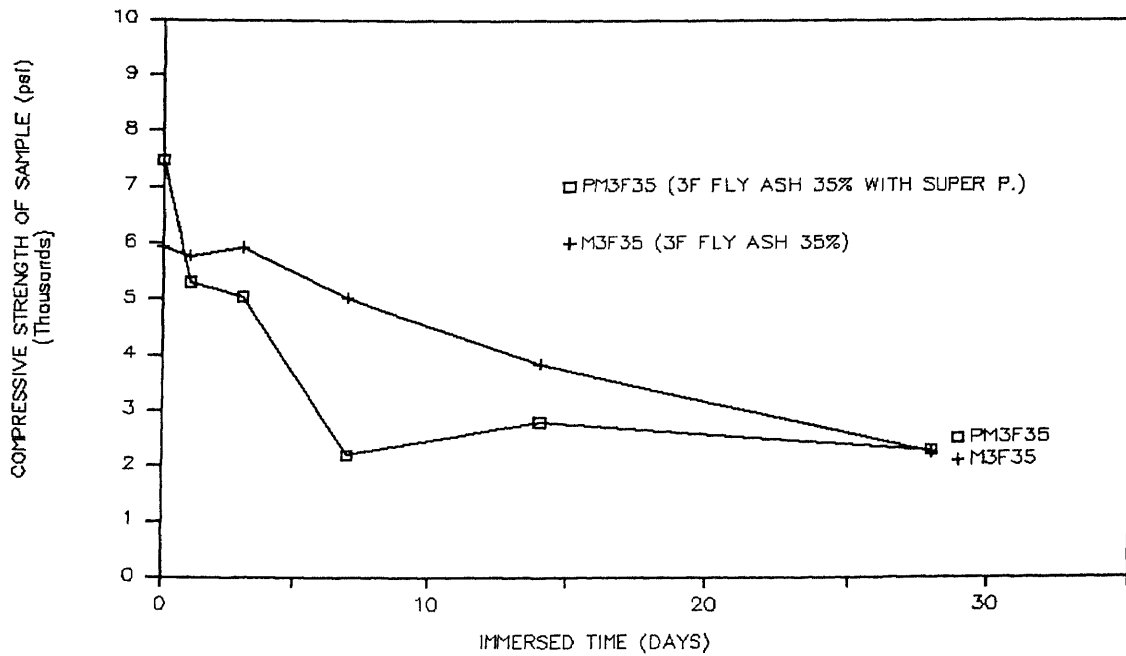
**Figure A 33** Weight of OR Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (50% Replacement)



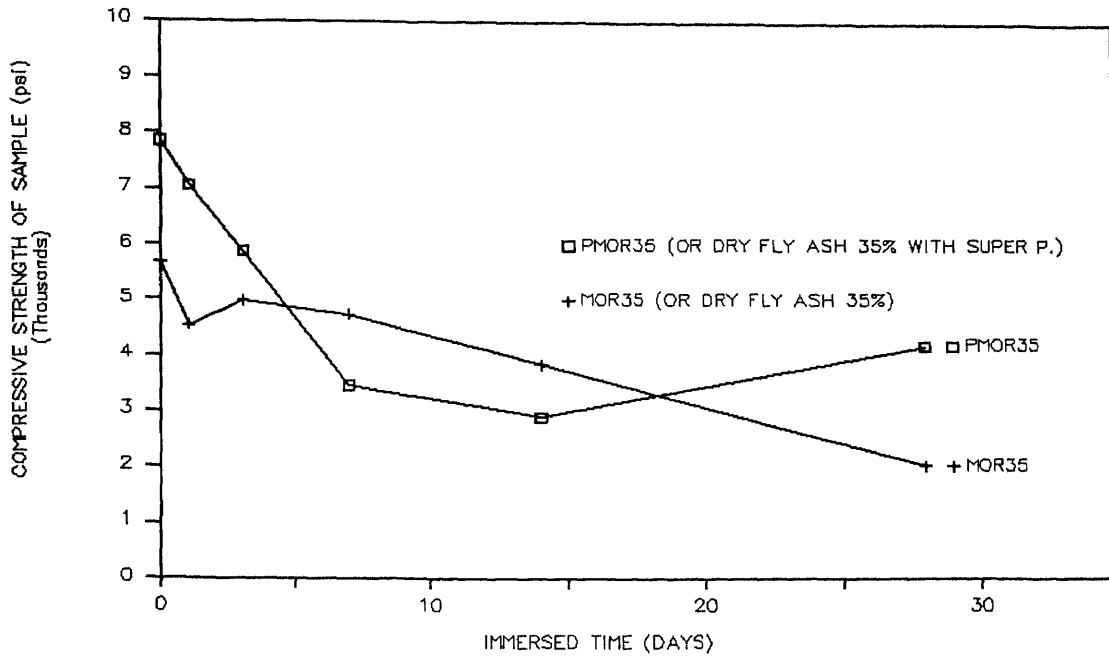
**Figure A 34** Weight of MM Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (50% Replacement)



