

BLENDING OF CLASS F FLY ASHES

Final Report

by

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Technical Report Documentation Page

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| 6. Abstract In this study, three fly ashes with LOI contents of 0.1%, 4.8% and 14% were blended to produce wide range of LOI fly ashes (0.1, 1.2, 3.3, 6, 7, 8.5, 9.4, 10.8 and 11.9%). The unblended and blended ashes were used in preparing concrete and mortar mixes to assess the impact of fly ash blending on durability and strength. Chemical, mineralogical, morphological and particle size distribution analyses were conducted on all as-received materials and the durability of cement-fly ash blends was assessed through mortar and concrete strength, and length expansion on mortar and concrete bars at selected periods of exposure to a 5 % sodium sulfate solution and saturated lime. It was found that blending of ashes, incorporating a rejected ash source, generated blends that showed potential long term durability problems. Performance of these blends showed significantly lower durability and strength in sulfate environments if compared to unblended fly ash mixes that pass the current FDOT specifications. Particle size distribution analysis up to 1000 microns together with morphological analysis were found to be excellent potential indicators in assessing the properties of the as-received ashes and their blends. It is recommended that prior to allowing the incorporation of blended Class F fly ash in concrete construction practices, a study has to be initiated whereby several blended fly ashes with LOI below 6% have to be carefully investigated for their materials properties and long term durability. Examination and application of the verified indicators to the current FDOT specifications and guidelines can then be established. This will ultimately lead to amending the current FDOT specifications if blending of ashes is to be approved. | | | | | |
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METRIC CONVERSION FACTORS

| UNITS | CONVERT | TO | MULTIPLY BY |
|--------------|---------------------|------------------|-------------|
| LENGTH | inch | mm | 25.4 |
| | foot | mm | 304.8 |
| | yard | meter | 0.9144 |
| | meter | foot | 3.281 |
| | meter | inch | 39.37 |
| FORCE | pound (lb) | newton (N) | 4.448 |
| | kip(1000 lb) | kilo newton (KN) | 4.448 |
| | newton (N) | pound | 0.225 |
| | kilo newton (KN) | kip K | 0.225 |
| FORCE/LENGTH | kip/ft | KN/m | 14.59 |
| | KN/m | lb/ft | 68.52 |
| | KN/m | kip/ft | 0.0685 |
| STRESS | pounds/sq.in. (psi) | N/sq.mm (Mpa) | 0.0069 |
| | kip/sq.in. (Ksi) | N/sq.mm. (Mpa) | 6.895 |
| | newton/sq. mm. | ksi | 0.145 |
| MOMENTS | ft-kip | KN-m | 1.356 |
| | in-kip | KN-m | 0.113 |
| | KN-m | ft-Kip | 0.7375 |

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EXECUTIVE SUMMARY

Scope

The objective of this investigation was to study the properties that will be affected through the blending of different quality Class F fly ashes. The impact of fly ash blending on concrete durability was also addressed. Three Class F fly ashes were used in this investigation with ASSHTO Type II cement. From the three as-received ashes, several blends were generated at different weight percentages to obtain variable loss on ignition values for fly ash content. The as-received materials were subjected to battery of tests including chemical analyses, particle size distribution, particle morphology, and mineralogical analysis using x-ray diffraction. Concrete and mortar specimens were prepared in two separate regimes. In the first, which simulates ready mix concrete batching, the aggregates were not proportioned, and the air entraining admixture dosage was varied to maintain a constant air content. The slump was maintained at 4 inches. In the second regime, the air entraining admixture dosage was maintained constant and batches were prepared for a constant slump of 4 inches. The concrete mix in both cases was a Class II Florida Department of Transportation [FDOT] mix. In the first regime 10 mixes including the control were prepared while for the second regime 14 mixes were prepared. The effect of blending fly ashes on concrete durability was examined through length change measurements on concrete and mortar prisms and rapid chloride permeability tests.

The findings of this investigation indicate that blending fly ashes passing FDOT specifications with rejected ashes, can possibly yield a blend of lower strength than that of the passing fly ash. In addition, through blending of different ashes from the same sources or different sources, blends of passing fly ashes can be produced that have potential durability problems. Strength monitoring over a period of 735 days did show strength gain profiles that are dependent on particle fineness, LOI and fly ash sources.

Monitoring expansion for a period of one year in the case of mortar or two years in the case of concrete, shows that concrete durability, is in general a function of fly ash LOI. Increasing the LOI increased the rate at which deterioration occurred. In addition, it

was found that the level of deterioration was dependent on the blended fly ash source and not only its LOI. Blended fly ash with LOI content [3.3%] well within the current FDOT specifications had poor durability performance if compared to unblended fly ashes with LOI content of less than 6% [0.1% and 4.8 %]. The results also indicate that durability performance of the blended ash is dependent on the quality of the source ash. The findings from this investigation shows that the current standards of accepting Class F fly ash might not be sufficient in addressing acceptance of blended ashes.

Conclusions and Recommendations:

The findings and conclusions of this investigation on blending of fly ashes can be summarized as follows:

1. Blending of two fly ashes of different sources with one passing the current FDOT specifications, generated blends that had a strength behavior controlled by the quality of the original ashes.
2. Blending of two ashes from the same source with one passing the current FDOT specifications, generated a blend that had a strength behavior controlled by the quality of the original ashes.
3. Durability of the blended ashes as assessed by mortar expansion, indicated: whether the ashes are from the same source or different sources, and provided that one of the ashes is not passing the current FDOT specifications, the resultant blend will be of unequal performance to that attained by current passing FDOT ashes.
4. Material parameters that were found to be affected by blending of ashes include: particle size distribution, morphology and LOI.
5. Though it is critical to study the original sources of the ashes to be able to determine if there is a blend, particle size distribution of the ashes up to 1000 microns was found

to be a promising tool that needs to be further studied for its use as a possible indicator criteria for accepting/rejecting blended Class F ashes.

Based on the findings of this investigation, it is recommended that:

1. As current FDOT specifications do not necessarily regulate blending of Class F fly ashes, it is recommended that a study be initiated whereby the critical indicators established by this study can be verified on a larger number of blended fly ashes with LOI ranges up to 6.0%.
2. Based on the first recommendation, amending the current FDOT specifications would be required.

INTRODUCTION

OBJECTIVE:

The objective of this investigation is to study the material parameters that would be affected by blending different quality Class F ashes and would in turn affect the durability of fly ash/cement blends. In addition to identifying those material parameters and their impact on blended ash/cement durability, a third objective of this study was to identify a possible critical indicator that can be used to approve/reject blended Class F ashes. In the following paragraphs, the scope of the project will be addressed.

SCOPE:

Due to current shortage in the supply of Class F fly ash, this research project was initiated to study the effect of blending different quality Class F fly ashes on quality of fly ash and concrete properties and durability. Currently, the Florida Department of Transportation Specifications do not allow the use of Class F fly ash with an LOI content of more than 6.0%. The current standards do not address blending of Class F ashes. This study also considered identifying indicators that can be used in accepting/rejecting blended ashes. The scope of this work was divided into stages. The first stage was focused on studying and characterizing the as-received materials. The properties studied included:

1. Inorganic oxide content
2. Particle size distribution
3. Mineralogical content

In the second stage, the strength and durability performance of concrete and mortar mixes was assessed through the following measurements: concrete compressive strength at different curing environments, concrete and mortar length expansion and finally rapid chloride permeability tests.

Finally, data generated from the tests conducted on the as-received material and concrete and mortar mixes were analyzed and interpreted to identify a possible indicator that can be considered for purposes of accepting/rejecting blended ashes.

REPORT:

This report documents the results of the tests, provide interpretation of results and identifies a possible indicator for rejecting/accepting blended ashes. Conclusions and recommendations as to what is needed to be done in order to ensure better quality at a timely fashion were also given.

LITERATURE REVIEW

Coal fired electric generating power plants are facing increased challenges to adhere to more stringent environmental regulations. This necessitates operational changes for conformity with air emission requirements. As a result, the volume of low LOI fly ashes available for use in concrete has dropped significantly. Due to shortage of low LOI fly ashes in the State of Florida, this study was initiated to address the possibility of blending low LOI (<6.0%) with high LOI fly ash (>6.0) and its impact on concrete durability.

Fly ashes collected from coal burning power utilities is in itself a variable material. Due to differences in the inorganic impurities in the source coal, in the coal preparation (pulverizing), in the combustion conditions (i.e. temperature and if any other substances, such as oil, are utilized in the combustion process), and in the collection and handling methods at each site, the fly ash will vary. Because no two utilities have all of these factors in common, each plant's fly ash is unique, and may in itself vary with time [1]. In addition to particle fineness, morphology, LOI/unburnt carbon, mineralogy and chemistry are some of the ash properties that will vary with operation time, even within the same power plant. Each of these materials aspects will have impact on the quality of the produced fly ash.

It is a well established fact that there is a general relationship between loss on ignition and concrete air-entrainment potential. High carbon fly ash decreases the efficiency of air-entraining admixture to achieve a desired air content [2,3]. Air content is critical when considering concrete durability. Variability in ash quality that will introduce variability in concrete air-entrainment capability is thus a problem. Hill et al [4], suggested that it is the morphology of the carbon particles rather than the loss on ignition that is of significance to the potential of a fly ash to entrain air in concrete. Differences can exist in the nature of the carbon particles that might not necessarily be reflected by significant variation in measured LOI. In other words, fly ashes having the same LOI might have different air-entrainment characteristics in concrete. In this work two fly ashes from the same source and two other ashes from an additional source were examined for their air-entrainment capabilities in conjunction with LOI. The findings concluded

that the morphology of the carbon particles as opposed to the LOI were of significance in characterizing air-entrainment profiles for an ash.

Morphology of the fly ash particles was also considered of significance in determining water requirement demands for a given fly ash. In the work of Kokubu [5] and Yamazaki [6], the morphology of fly ash particles was found to be important together with fly ash fineness in dictating water demand. Spherically shaped particles reduced water demand while irregular shaped particles increased it.

Others [7,8] believe that the dominant factor in determining the water demands for a particular ash is fineness, as determined by percent retained on 45 microns sieve, or LOI. As the LOI increased the slump was found to decrease indicating a need to increase mix water. If the same LOI value can be considered as indicative of same carbon particle morphology then those results are in agreement with Hill et al [4].

As to the contribution of fly ash to concrete strength, a study [9] indicates that there is no relationship between LOI and strength development in concrete. As a matter of fact, the highest strength was that for the highest LOI fly ash. However, it has to be mentioned that in this study higher LOI ash had the higher fineness. In addition, the mixes containing high LOI fly ashes had high range water reducer incorporated in the mix to maintain constant slump and cut on the water demand. In addition, mineralogical and morphological analysis was not conducted on the as-received materials. It is well established fact that the pozzolanic reactivity of Class F fly ashes are affected by the amorphous content in the ash. Hornain[2] indicated that both fineness and LOI are the proper indicators for the contribution of fly ash to concrete strength.

The literature is rich with data establishing the effect of Class F fly ash on improving concrete durability. However, most of the work done in this area is based on single source fly ash, or variable source fly ash with no relevance to particle morphology, mineralogy or fineness [9,10,11,12,13].

As can be seen from the previous discussion, there are several materials aspects that need to be considered in addressing the performance of fly ash in concrete. In addition, depending on the scope of the study, the findings might be contradictory or reinforcing. This current study was initiated to identify the materials parameters of a fly ash that can be affected by fly ash blending. In addition to identifying the material

properties, the significance of those properties to concrete durability was to be addressed. Through satisfying the above, suggestions can be made regarding critical indicators that can be used to accept/reject blended ashes.

There is ample data in the literature on LOI effect on the concrete and mortar strength. However, in these studies, it was found that, in general, several parameters were varied at the same time with most of them having direct impact on strength. No study was found that directly addresses blending of ashes while maintaining, correcting or taking into consideration the impact of material properties on fly ash concrete properties. Based on the above review this investigation was initiated to address the possible material parameters that will be affected by blending and will in turn affect concrete durability.

RESULTS AND DISCUSSION

INTRODUCTION:

In recent years, many coal fired electric generating plants have made operational changes to conform to more stringent air emission requirements. These changes have had negative effects on the volume of low LOI (<6.0%) fly ashes available. Many of these changes have resulted in increasing fly ash LOI content and often the LOI increased above 6.0%. In accordance with current Florida Department of Transportation Specifications, an upper limit for carbon content has been set at an LOI of 6%.

Due to shortage and limited supply of low LOI Class F fly ashes in Florida, this investigation was initiated to address the effects of blending high and low LOI class F fly ashes and to study the impact of blending of fly ashes on fresh and hardened concrete properties. In the following sections the results of this investigation will be presented.

AS-RECEIVED MATERIAL CHARACTERIZATION:

General

The materials used in this investigation included an ASTM and AASHTO approved Type II cement, #57 Brooksville limestone coarse aggregate, silica sand and Type D admixture. Three Class F fly ashes were used in this study. The ashes were from two sources. A single ash was used from the first source and had an LOI of 0.1% [FA1]. The other two ashes were obtained from a second source. These ashes had LOI values of 4.8 [FA2] and 14% [FA3]. The fly ashes were blended based on their mass and LOI content to produce fly ashes different LOI values. These included the following: 1.2, 3.3, 6.0, 7.1, 9.4, 10.8, and 11.9. The exact amount used from the as-received ashes in generating the blended ashes are presented in Tables 3 and 4. Mortar and concrete mixes were prepared for the blended and as-received fly ashes in addition to a plain control mix. The following sections will present the data for the as-receive material followed by mortar data and finally concrete data.

Chemical Analyses

Chemical analyses for the as-received fly ashes and cement was conducted in accordance with ASTM C311, AASHTO T105-87 and ASTM C114 procedures. The results presented in Table 1 indicate that FA 2 and 3 have similar silica and iron oxide content; however, they are lower in silica and higher in their iron oxide content than FA1. This is indicative of the differences in the coal source. In addition, the alkali content is lower for FA1 than FA 2 and 3. The analyses confirm that the ashes used for this study are in compliance with ASTM C618 classification for Class F fly ashes. However, FA3 is not in compliance with the LOI criteria set in the former.

Table 1: Oxide Chemical Analyses for the As-received Fly Ashes

| Oxide | FA 1 (%) | FA 2 (%) | FA3 (%) | Cement (%) |
|-------------------------------|-------------|-------------|------------|---------------|
| Silica | 62.44 | 47.38 | 43.77 | 20.9 |
| Alumina | 20.43 | 18.93 | 16.44 | 5.2 |
| Iron oxide | 5.43 | 17.97 | 13.02 | 3.9 |
| Calcium Oxide | 7.1 | 4.86 | 6.07 | 64.7 |
| Magnesia | 1.81 | 1.3 | 1.8 | 0.7 |
| Sulfur trioxide | 0.18 | 1.5 | 1.02 | 2.9 |
| Sodium oxide | 0.26 | 0.78 | 0.89 | |
| Potassium oxide | 0.96 | 2.29 | 1.87 | |
| Titanium Oxide | 1.45 | 1.05 | 0.85 | |
| P2O5 | 0.07 | 0.19 | 0.17 | |
| Mn2O3 | 0.08 | 0.05 | 0.05 | |
| SrO | 0.15 | 0.05 | 0.05 | |
| LOI | 0.1 | 4.8 | 14.0 | |
| Alkali (as Na2O) | 0.9 | 2.29 | 2.12 | |
| Moisture | 0.05 | 0.37 | 0.51 | |
| Silica+Alumina+ Iron oxide | 88.29 | 84.29 | 73.24 | |

Particle Size Distribution:

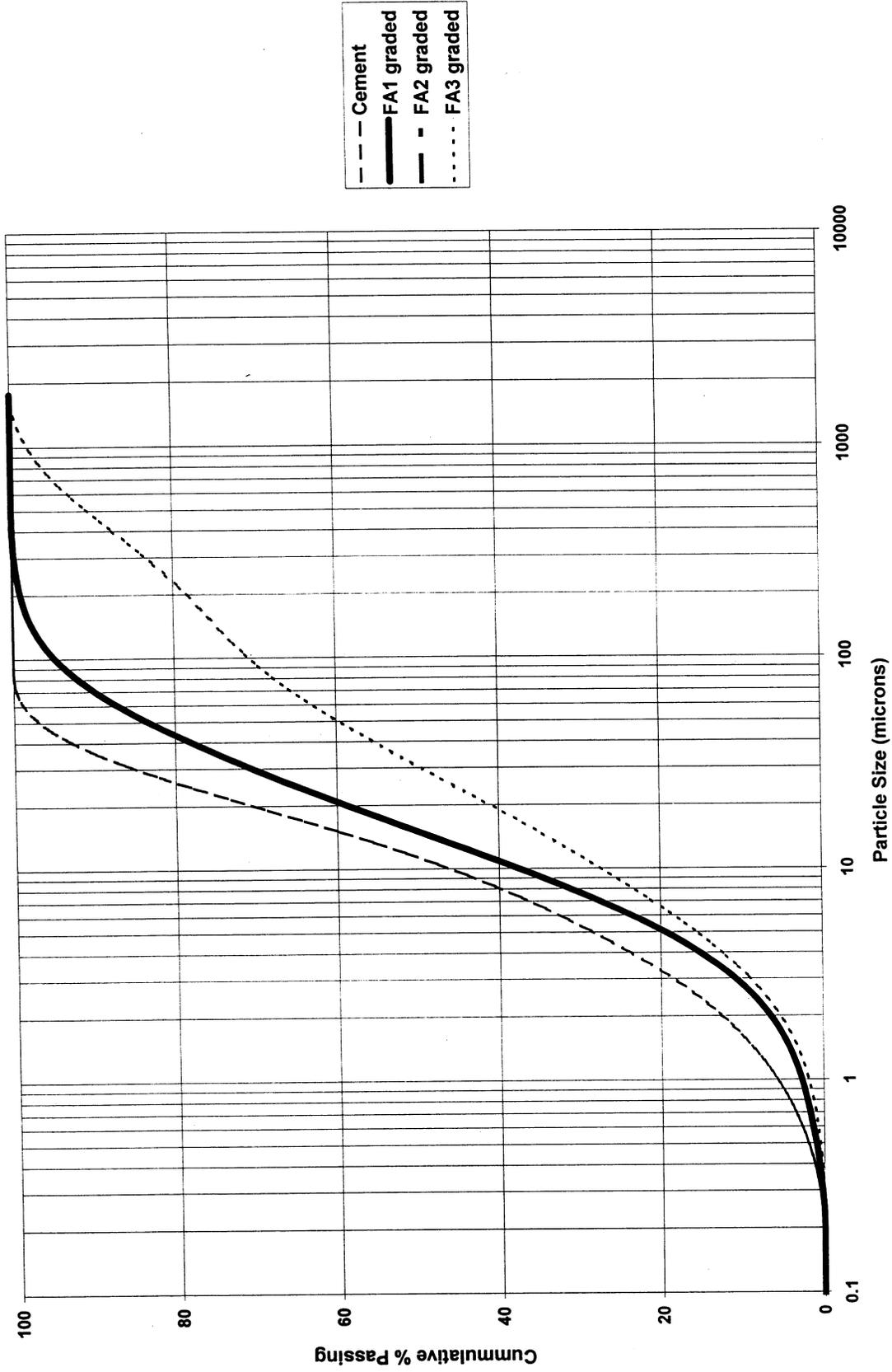
Particle size distribution was studied using Malvern Mastersizer (Model 2000) laser particle size analyzer. The results are depicted in Figure 1 for the cement and fly ashes. From Figure 1, when considering the results expressed as cumulative percent passing as a function of particle size, it can be seen that FA1 and FA2 displayed similar fineness for

the whole spectrum. However, FA3 showed coarser particle distribution down to particle size of one micron. Finer particle size distribution has been associated with higher reactivity for fly ashes and better burning conditions [1].