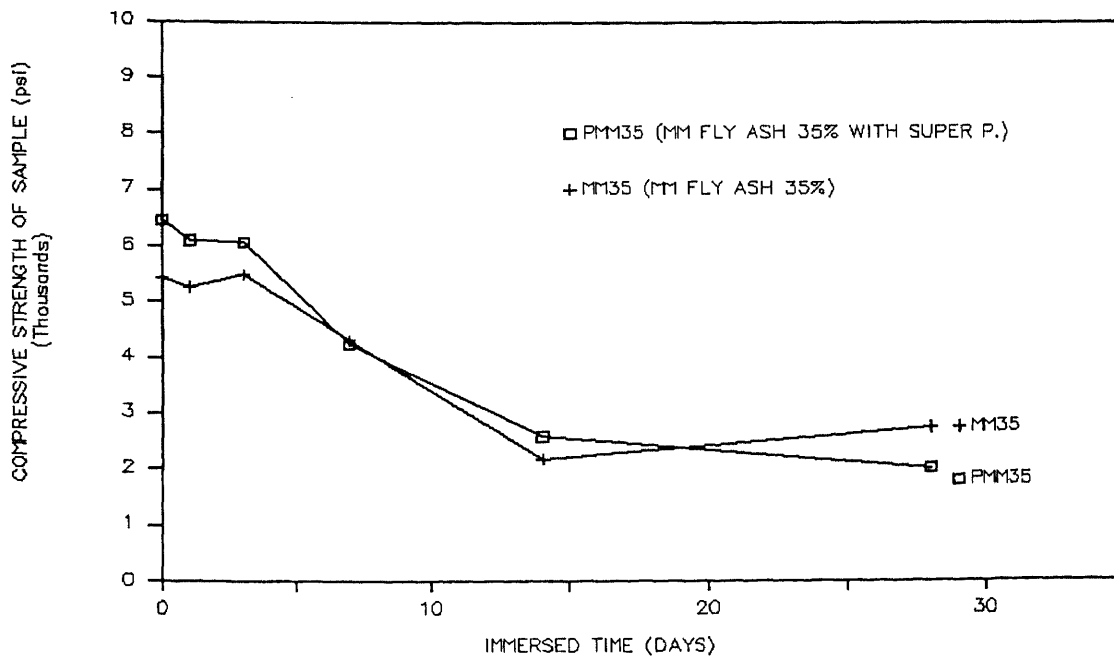
**Figure A 35** Compressive Strength of Normal Cement Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l with and without Superplasticizer



**Figure A 36** Compressive Strength of 3F Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l with and without Superplasticizer (35% Replacement)