In Figures 2 through 6, particle size distribution data for the as-received ashes as well as for blended ashes is shown. For each fly ash, the data is depicted in two ways, first as percent of the material retained at that size and second as cumulative percent passing. Considering the graphs that depict the individual percent retained, it is interesting to note that with the exception of the unblended FA1, FA2 and FA3 had materials retained at particle size as high as 500 microns. In addition, FA3 had particles that were larger than 1000 microns. Another interesting feature for this same ash is the presence of a second peak around a particle size of approximately 350 microns. Though FA2 had significantly lower LOI than FA3, however, around the same particle size of 350 microns, a shoulder appears in the data for that ash too. It is not surprising that there is similarity between FA2 and FA3. That can be attributed to the fact that both ashes are generated from the same power plant. It is to be remembered that both FA2 and FA3 came from the same power plant and the only difference that was observed from their chemical analysis was the LOI content which in the case of the former was 4.8% while for the latter it was 14%.

Considering the distribution patterns for the blended ashes, it can be seen that for all the ashes containing FA3 in its blend, a similar peak to the one observed for FA3 is observed. In addition, the peak amplitude appears to be a function of the FA3 content in the blended ashes. This can be clearly seen when comparing the patterns for the blend with an LOI of 3.3% to that with an LOI of 11.9%. It is recognized that there are some peaks that display splitting and that is to be expected and even enhanced with increasing the variability in the practical methods of blending the ashes. Even with the existence of such splits, the trends are still clear. The only fly ash which did not display a reasonable peak formation was that with an LOI of 1.2%. This finding seems consistent with the previously discussed trend, as the blend did not include FA3.

Figure 1: Particle Size Distribution for As-Received Material



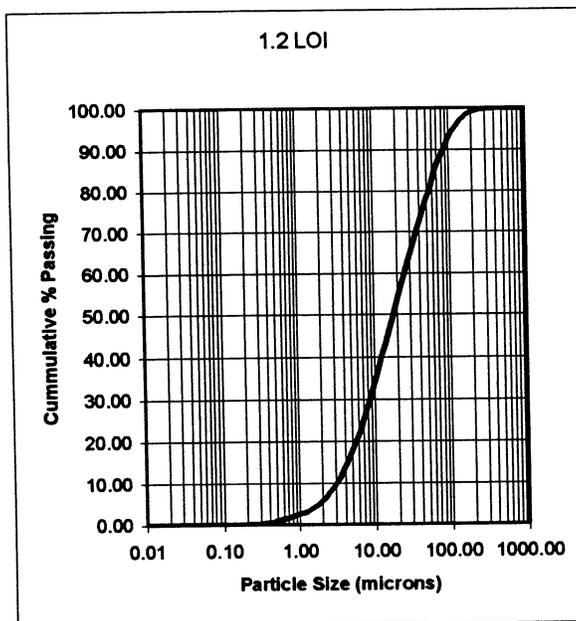
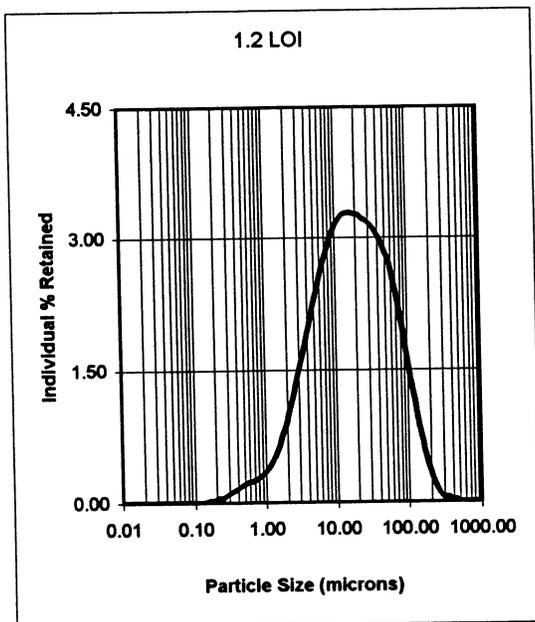
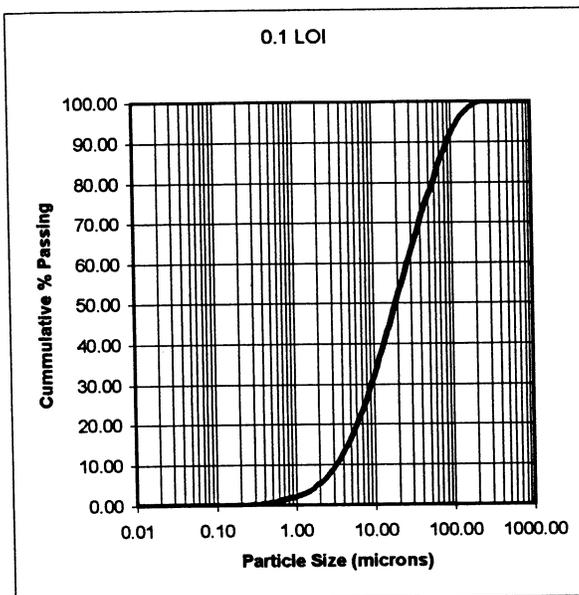
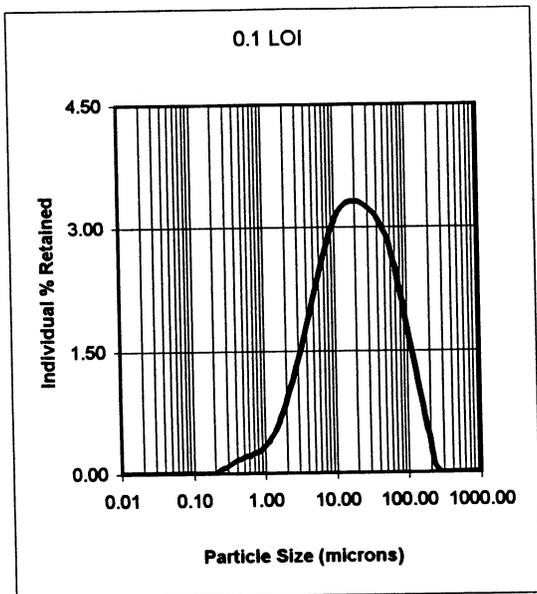


Figure 2. Particle Size Distribution for Fly Ashes with LOI of 0.1 and 1.2.

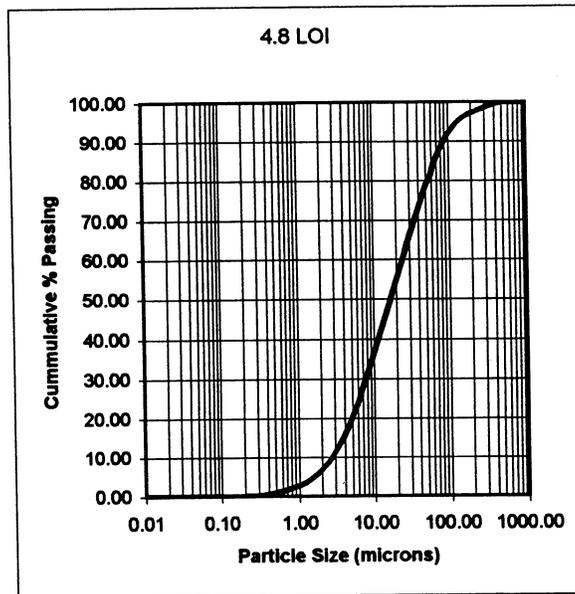
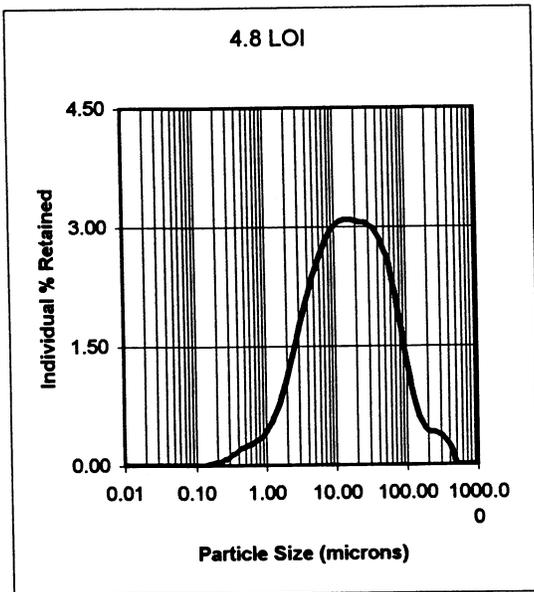
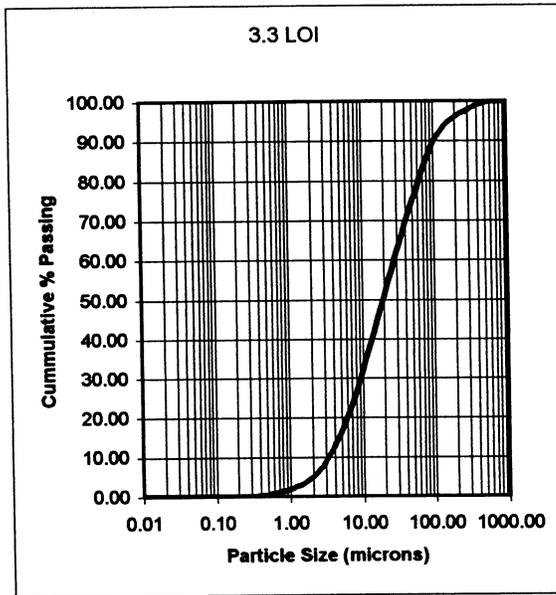
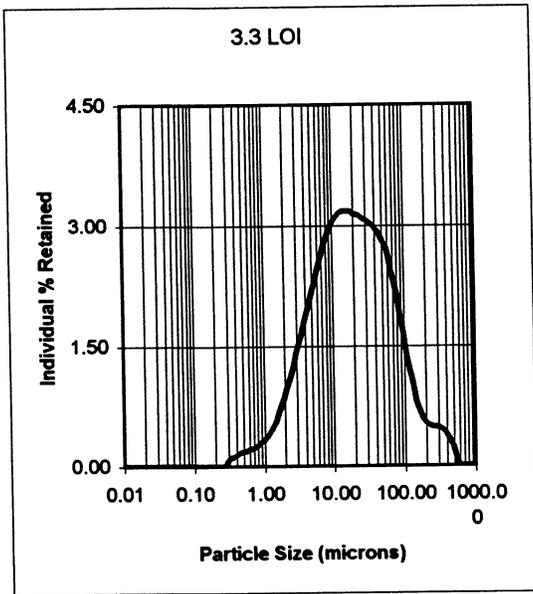


Figure 3. Particle Size Distribution for Fly Ashes with LOI of 3.3 and 4.8.

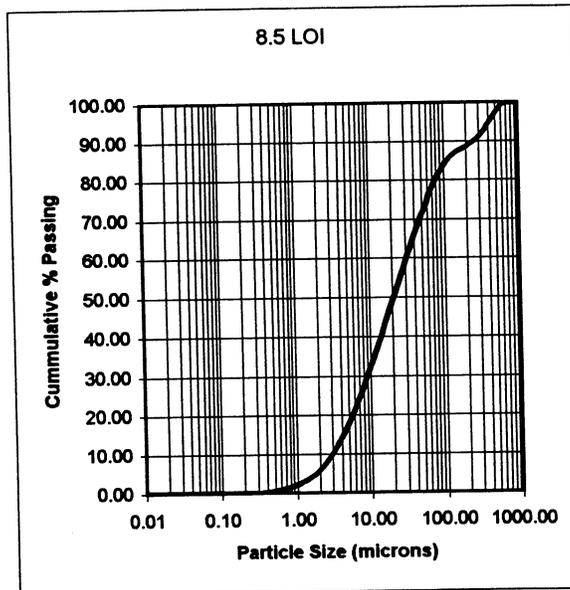
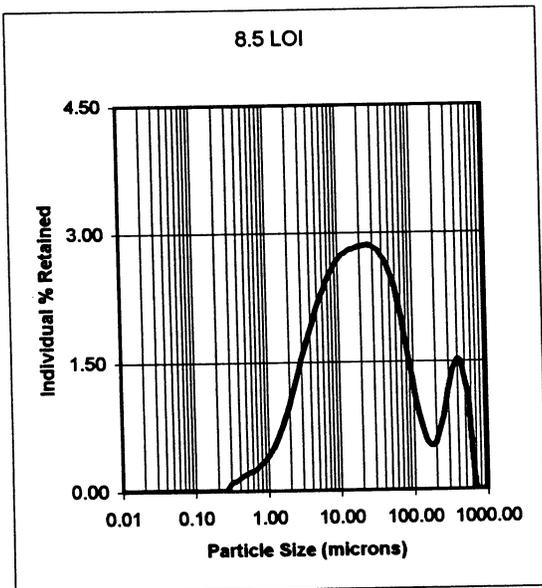
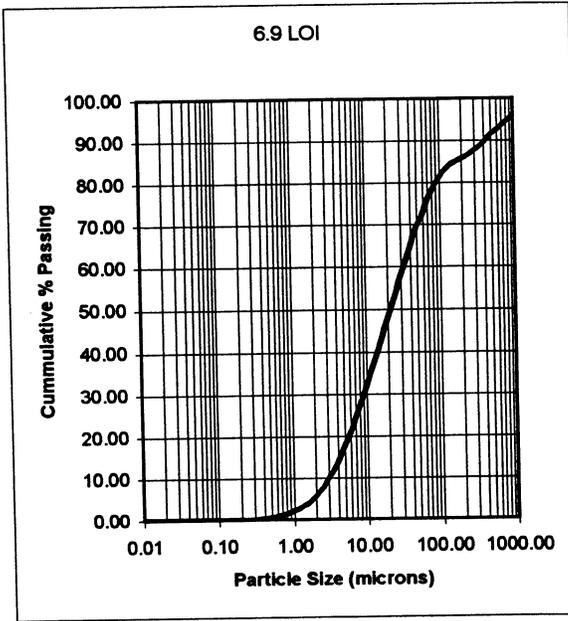
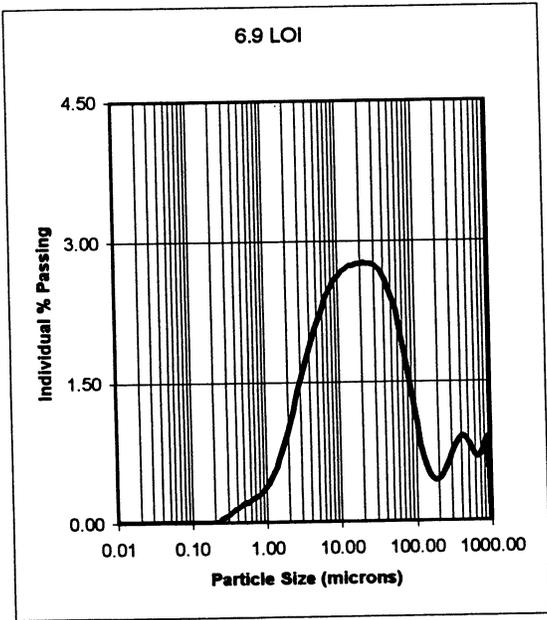


Figure 4. Particle Size Distribution for Fly Ashes with LOI of 6.9 and 8.5.

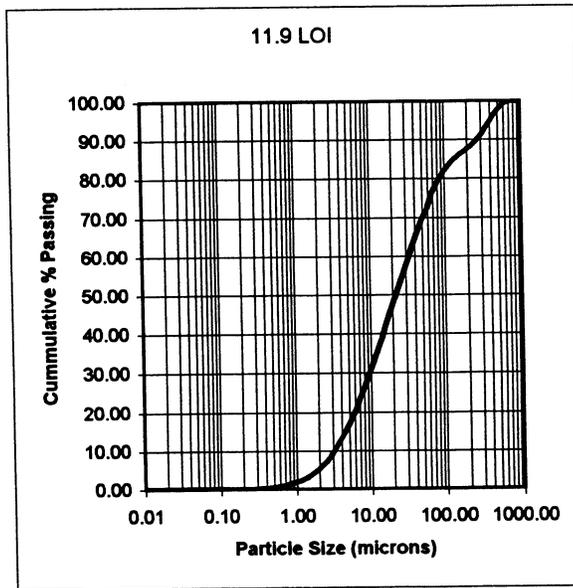
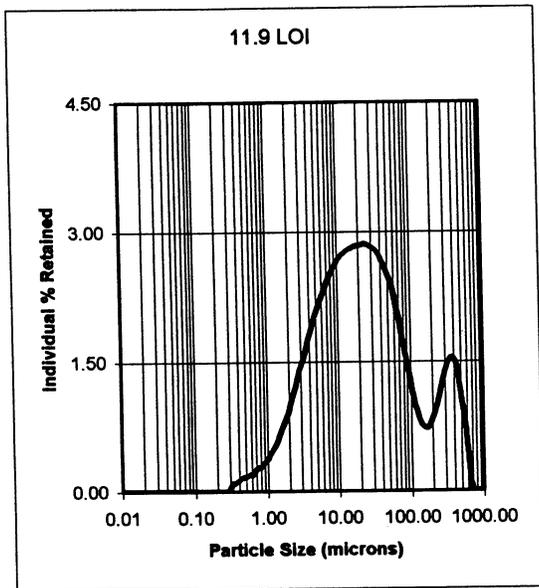
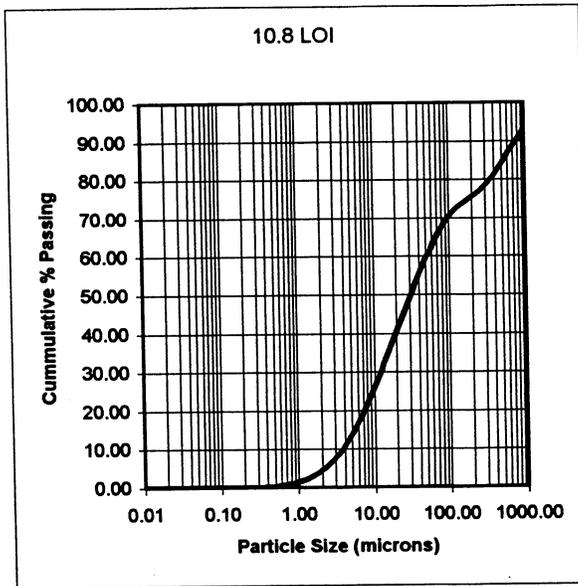
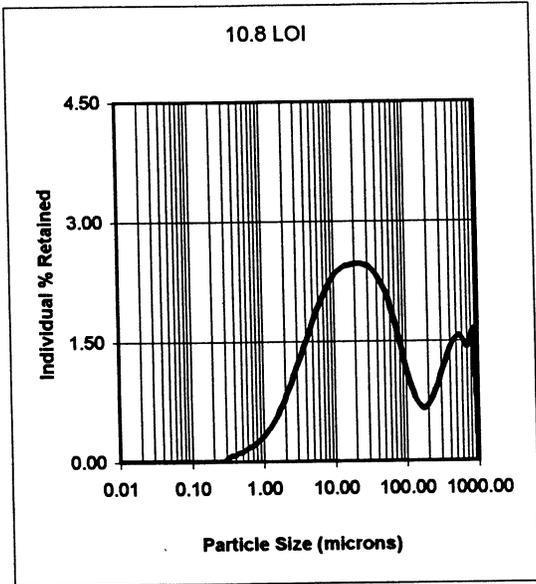


Figure 5. Particle Size Distribution for Fly Ashes with LOI of 10.8 and 11.9.

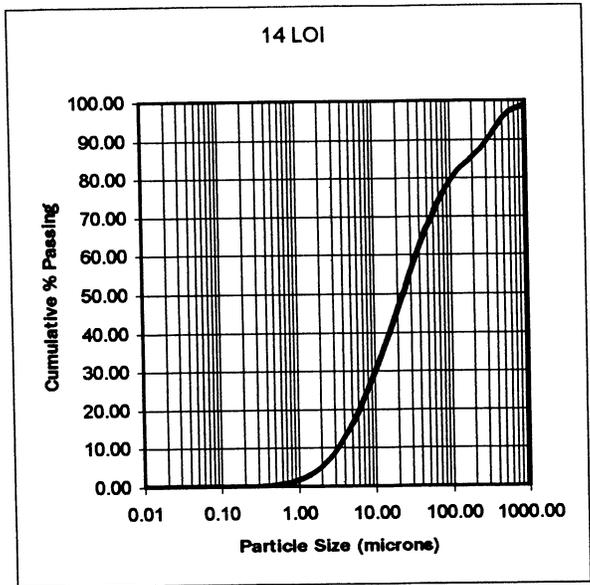
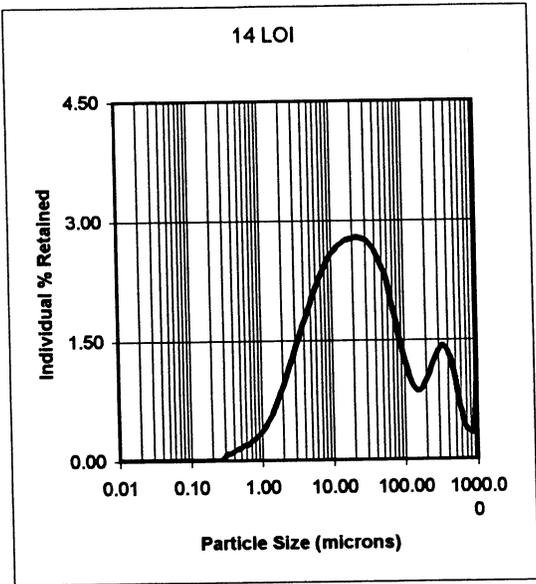


Figure 6. Particle Size Distribution for Fly Ash with LOI of 14.

Morphology:

The as-received fly ash material was examined under the optical microscope to assess the particle morphology. From the micrographs presented in Figure 7, 8 and 9, it can be observed that FA1 had well defined spherical particles. The average sphere diameter was approximately 20 microns. Larger spherical particles of 35 to 50 microns in diameter were also observed. There was no sign of contamination in this ash. For FA2, the particle size appeared to be slightly larger than FA1. In addition, contaminants were observed as seen in Figure 8. Spherical particles had an average diameter of 20 to 30 microns with particles as large as 50 to 60 microns. The contaminants were in the 15 to 30 microns range. For FA3 images shown in Figures 9a and 9b, visible contamination ranging in size from 30 to 100 microns can be seen. Contaminants with sizes greater than 300 microns were also observed. FA3 particles were observed to be largest among the as-received with cenospherical appearance and size range of 20 to 140 microns. The microscopic observation seems to be in agreement with particle size analysis where it was found that the lower the LOI, the finer the particle size distribution for a particular ash.