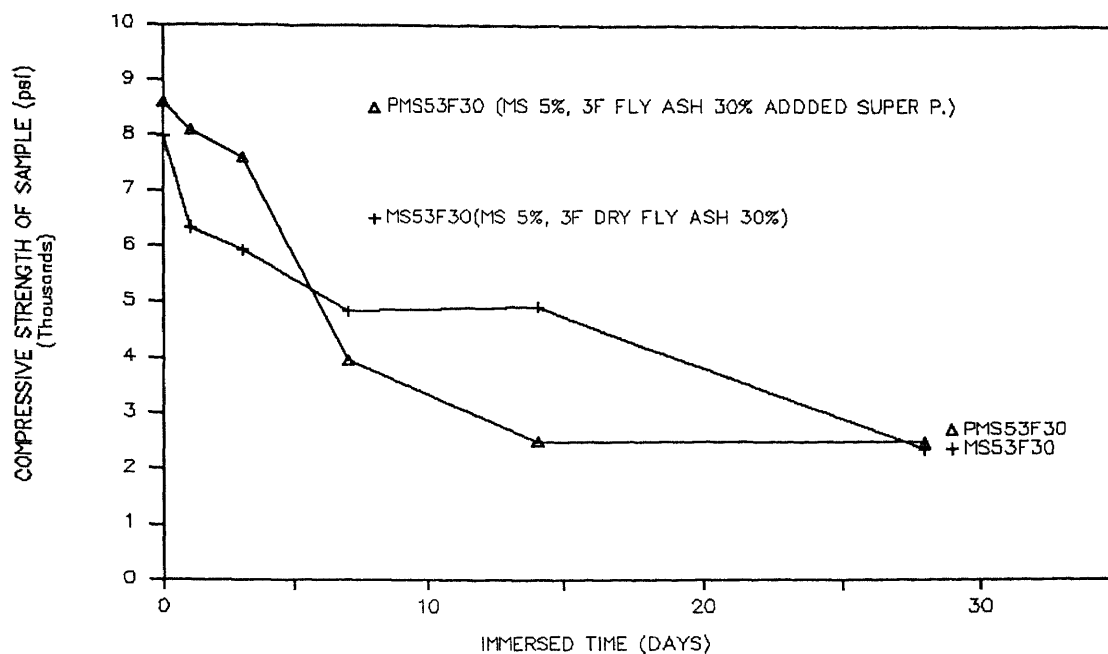


**Figure A 37** Compressive Strength of OR Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (35% Replacement)

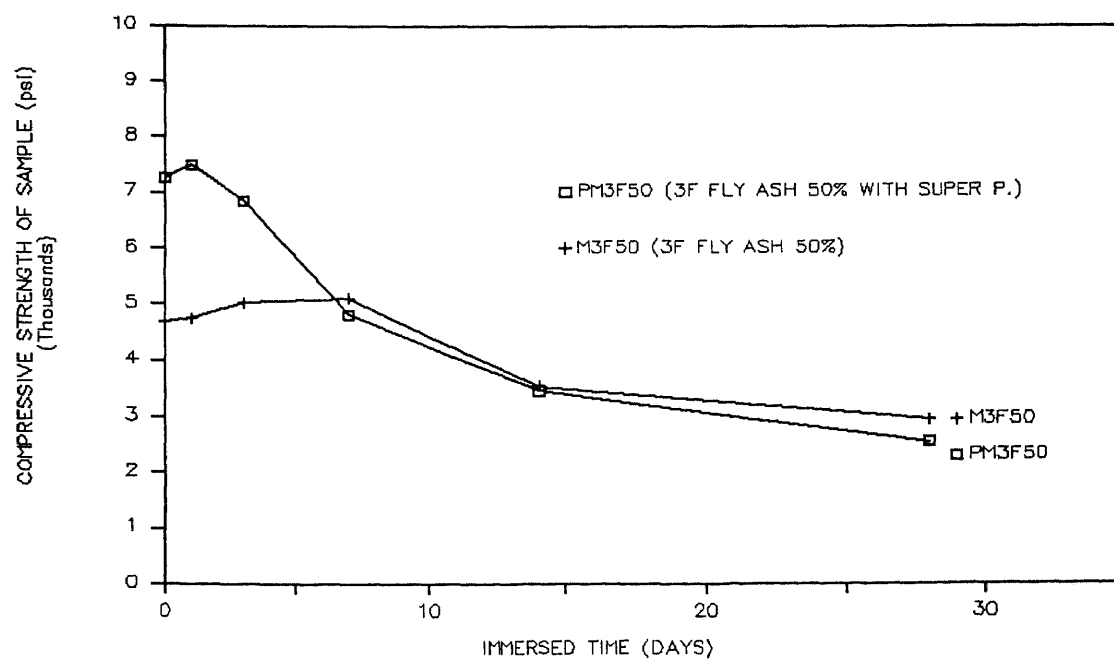


**Figure A 38** Compressive Strength of MM Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (35% Replacement)

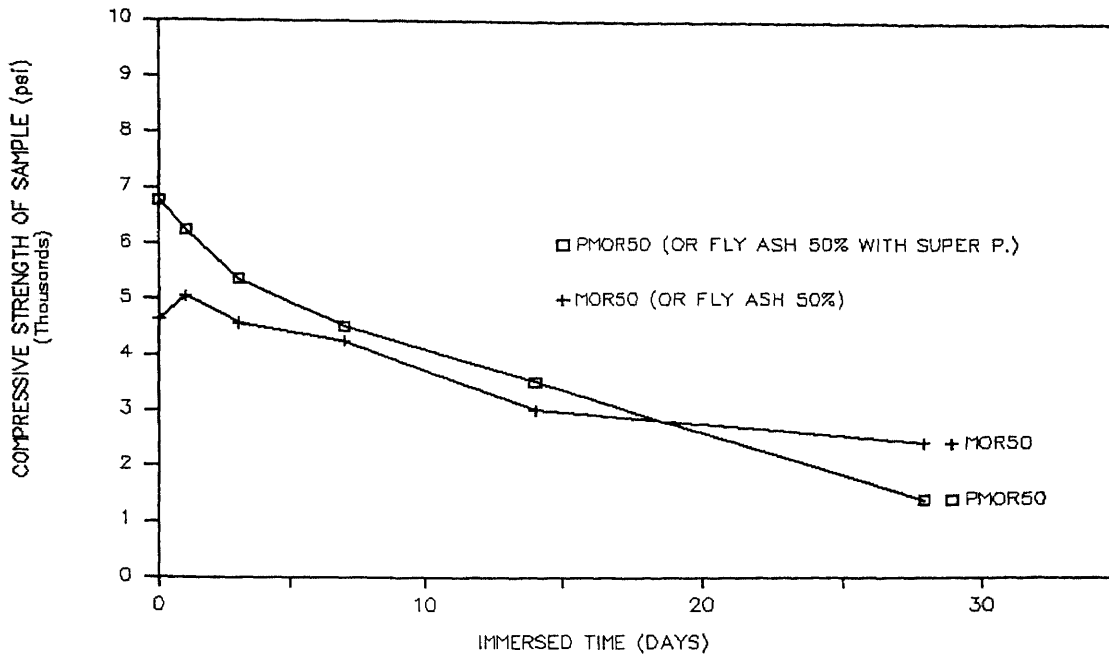