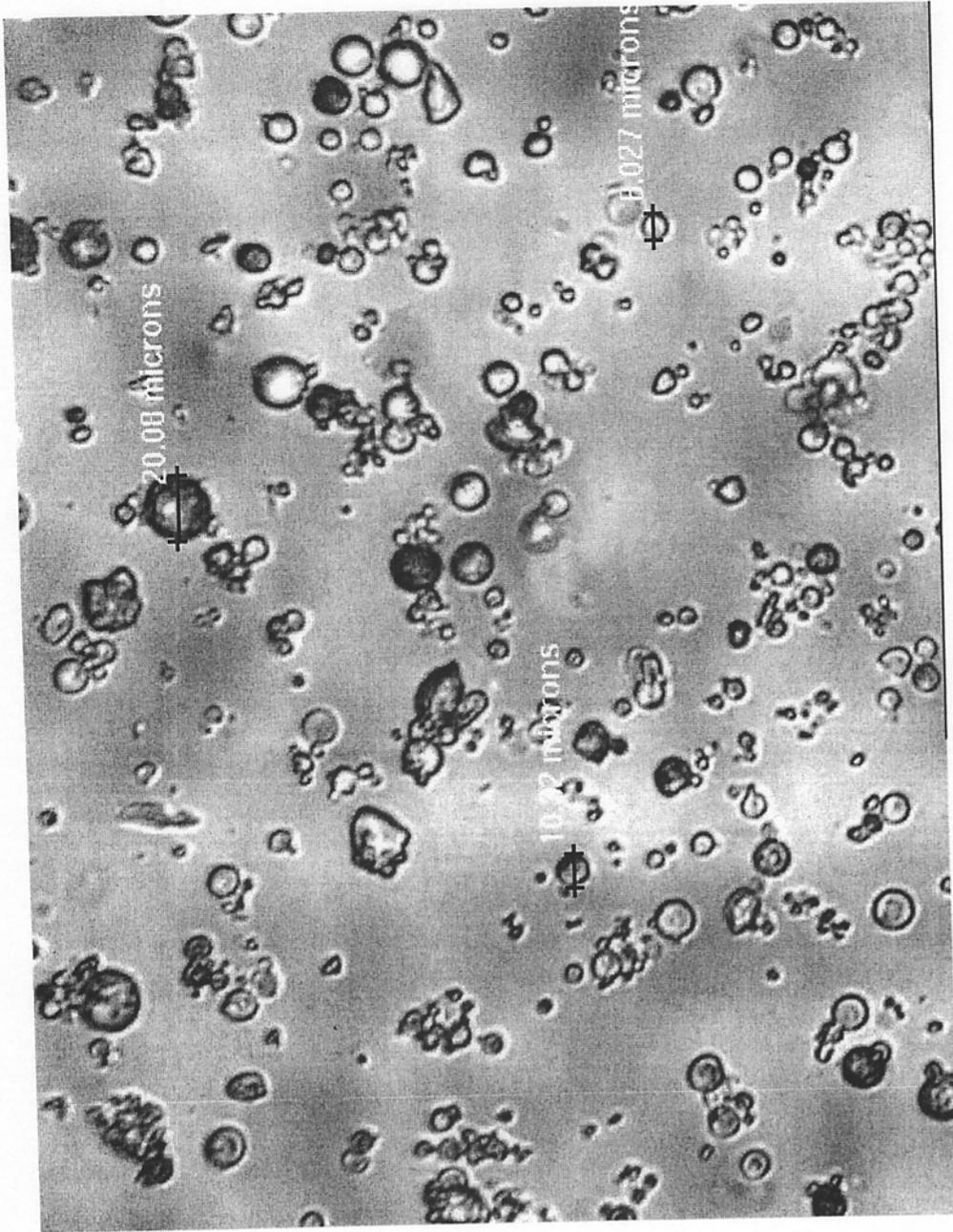


Figure 7: Morphology of FAI

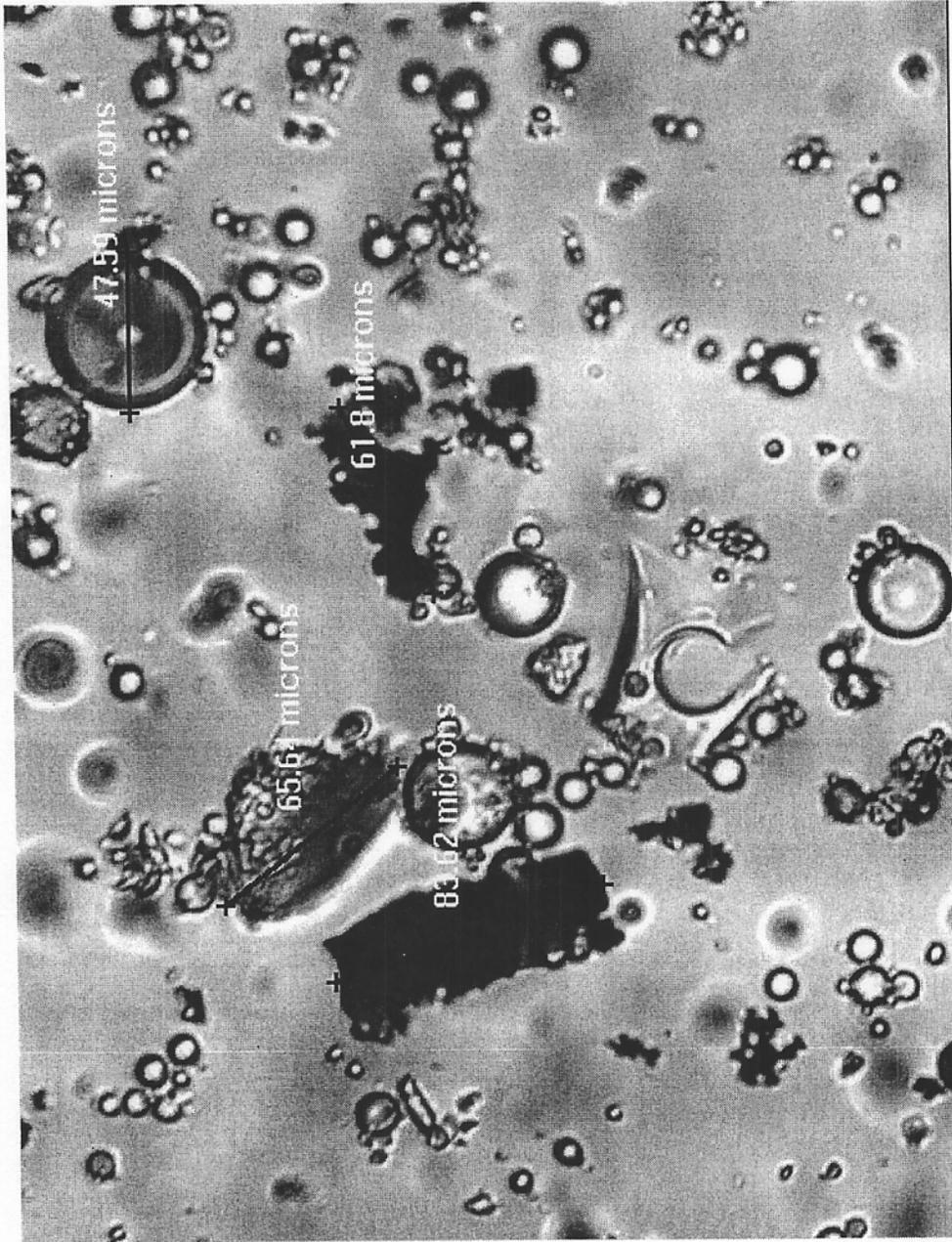


Figure 8: Morphology of FA2

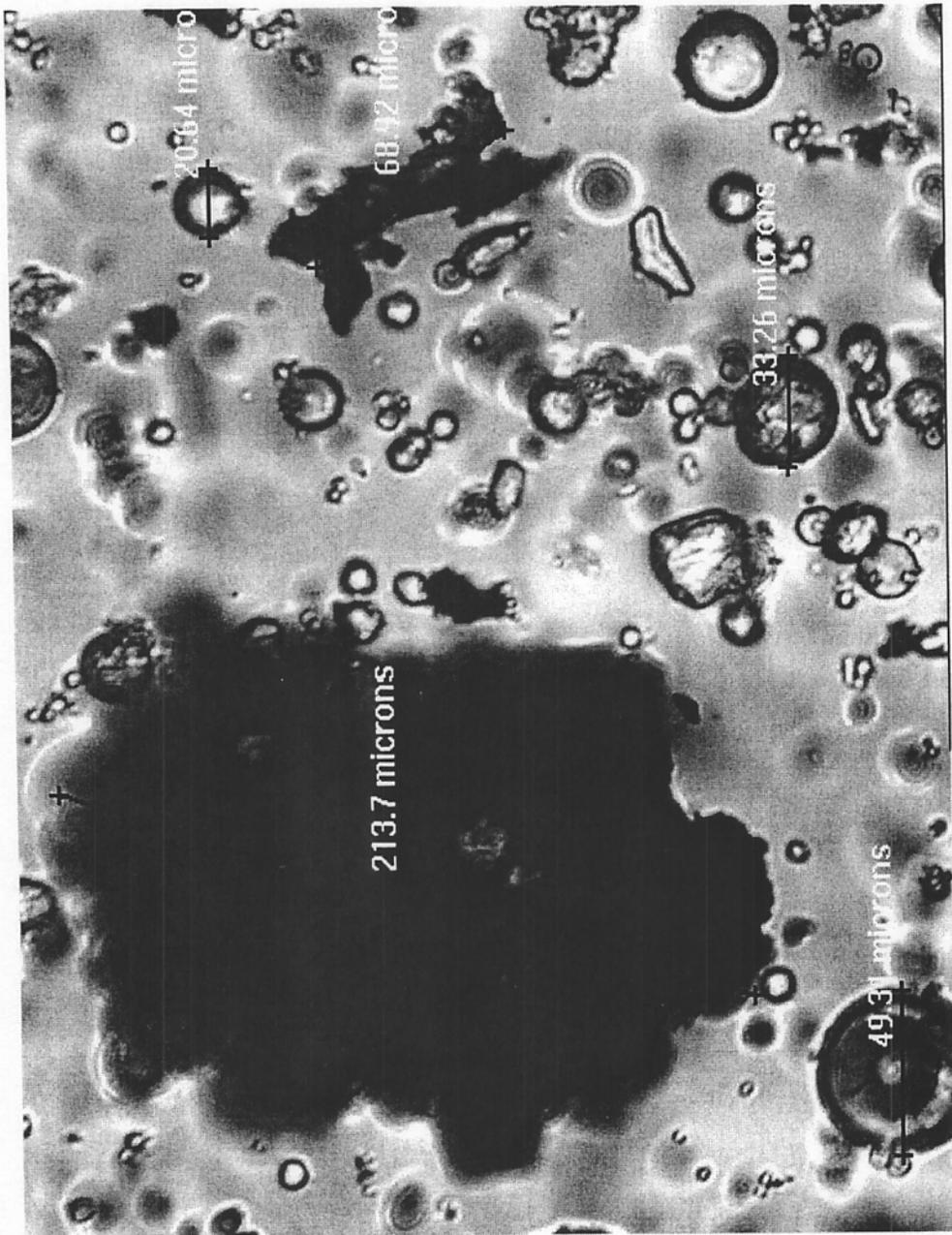


Figure 9 (a): Morphology of FA3

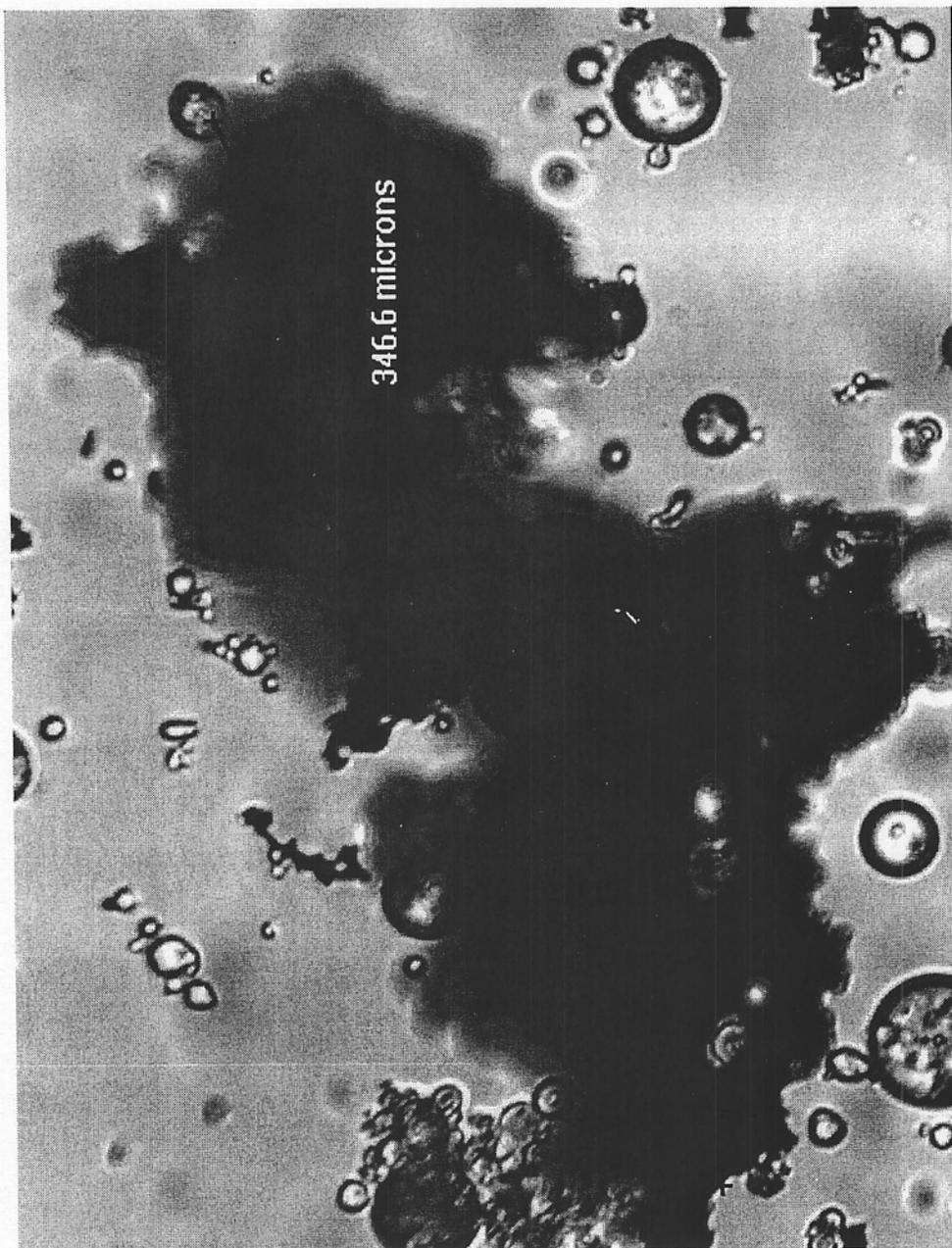


Figure 9 (b): Morphology of FA3

Mineralogical Analyses:

In studying the as-received fly ash material properties, quantitative and qualitative mineralogical analyses of the ashes was done using x-ray diffraction. The results presented in Table 2 and Figure 8 indicate that FA1 and 2 have similar amorphous content of approximately 78% while FA3 has a lower amorphous content of 68% on weight basis. In addition, quartz was higher in FA1 as compared to FA2 and 3, while the iron bearing phases (hematite and magnetite) were higher in FA2 and 3. These results are in agreement with oxide chemical analysis depicted earlier in Table 1.

From the above characterization tests, it can be concluded that FA1 and 2 are expected to have, comparatively, higher reactivity than FA3 due to their finer particle size distribution and amorphous content.

Table 2: Mineralogical Analyses of Fly Ashes

| Phases % | FA 1 | FA 2 | FA 3 |
|--------------------------|-------------|-------------|-------------|
| Quartz | 14.8 | 4.1 | 6.2 |
| Mullite | 6.3 | 3.5 | 4.2 |
| Hematite | 0.4 | 2.5 | 1.6 |
| Magnetite | | 6.4 | 4.5 |
| Anhydrite | | 1.0 | 1.1 |
| Lime | | | 0.3 |
| Periclase | | | 0.5 |
| Loss on Ignition | 0.1 | 4.8 | 14.0 |
| Amorphous content | 78.4 | 77.7 | 67.6 |

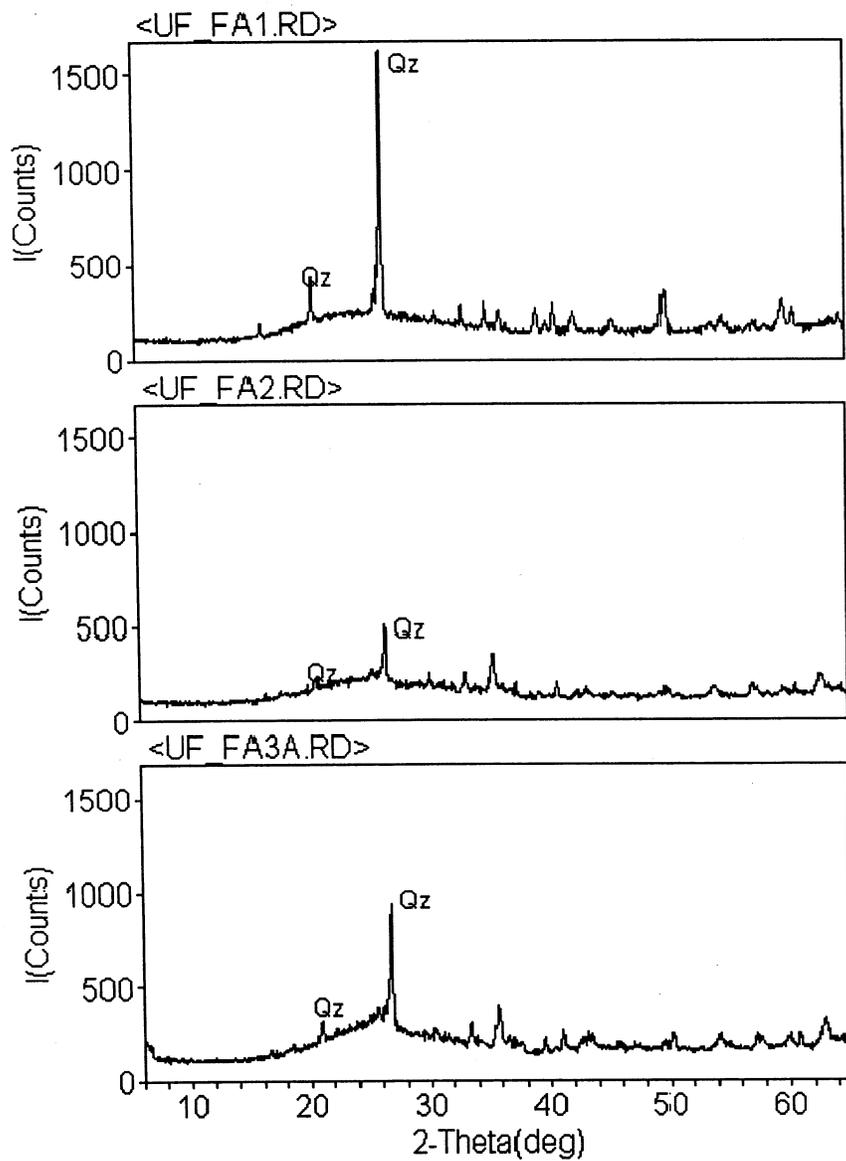


Figure 10: XRD Patterns for As-Received Ashes

Concrete Mix Design:

As mentioned before, the concrete mix used in this investigation was a Class II FDOT mix. The cement content for the control mixes contained 564 lbs /cyd of cement or 440 lbs/cyd cement and 124 lbs/cyd fly ash for the blended mixes. The target slump was 4.0 inches. Two series of concrete mixes were made. The first series included 10 mixes (a plain control mix and 9 mixes with fly ash). The mix quantities for this series are shown in Table 3. The target air content was 4.0% and the air-entraining admixture was varied in order to achieve the target air content. In the second series, 11 mixes (a plain control mix and 10 mixes with fly ash) were prepared. All of the mixes in the second series were prepared using a constant air-entraining admixture dosage. The mix designs for the second series are presented in Table 4.

For the first series, each concrete batch was 4.0 cft. Each batch was tested for fresh concrete properties that included slump, air content and unit weigh. Testing for hardened concrete properties included compressive strength and sulfate expansion. Test specimens cast for each batch included 52 (4"x 8") cylinders and nine 3"x3"x11 ½" bars for expansion monitoring in sulfate environment. For the compressive strength data, 21 cylinders were placed in saturated lime water solution and 21 cylinders were placed in 5 percent sodium sulfate solution (by mass). The test ages included the following: 3, 7, 28, 91, 182, 364 and 735 days for both solutions. For the expansion measurements, the number of bars per batch included 6 bars exposed to the sodium sulfate solution.

For the second series, the air entraining admixture dosage was maintained constant for all of the mixes. The water content and the water reducer admixture dosage was varied to maintain a constant target slump of 4 inches. Each concrete batch had a volume of 1.5 cft. Slump, air content, unit weight were performed on each fresh batch in addition to compressive strength and rapid chloride permeability. Twenty six concrete cylinders [4"x 8"] were cast and stored in a 5 percent sodium sulfate solution and saturated lime until the age of testing. In the second series, two additional mixes were prepared; namely, control and 0.1 LOI mix. These additional mixes were prepared in the same proportions listed in Table 4 with the only exception of not including air-entraining admixture. The purpose of these additional mixes was to generate for these two the same

measured air content, so that variation in air content can be excluded when interpreting compressive strength data.

Concrete Fresh Properties:

The effect of the LOI on fresh concrete properties was evident. The air entraining dosage required to maintain a 4 inch slump increased with increasing fly ash LOI content in agreement with literature [1,2,3,5]. This trend can be seen for the first concrete mix series as shown in Table 3. The effect of LOI on the efficiency of air-entraining agents is also demonstrated in the second series of mixes. From Table 4 and Figure 11, it can be seen that at low LOI, a standard dosage of 2 oz/cyd produced 3 to 3.8% air (mixes numbers 2543 and 2544). In mixes 2547 (LOI= 4.8%) through 2553 (LOI=14%) there is only the normally entrapped air (1%) with no air entrainment.

Considering the effect of LOI on water demand, Table 3 and 4 show that as the LOI increases there is an increase in the water demand required to maintain a constant slump. These results are in agreement with the findings reported by others [1,2,4,6,7]. Figure 12 shows the impact of increasing LOI on the water demand for the second series of mixes. The general trend of data indicates that as the LOI increases, the water demand increases. The water demand, required to maintain a constant slump, increased from 260 (LOI = 0.1%) up to 298 lbs/cyd (LOI= 14%). The results, therefore, seems to indicate that for an increase in percent loss on ignition of 13.9%, there seems to be a corresponding increase in water demand of approximately 13% (Table 4).

Table 3: Mix Design for Concrete Mixes (Series 1)
(Constant measured air and constant slump)

| Mix # | 2017 | 2037 | 2111 | 2108 | 2042 | 2067 | 2069 | 2095 | 2106 | 2043 |
|-----------------------|------|------|------|------|------|------|------|------|------|------|
| LOI % | 0.0 | 0.1 | 1.2 | 3.3 | 4.8 | 7.1 | 9.4 | 10.8 | 11.9 | 14.0 |
| Water lbs/cyd | 246 | 240 | 254 | 256 | 249 | 246 | 251 | 258 | 256 | 261 |
| Cement (lbs/cyd) | 566 | 441 | 437 | 439 | 427 | 436 | 445 | 442 | 438 | 444 |
| FA 1 (LOI = 0.1%) | | 124 | 95 | 95 | | 62 | | 29 | | |
| FA 2 (LOI = 4.8%) | | | 28 | | 121 | | 63 | | 28 | |
| FA 3 (LOI = 14.0%) | | | | 28 | | 62 | 63 | 96 | 95 | 125 |
| Coarse Agg (lbs/cyd) | 1815 | 1812 | 1798 | 1805 | 1759 | 1793 | 1830 | 1817 | 1802 | 1825 |
| Fine Agg (lbs/cyd) | 1189 | 1187 | 1190 | 1183 | 1153 | 1158 | 1199 | 1190 | 1180 | 1196 |
| Admixture (oz/cyd) | 22.6 | 17.6 | 17.5 | 17.6 | 17.1 | 17.4 | 17.8 | 17.7 | 17.5 | 17.8 |
| AEA (oz/cyd) | 2.0 | 2.0 | 5.0 | 8.5 | 16.5 | 11.9 | 14.2 | 8.5 | 14.9 | 10.6 |
| Slump (inch) | 4.00 | 4.00 | 4.25 | 4.00 | 4.00 | 4.50 | 4.00 | 4.00 | 4.00 | 4.00 |
| Air (%) | 4.6 | 4.5 | 3.8 | 4.1 | 4.8 | 4.2 | 4.0 | 3.8 | 3.8 | 4.0 |
| Unit weight (lbs/cft) | 141 | 141 | 141 | 141 | 137 | 139 | 143 | 142 | 141 | 143 |

Table 4: Mix Design for Concrete Mixes (Series 2)
(Constant Air-Entraining Agent Dosage and Constant Slump)

| | | | | | | | | | | | |
|-----------------------|------|------|------|------|------|------|------|------|------|------|------|
| Mix # | 2543 | 2544 | 2545 | 2546 | 2547 | 2548 | 2549 | 2550 | 2551 | 2552 | 2553 |
| LOI % | 0.0 | 0.1 | 1.2 | 3.3 | 4.8 | 6.0 | 7.1 | 8.5 | 10.8 | 11.9 | 14.0 |
| Water (lbs/cyd) | 260 | 260 | 264 | 269 | 275 | 284 | 281 | 286 | 286 | 288 | 298 |
| Cement (lbs/cyd) | 562 | 450 | 448 | 444 | 446 | 446 | 441 | 443 | 442 | 441 | 439 |
| FA 1 (LOI = 0.1%) | | 127 | 97 | 96 | | | 62 | | 29 | | |
| FA 2 (LOI = 4.8%) | | | 29 | | 126 | 109 | | 62 | | 28 | |
| FA 3 (LOI = 14.0%) | | | | 29 | | 16 | 62 | 62 | 96 | 96 | 124 |
| Coarse Agg (lbs/cyd) | 1841 | 1819 | 1869 | 1862 | 1871 | 1871 | 1849 | 1857 | 1854 | 1848 | 1840 |
| Fine Agg (lbs/cyd) | 1183 | 1168 | 1197 | 1196 | 1202 | 1202 | 1188 | 1192 | 1190 | 1187 | 1182 |
| Admixture (oz/cyd) | 16.9 | 13.5 | 13.4 | 13.3 | 13.4 | 13.4 | 13.4 | 13.2 | 13.3 | 13.2 | 13.2 |
| AEA (oz/cyd) | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| Slump (inch) | 4.25 | 4.50 | 4.25 | 4.25 | 4.00 | 4.25 | 4.50 | 4.50 | 4.25 | 4.00 | 4.25 |
| Air (%) | 3.8 | 3.0 | 2.0 | 1.8 | 1.0 | 1.0 | 1.2 | 1.0 | 1.1 | 1.1 | 1.1 |
| Unit weight (lbs/cft) | 143 | 142 | 145 | 144 | 145 | 146 | 144 | 145 | 144 | 144 | 144 |

Figure 11: Efficiency of Air-Entrainment (Series 2)

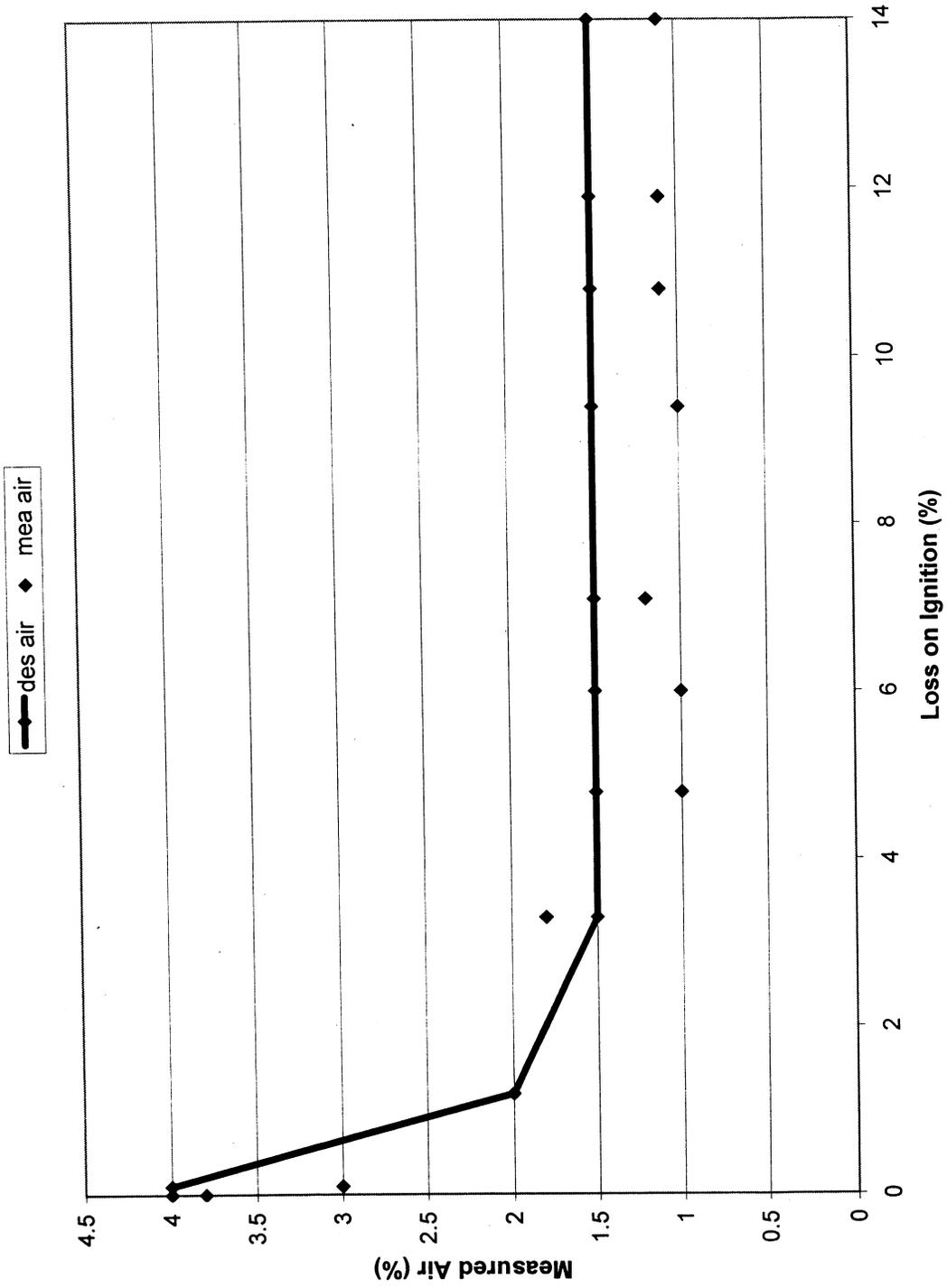
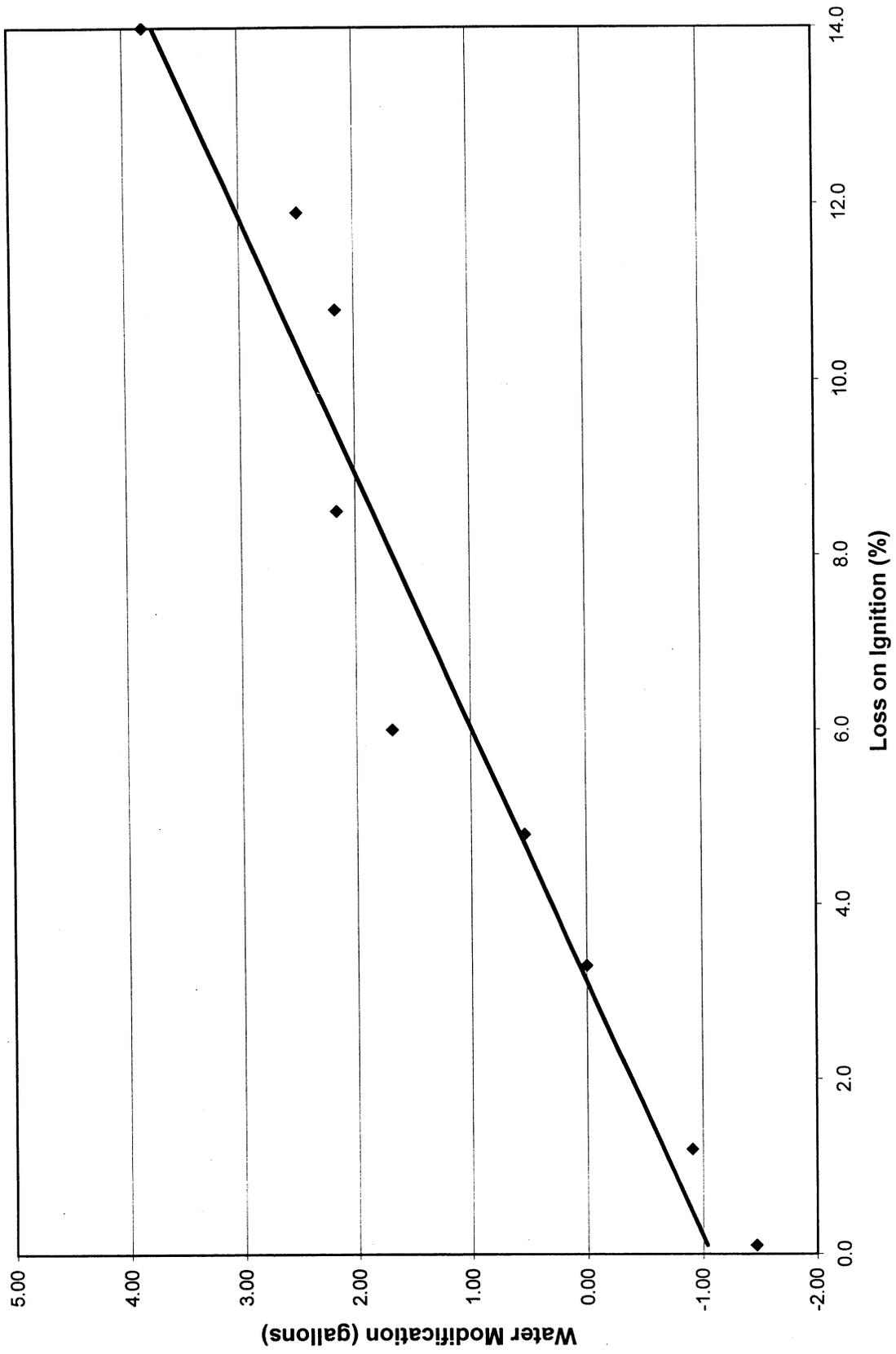


Figure 12: Effect of LOI on Water Required for Constant Slump (Series 2)



Concrete Compressive Strength:

Results for the compressive strength variation with respect to curing time in both the lime and sulfate environments are depicted in Figures 13 and 16 respectively, for the first series. It is to be remembered that those batches were prepared with aggregates that were graded but not proportioned. The air-entraining was also varied to achieve constant air content of 4%. Figures 13 and 14 show the strength for all the mixes as a function of curing ages in lime solution and sulfate environment. In Figure 13, it can be seen that the strength of all mixes started to level off at 365 days. At 28 days all mixes had higher compressive strength than the control except for the 14% LOI mix. That mix never attained the control compressive strength for all ages tested. None of the mixes experienced any strength drop on exposure to the sulfate environment, Figure 14. The only exception is for the control mix where a drop in compressive strength was noticed at 365 days.

To determine the effect of fly ash particle properties on the compressive strength of concrete and the blended mixes, compressive strength data were plotted as a function of mixes LOI. Figures 15 and 16 depict such variation for lime and sulfate curing. The trends are similar in both environments. At early age, there is minimal negative slope for the strength variation with increasing the LOI. This is to be expected as the pozzolanic reaction is not very active up to 28 days of hydration. However, at 735 days the trend seems to indicate more negative slope. This is indicative of the limitations imposed by higher content of unreactive carbon on concrete compressive strength.

Figure 13: Variation of Compressive Strength as a Function of Exposure Time in Saturated lime Solution (Series 1)

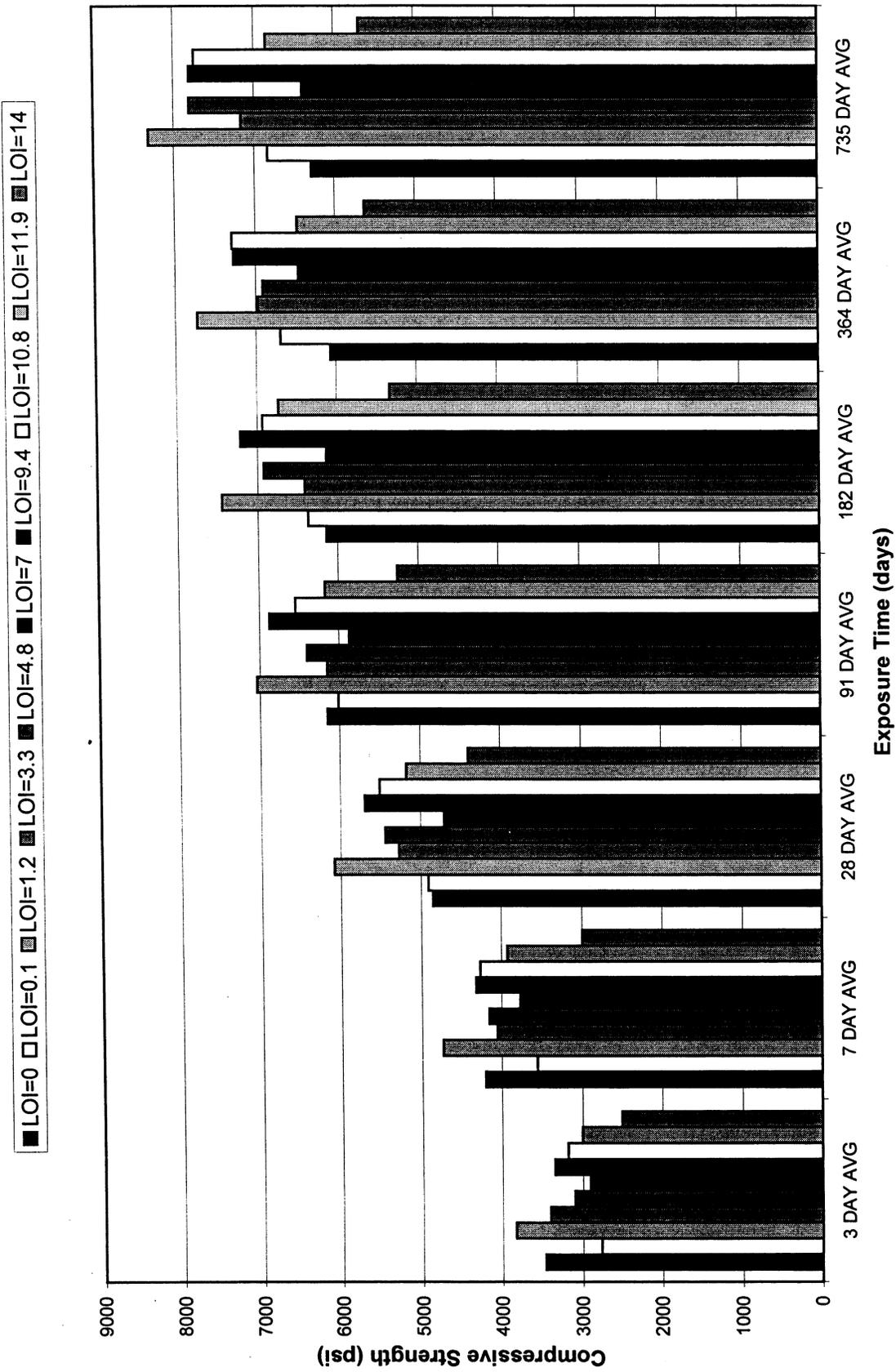
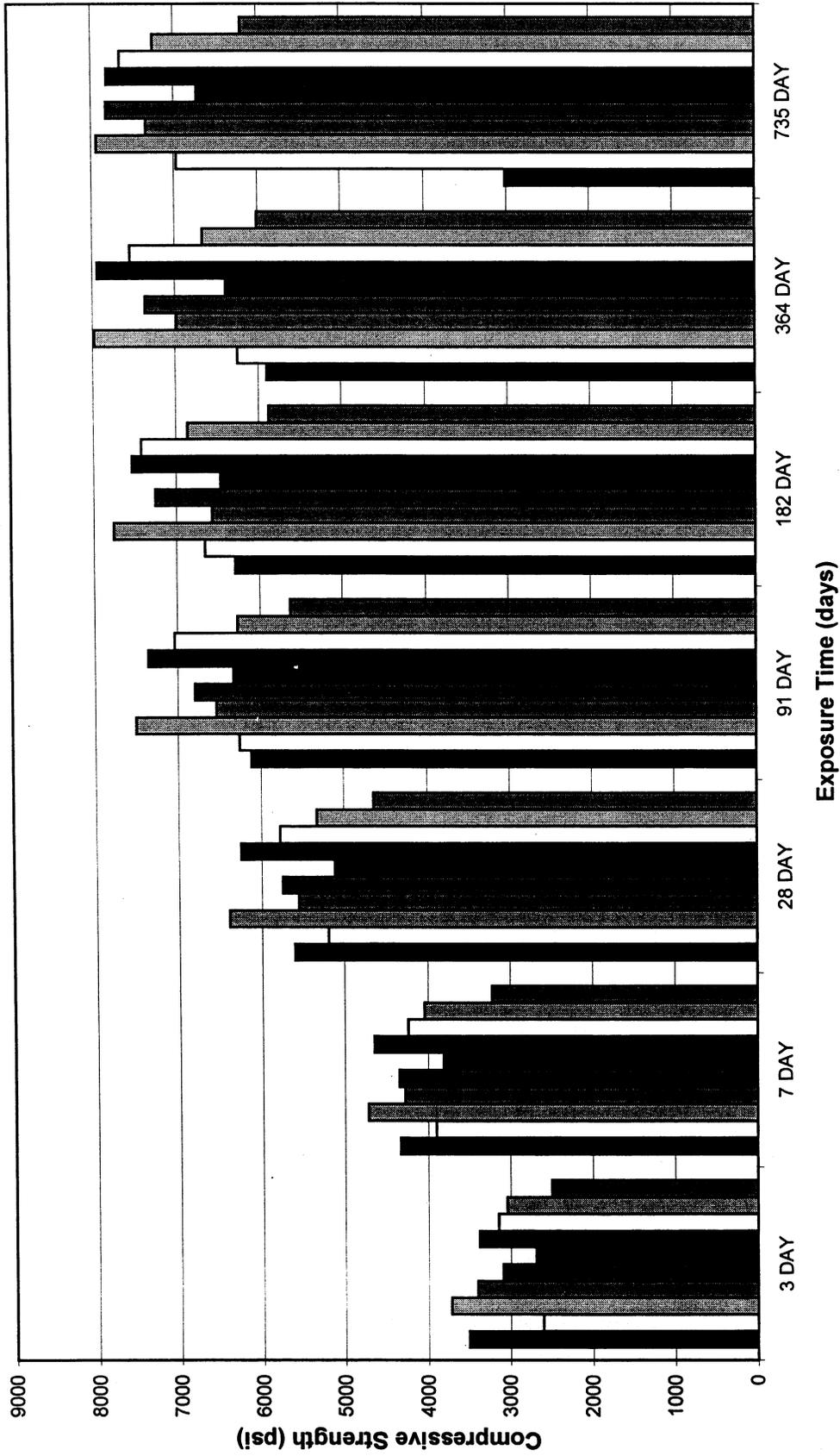


Figure 14: Variation of Compressive Strength with Exposure Time to Sulfate Environment (Series 1)