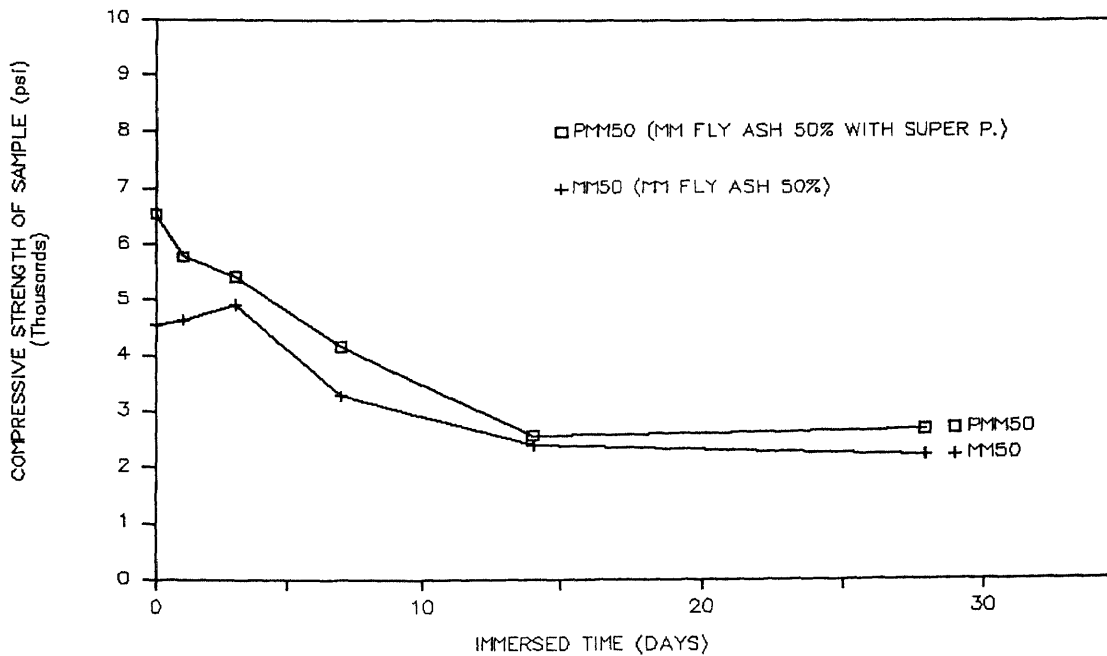
**Figure A 39** Compressive Strength of Microsilica and 3F Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l with and without Superplasticizer (35% Replacement)



**Figure A 40** Compressive Strength of 3F Fly Ash Mortar at Different Immersed Time in H<sub>2</sub>SO<sub>4</sub> 100 ml/l with and without Superplasticizer (50% Replacement)



**Figure A 41** Compressive Strength of OR Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (35% Replacement)



**Figure A 42** Compressive Strength of MM Fly Ash Mortar at Different Immersed Time in  $H_2SO_4$  100 ml/l with and without Superplasticizer (50% Replacement)

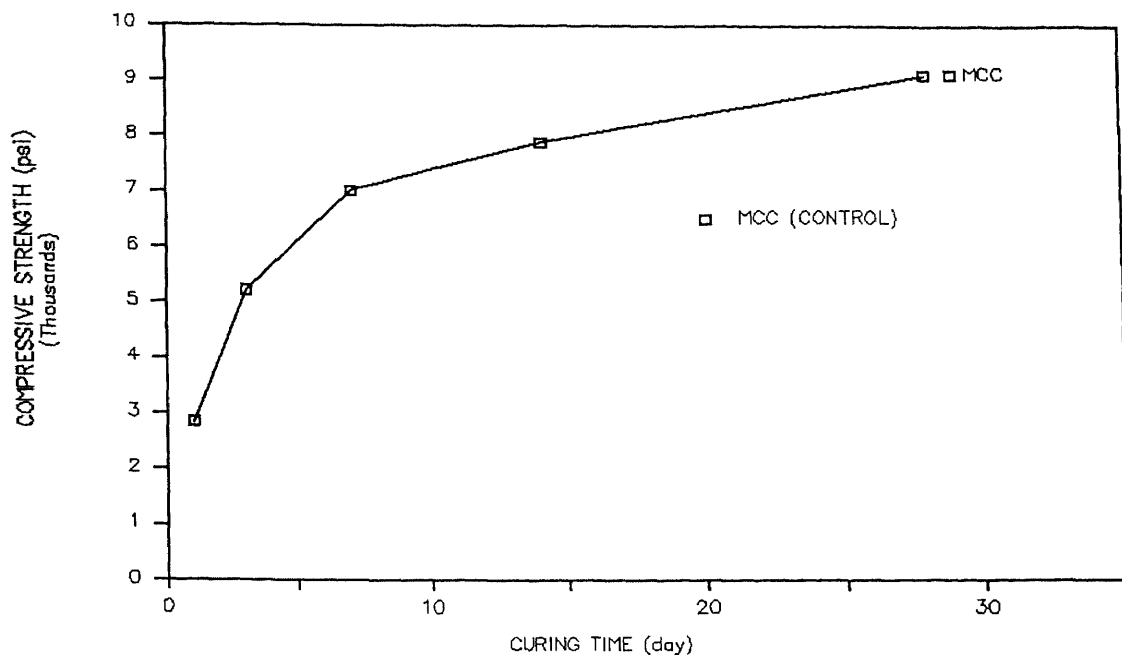


Figure A 43 Compressive Strength of Control Mortar and Age

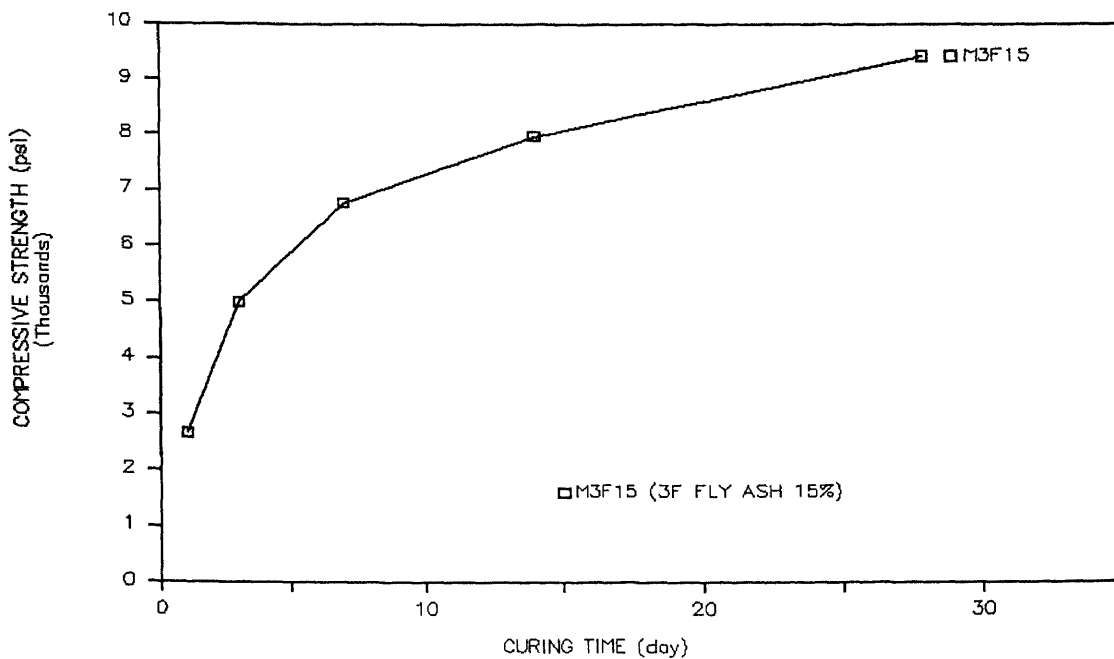
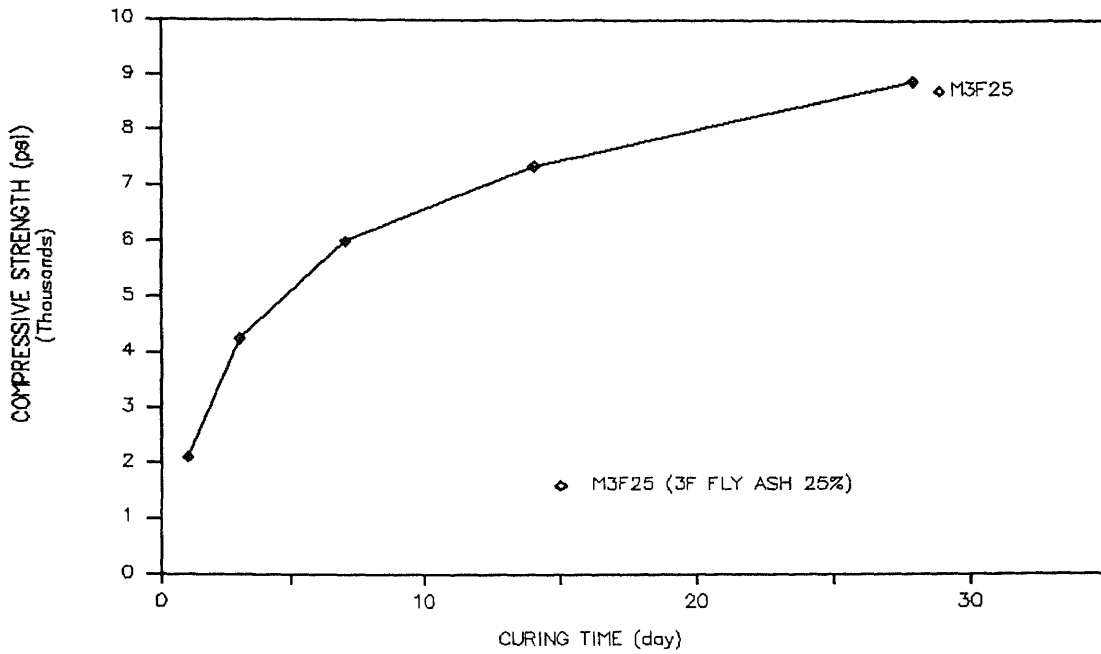
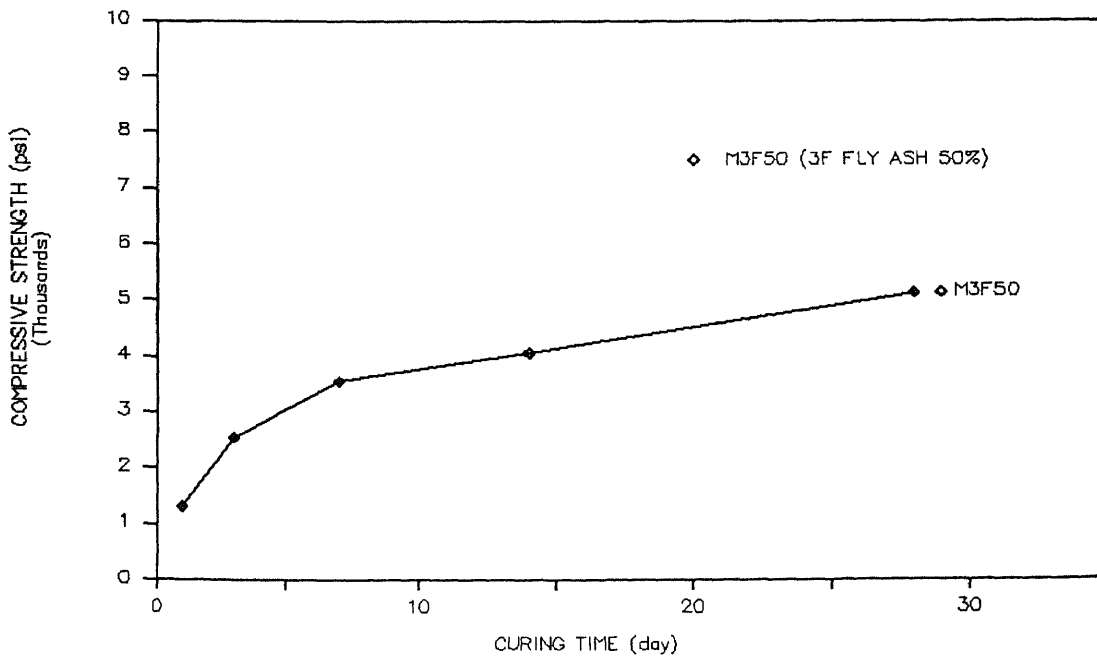