■ LOI=0 □ LOI=0.1 ▨ LOI=1.2 ▩ LOI=3.3 ■ LOI=4.8 ■ LOI=7 ■ LOI=9.4 □ LOI=10.8 ▨ LOI=11.9 ■ LOI=14



**Figure 15: Variation of Compressive Strength as a Function of LOI (Series 1)
in Saturated Lime Solution**

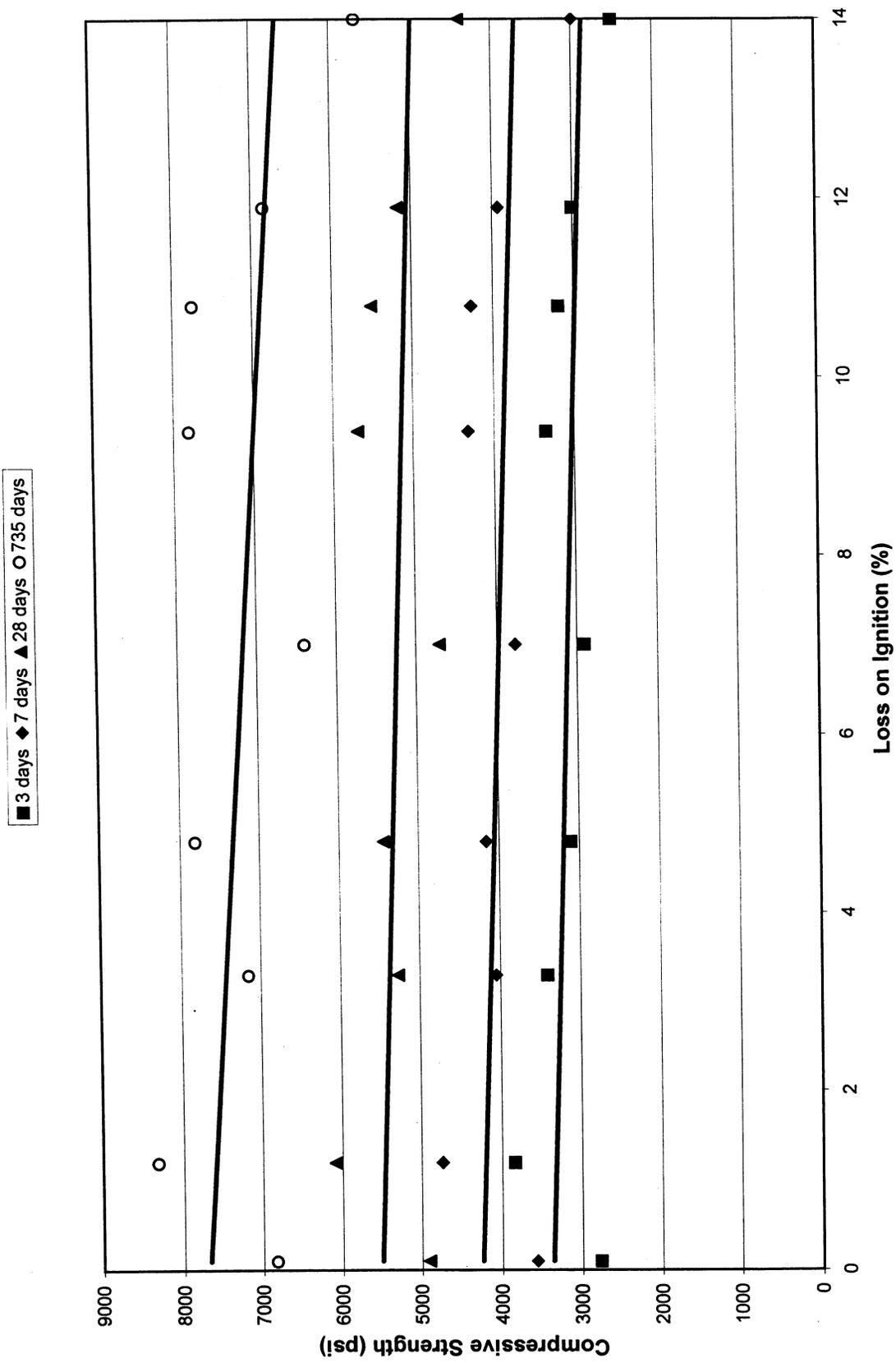
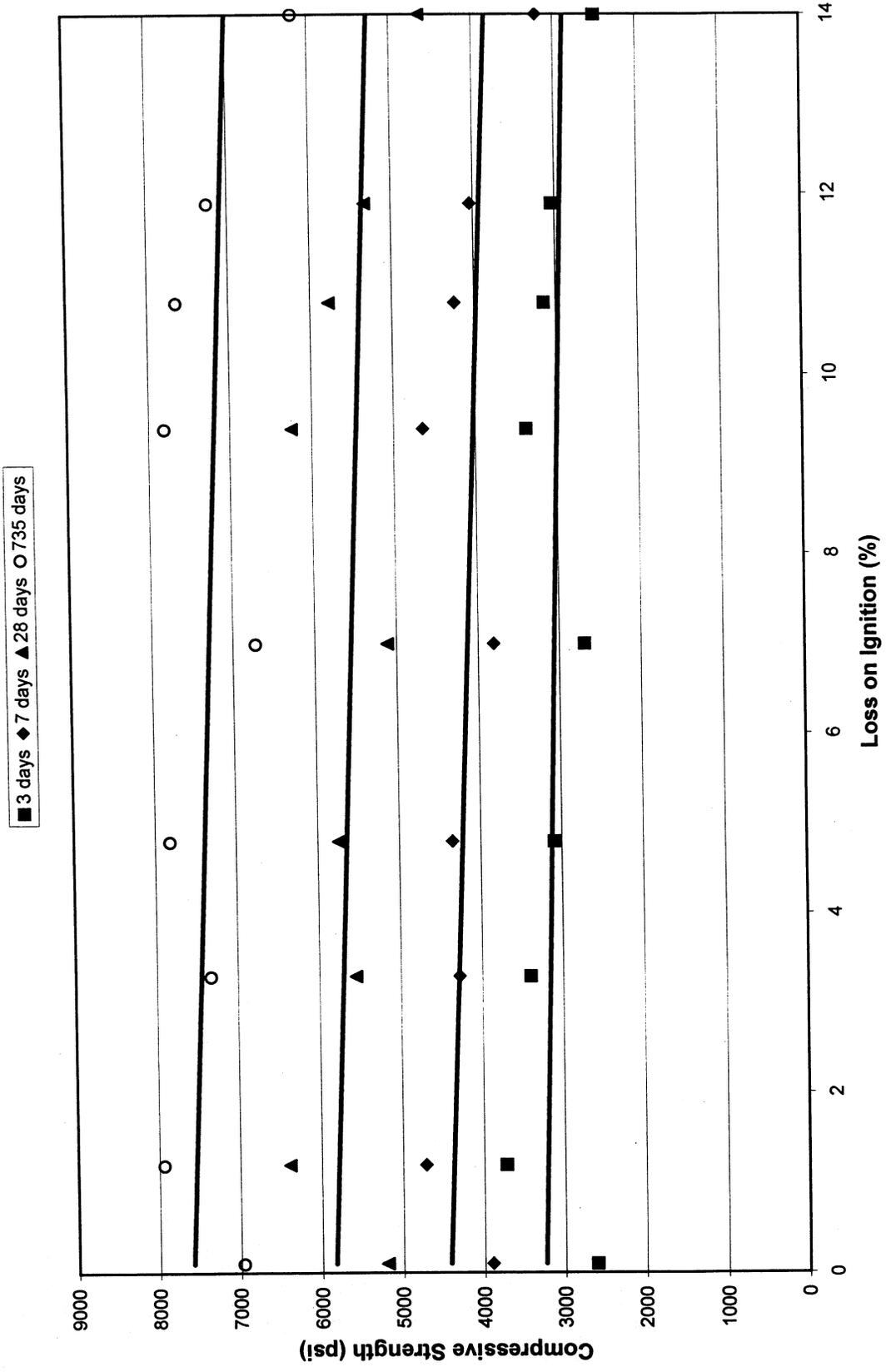


Figure 16: Variation of Compressive Strength as a Function of LOI for Concrete (Series 1) Exposed to Sulfate Environment



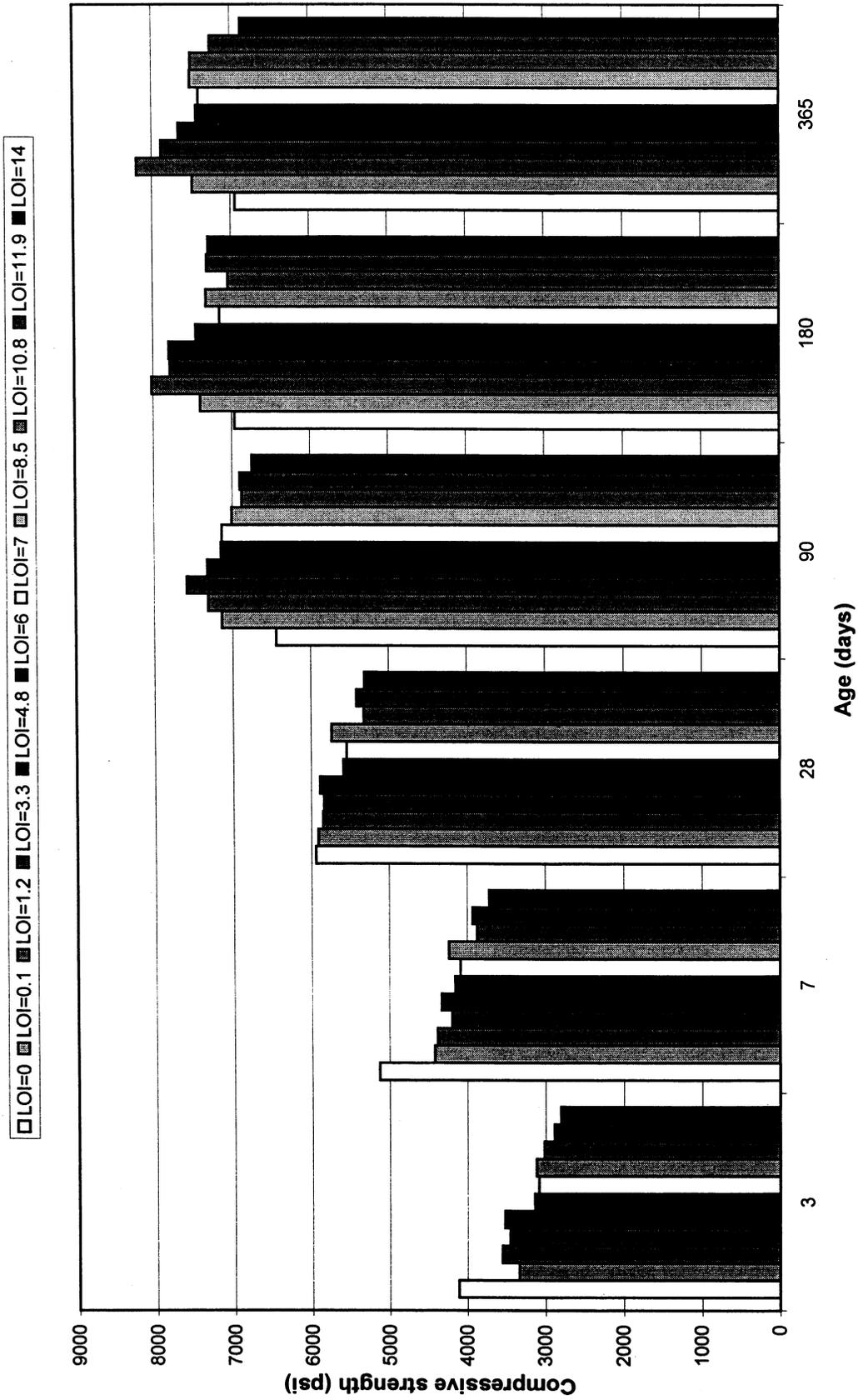
The compressive strength data for concrete mixes prepared with constant air dosage, but a variable measured air content, is shown in Figure 17 through 21. When compared to the constant air dose control batch, all the fly ash blended mixes showed lower strength than the control at early age; that is, up to 28 days. At 28 days, fly ash mixes of LOI less than 6% had comparable strength to the control. However, at 90 days, all blended ashes showed higher compressive strength than the control mix as can be seen from Figure 17. At 365 days, some of the high LOI blends started to show reasonable drop in strength due to sulfate exposure. This can be seen through considering the trendlines in Figure 18. An overlap occurs at 365 days strength data and 180 days for the higher LOI mixes. This is attributed to strength deterioration in blended mixes. Figure 18 also shows the apparent decrease in concrete strength with increasing LOI of the blended concrete mixes.

Additional information can be collected (Figure 19) when the strength of the blended fly ash mixes is compared to the control mix and the 0.1 mix batched to constant air content; that is, no air-entraining admixture. It can be seen that the strength of the plain mix is higher than all blended mixes up to 28 days. At 90 days, the strength of the control mix seems to maintain higher values than mixes containing fly ashes of LOI of 6% and higher. The extent of deterioration seems to be a function of LOI. This can be seen through considering Figure 20 where again it can be seen that there is an overlap in strength data at high LOI values at ages of 180 and 365 days. Figure 21 shows the strength of the unblended fly ash mixes and the control mix, all prepared to a constant air content. It can be seen that the control mix maintained highest strength up to 60 days. At 365 days of curing, all mixes had similar strength except for the mix containing LOI value of 14, which showed low strength gain potential over an extended period of time.

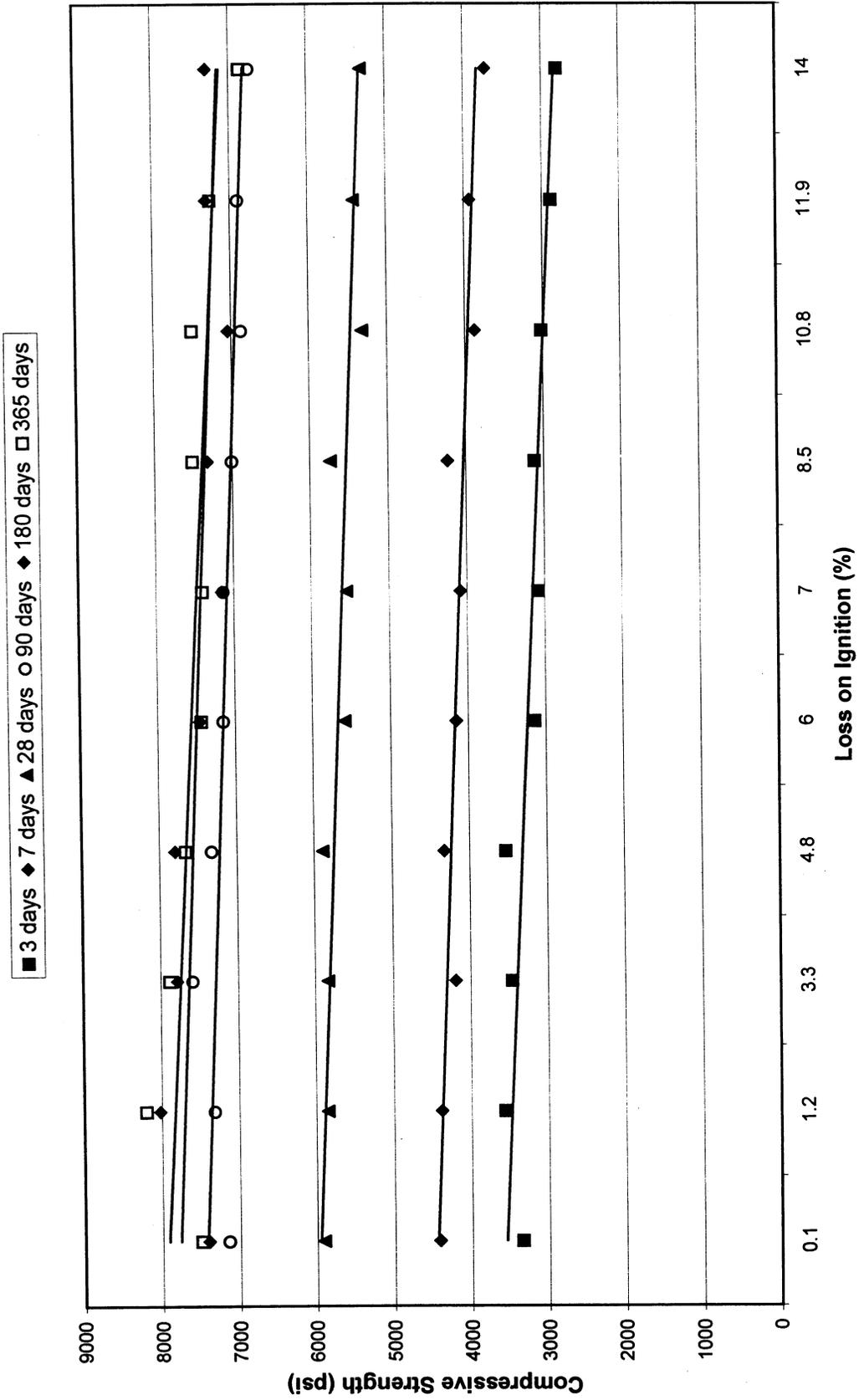
In conclusion, it is clear from the results of concrete strength prepared under several mix options, that there is a decrease in strength with an increase in the blended LOI mixes or the pure LOI mixes. Exposure to sulfate environment revealed, in general, a higher deterioration rate for specimens containing high LOI blends. From the micrographs presented earlier it was observed that FA2 and 3 showed contamination with particles larger than 100 microns. The amount observed was higher in FA3 with an LOI

of 14% than FA2 with an LOI of 4.8%. In addition, from mineralogical analysis conducted in this investigation, it was shown that excluding the LOI content, the fly ashes glassy content seems to be similar. However, the fly ashes are not expected to have similar reactivity due to particle size distribution differences. Particle size distribution results seem to be in line with LOI; that is, increasing the fly ash LOI seems to increase particles' coarseness. It is clear from the results that as the LOI increases, not only the reactive component in the fly ash decreases but the particle size of the pozzolan seems to become coarser. This would yield a fly ash which is less reactive with less potential of contributing to concrete strength.