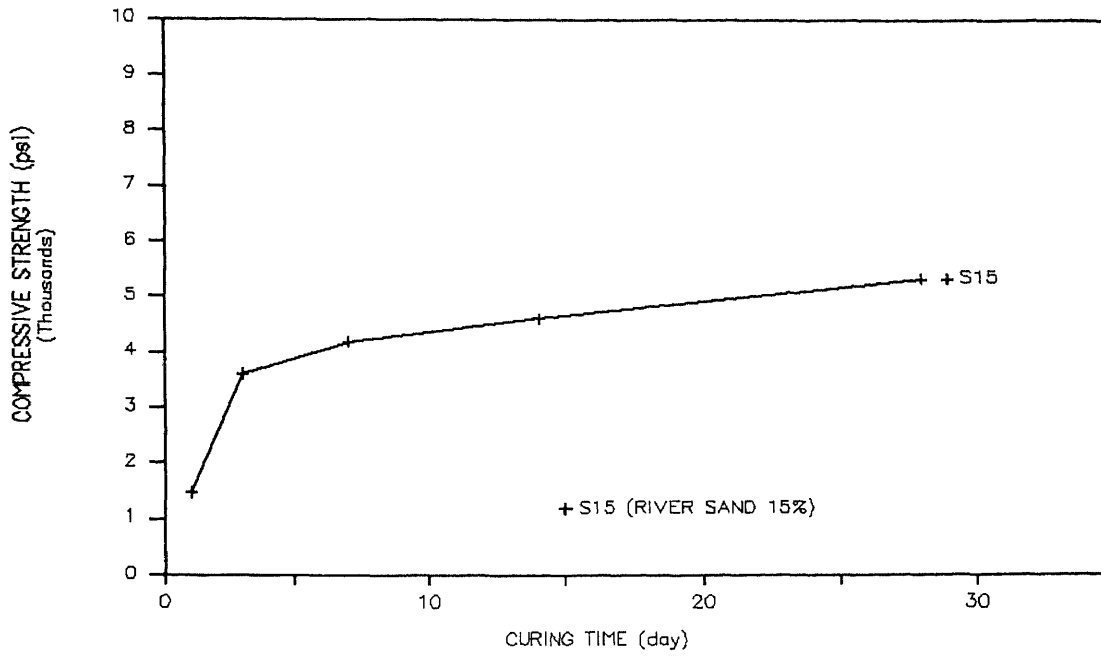
Figure A 44 Compressive Strength of 3F Fly Ash Mortar and Age (15% Replacement)