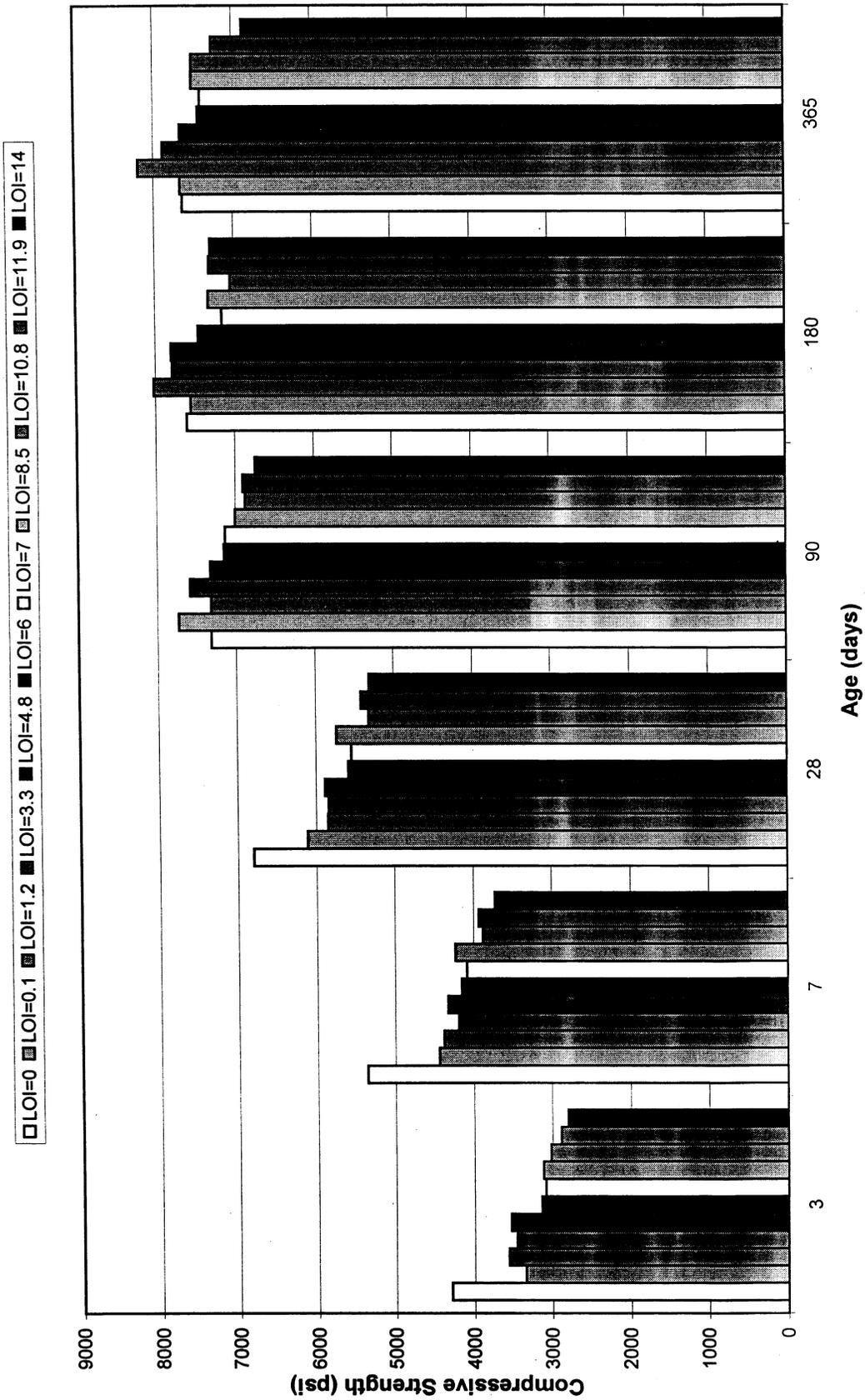
**Figure 17: Variation of Compressive Strength with Curing Time
(Series 2, Constant AEA Dosage)**



**Figure 18: Variation of Compressive Strength as a Function of LOI
(Series 2, Constant AEA Dosage)**



**Figure 19: Variation of Compressive Strength with Curing Time
(Series 2, Control and 0.1 prepared with no AEA)**



**Figure 20: Variation of Compressive Strength as a Function of LOI
(Series 2, Control & 0.1 Contain No AEA)**

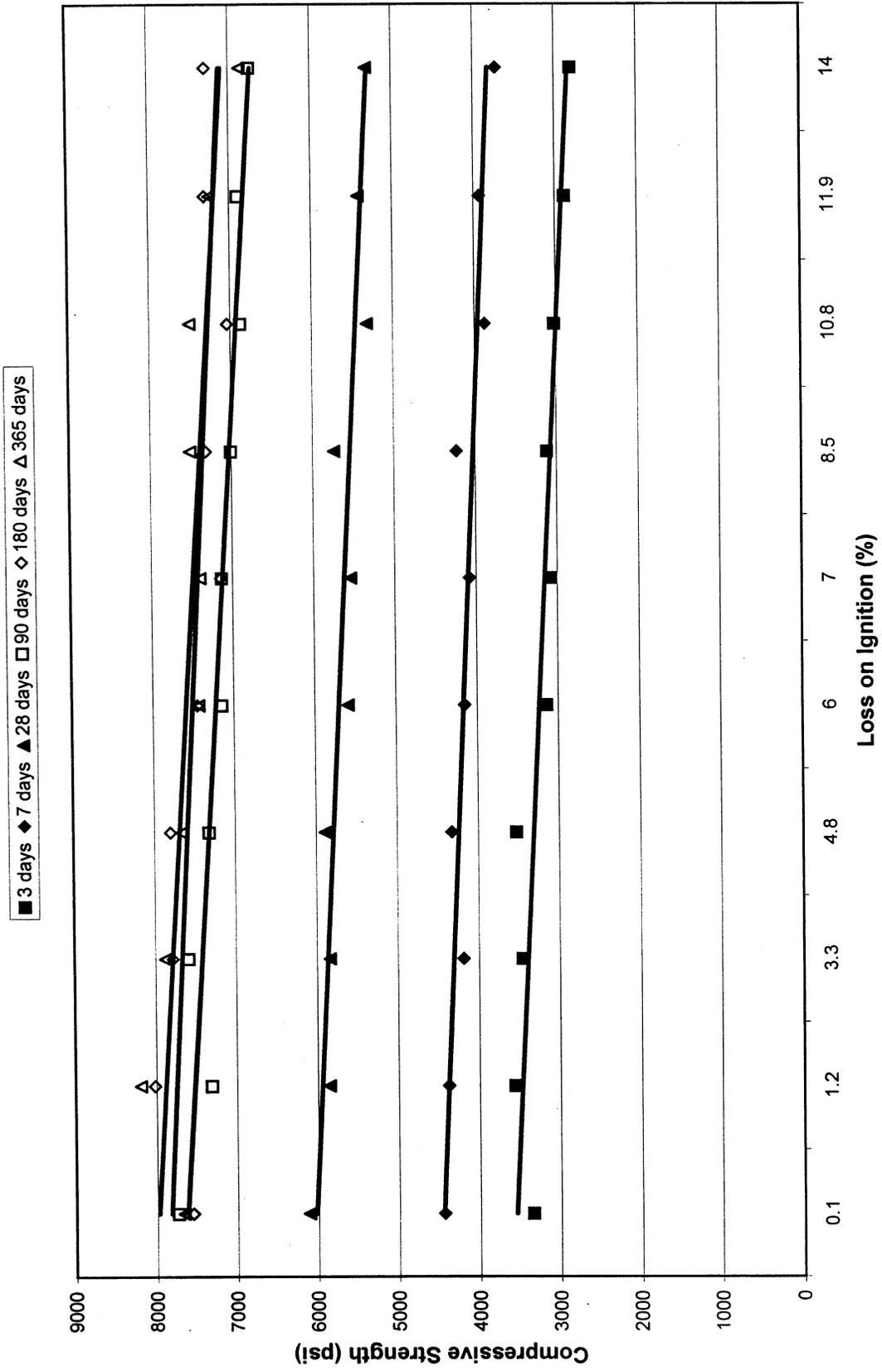
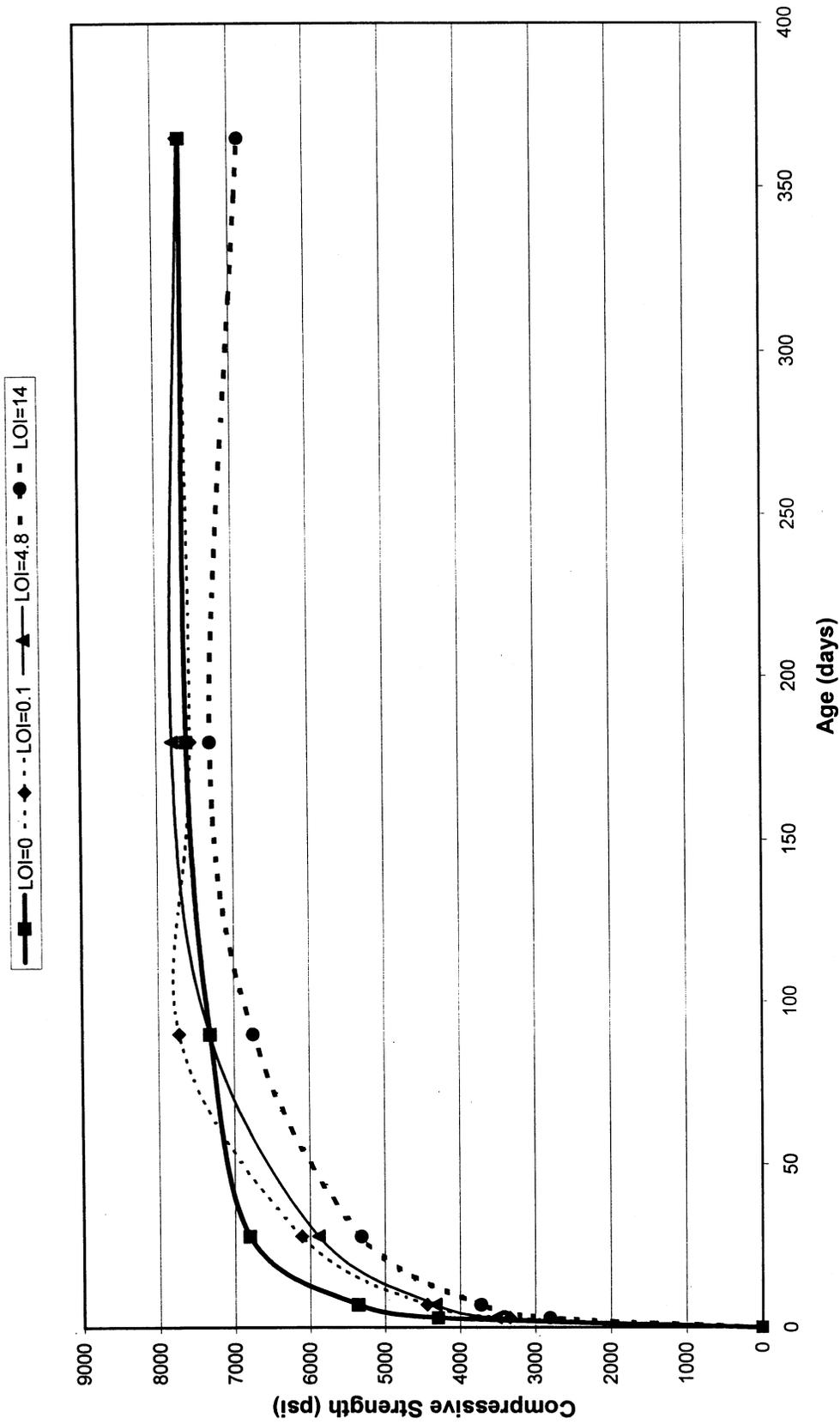


Figure 21: Compressive Strength For Control and Pure Blends
 (Series 2-No AEA for Control and 0.1)



Compressive Strength of Mortar Cubes:

Mortar cubes were prepared in accordance to procedures set in ASTM C019, except for the batch sizes. This variation had to be adopted as 42 mortar cubes had to be prepared per LOI sample. Compressive strength measurements (in lime and sulfate) were performed at the following test ages: 3,7, 28, 91,182, 364 and 735 days. The results of the tests are depicted in Figures 22 through 26 for lime and sulfate exposure. From Figure 22, it can be seen that the control mix had maintained a higher strength than all fly ash mixes except for 1.2% LOI mix up to 28 days. Figure 23 reveals similar trends, to those observed earlier for concrete mixes, for strength variation as a function of LOI. In other words, there is a general trend of reduction in strength on increasing the LOI of ashes.

From Figure 24, it can be seen that the control mix experienced a strength drop at 365 days of exposure to the sulfate environment. At 735 days of exposure all mixes incorporating FA3 in pure form or in blended form to achieve their set LOI has shown drop in strength. These mixes include the blended ash of an LOI of 3.3%. Though the drop is not similar to that experienced by the 14% LOI mix, it is however, worth noticing.

Figure 25 shows the strength variation as a function of LOI for mortar cubes exposed to sulfate environment. Two things can be noticed from this set of data; first, as the LOI increases, there is a general decrease in strength. Second, for long exposure times, the rate of deterioration in sulfate environment increased with increasing LOI content of the fly ash mixes. This can be seen from the overlap of the strength data generated at 365 and 735 days. Mortar mixes incorporating unblended ashes (LOI of 0.1 and 4.8%) did not show signs of deterioration up to 735 days of exposure to sodium sulfate environment as shown in Figure 26.

In conclusion, the general trends observed in this investigation for compressive strength variation with age for mortar or concrete mixes incorporating pure and blended fly ashes, are indicative of downward trend in strength as a function of increasing the LOI of the used ash. This was true for all environments. In addition, unblended fly ash mortar mixes with LOI values within the current FDOT specifications showed better performance in the sulfate environment than all blended fly ash mortar mixes, including blends that generated LOI values within the current FDOT specifications.

Figure 22: Compressive Strength for Mortar as a Function of Time (Lime)

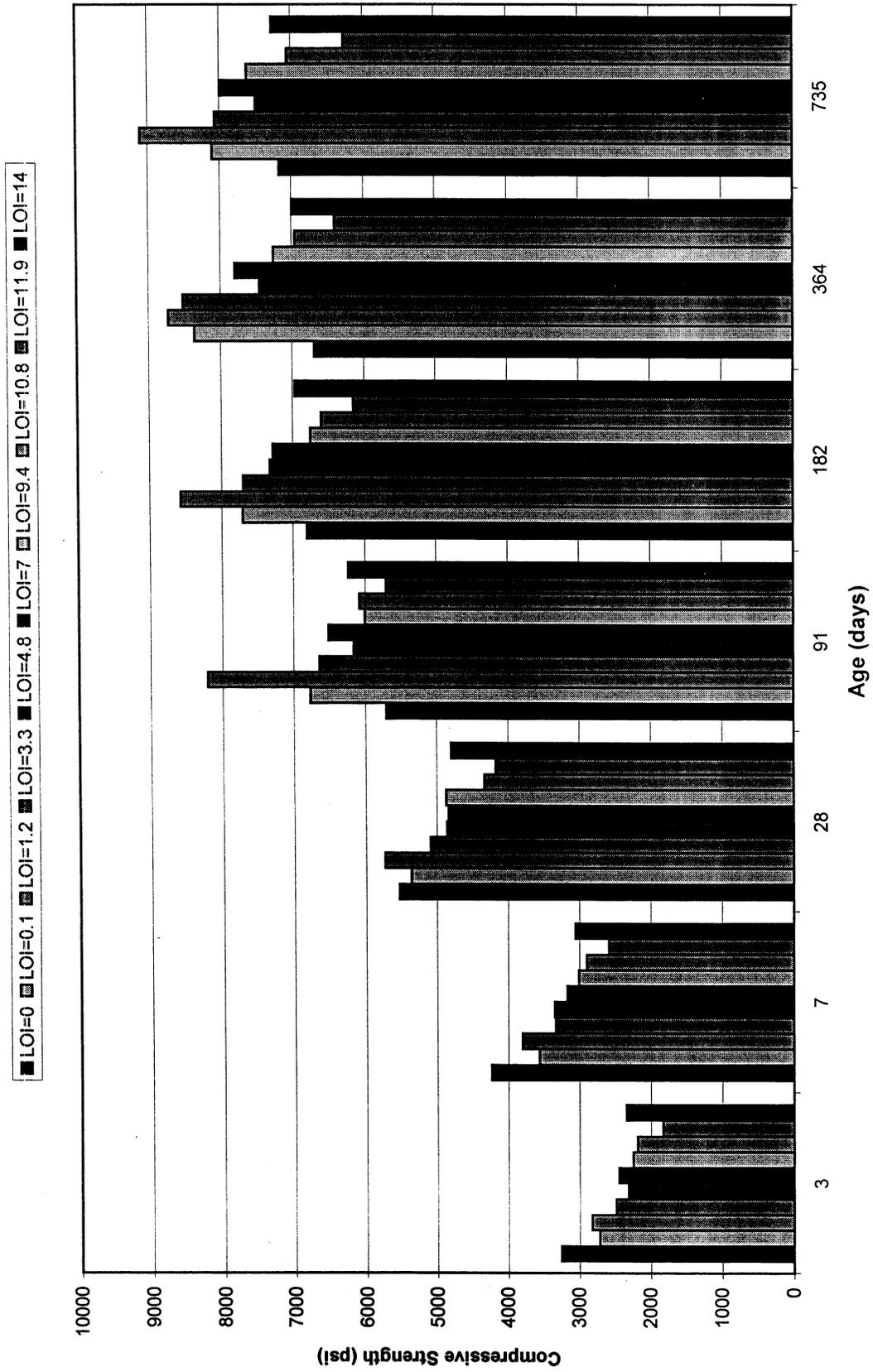


Figure 23: Variation of Compressive Strength as a Function of LOI for Mortar (Lime)

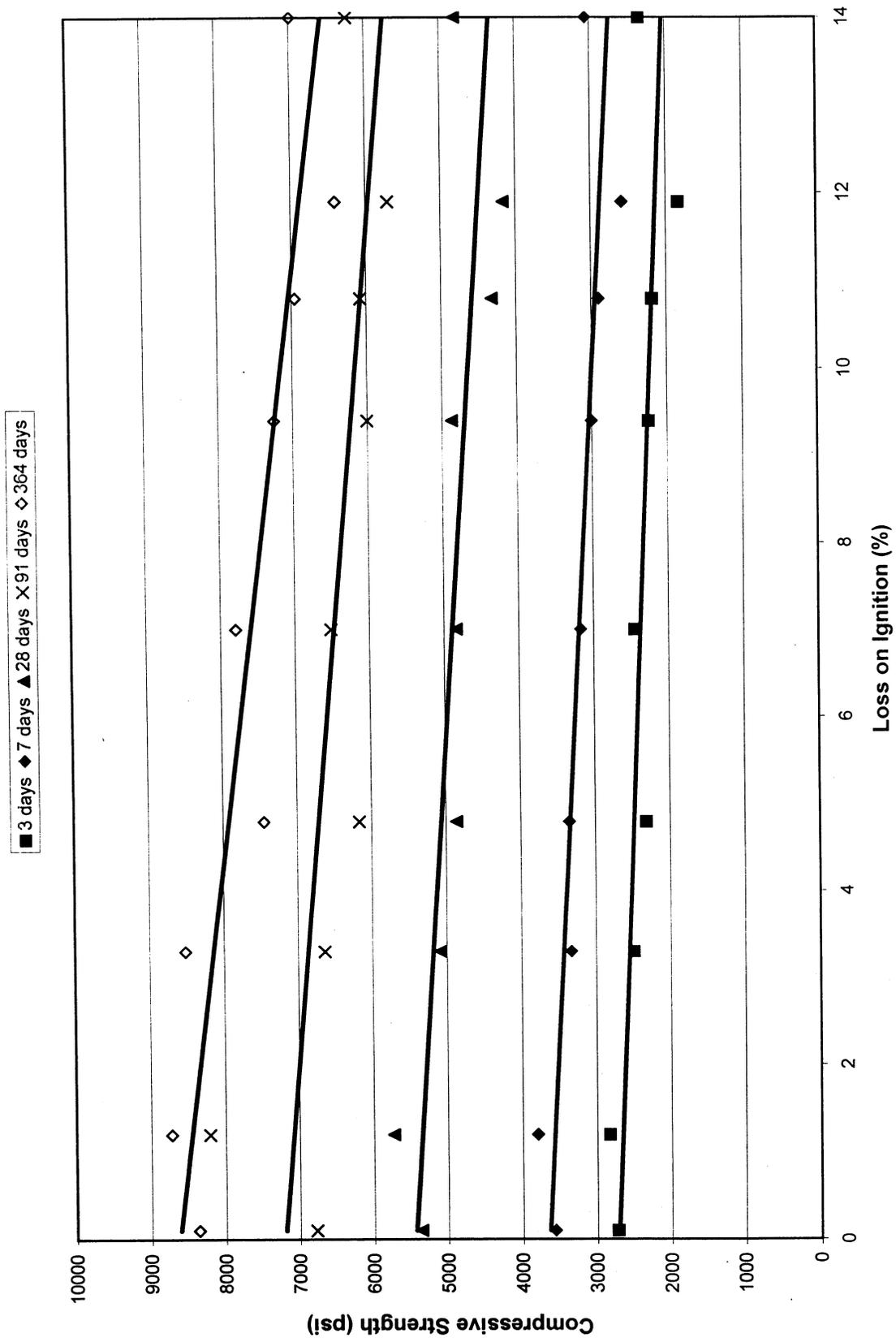


Figure 24: Compressive Strength Gain for Mortar (Sulfate)