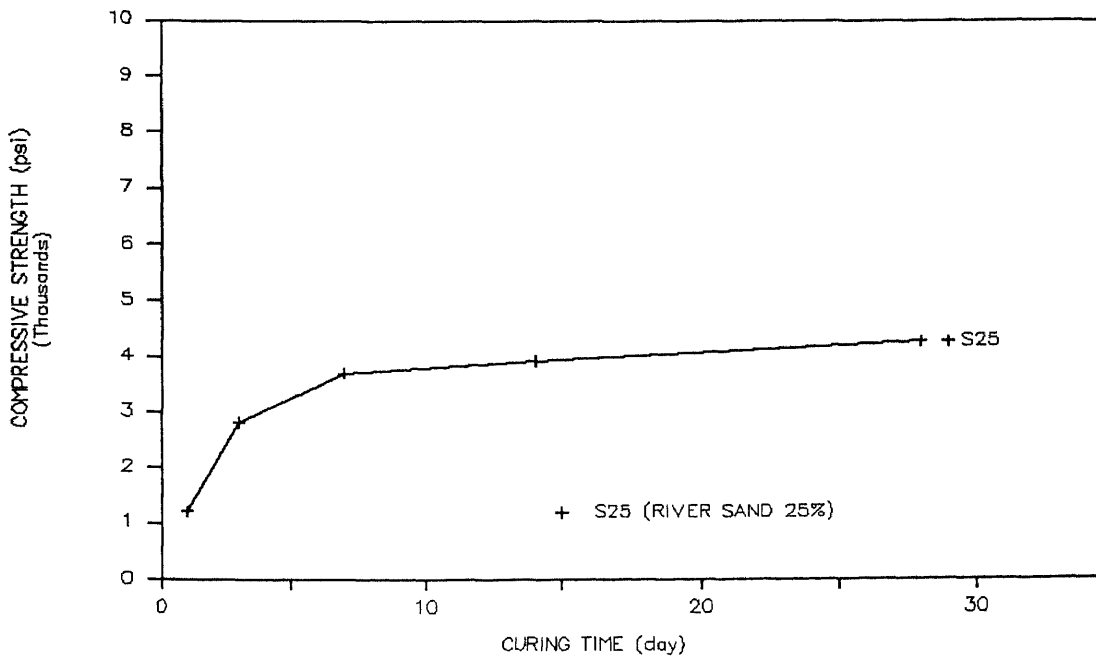
**Figure A 45** Compressive Strength of 3F Fly Ash Mortar and Age (25% Replacement)



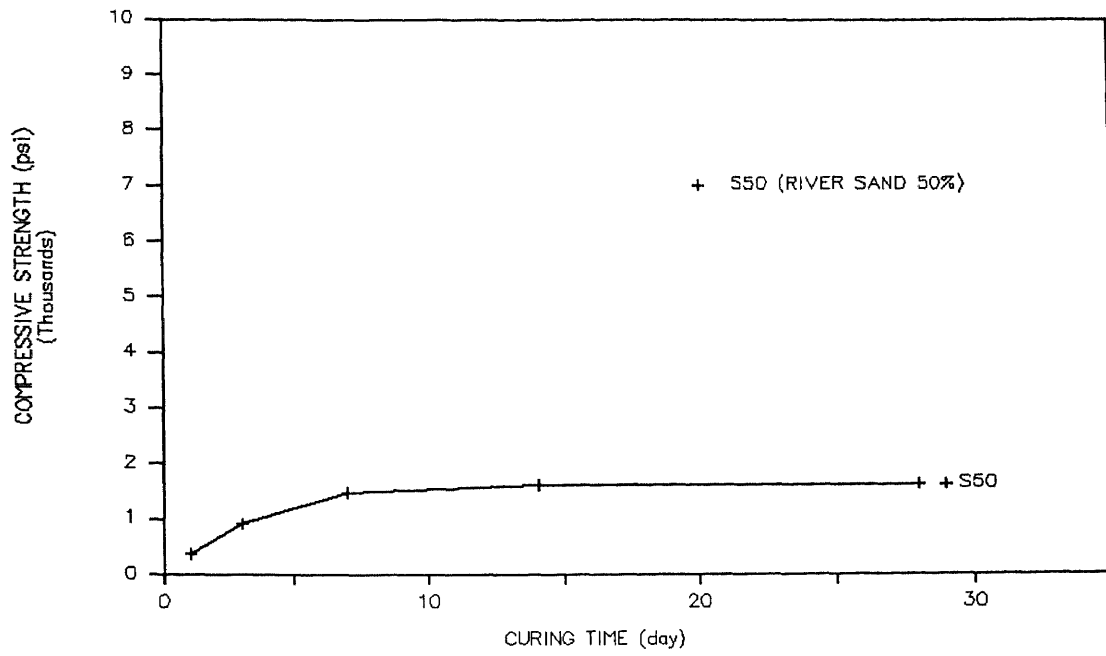
**Figure A 46** Compressive Strength of 3F Fly Ash Mortar and Age (50% Replacement)



**Figure A 47** Compressive Strength of Sand Mortar and Age (15% Replacement)



**Figure A 48** Compressive Strength of Sand Mortar and Age (25% Replacement)



**Figure A 49** Compressive Strength of Sand Mortar and Age (50% Replacement)

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