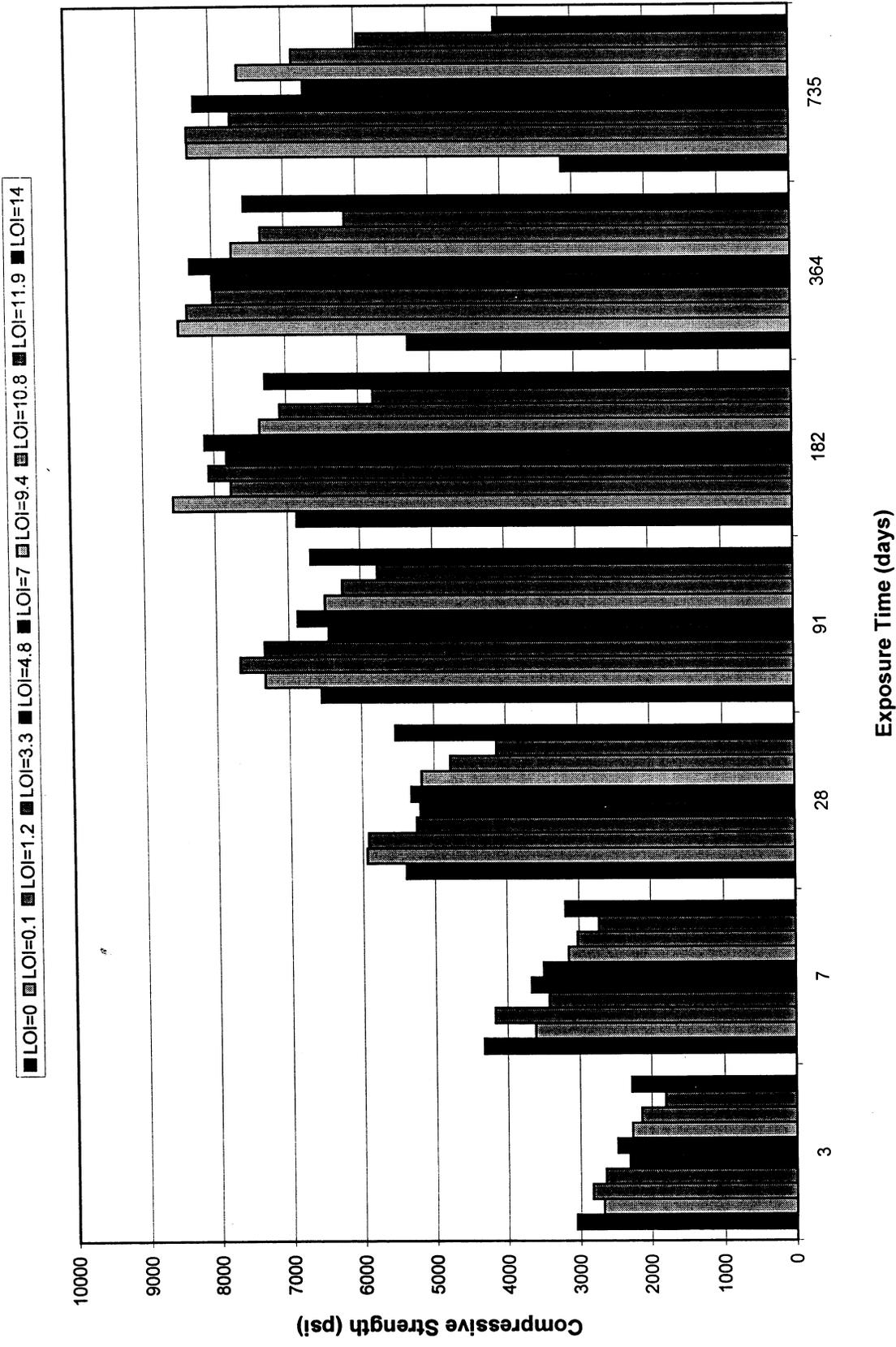
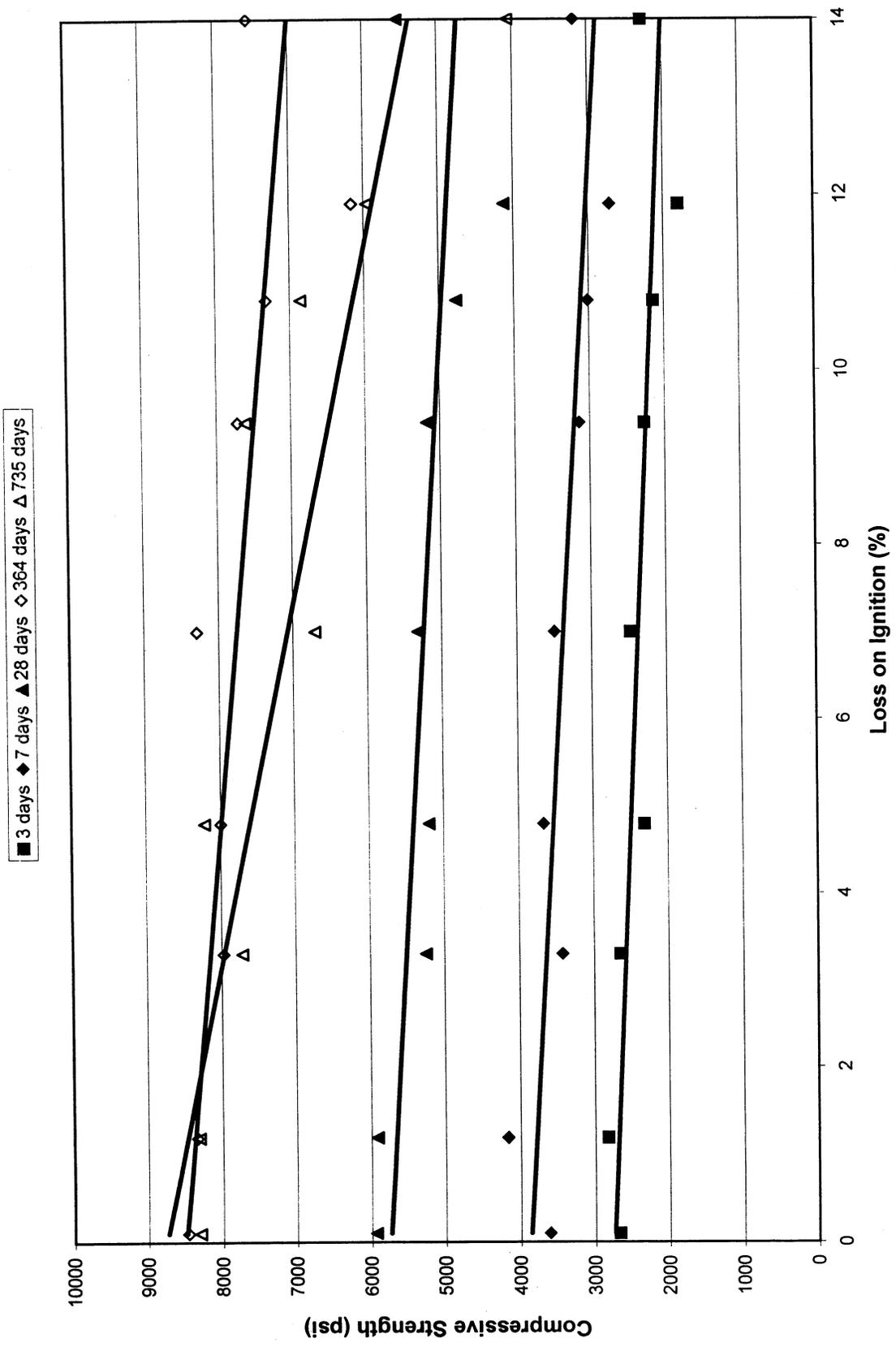
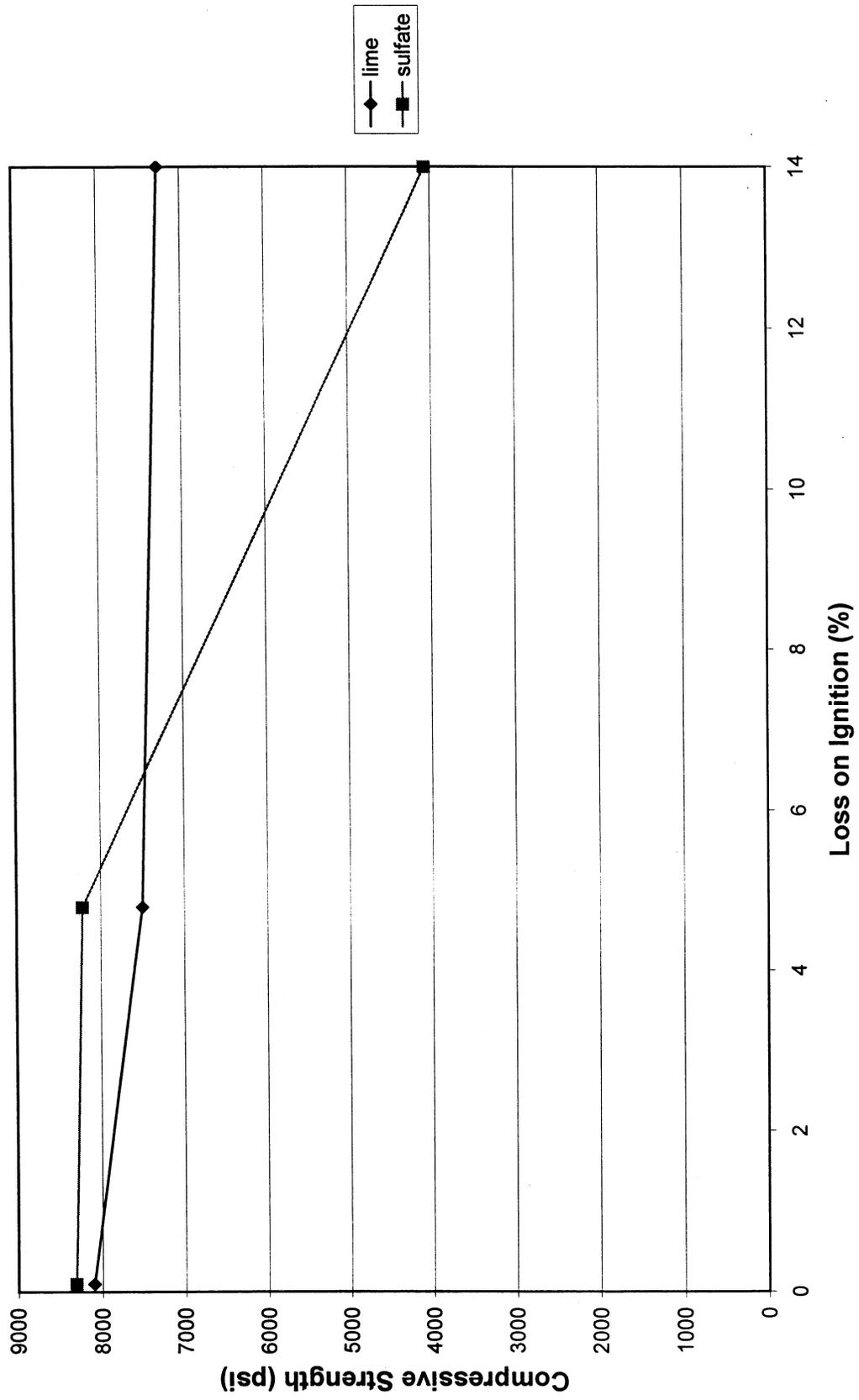


Figure 25: Compressive Strength for Mortar as a Function of LOI (Sulfate)



**Figure 26: Compressive strength of Mortar Cubes at the Age of 735 Days
(for Mixes containing LOI 0.1, 4.8, 14)**



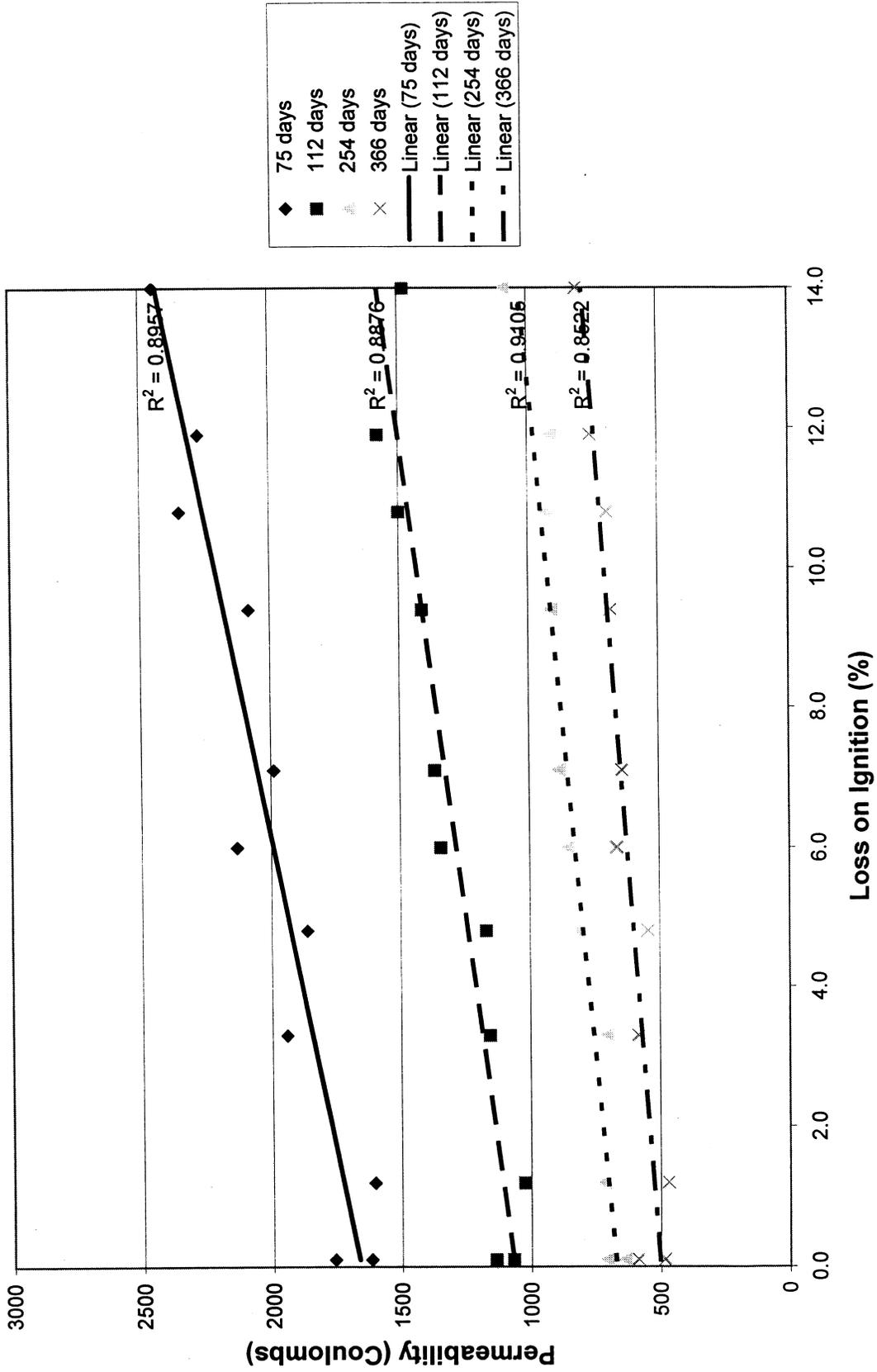
Durability Assessment:

Rapid Chloride Permeability Test:

Concrete cylinders used for the RCP test were prepared with constant air admixture dosage. The mixes had the following LOI: 0.0 (control), 0.1, 1.2, 3.3, 4.8, 6.0, 7.1, 9.4, 10.8, 11.9 and 14. As the air content for mixes 0.0 and 0.1, showed higher air content than all other mixes, two additional mixes for the latter were designed and prepared to contain the same air content measured in the mixes of LOI of 1.2 through LOI of 14. The exact proportions for those mixes are given in Table 4. Concrete cylinders were exposed to sodium sulfate environment until the test age. The results for those tests are depicted in Figure 27, where chloride permeability expressed in Coulombs is plotted as a function of LOI. The results show good correlation coefficients for all ages indicating that permeability for the tested ashes was found to be a function of LOI at all ages. Increasing the LOI in fly ashes implies that the carbon content and contaminants are higher. The measured permeability increased by approximately 50% for FA3 mix as compared to the control mix. This increase seems to be constant for all ages tested.

It is fairly established that carbon is a nonreactive component in fly ash and, as such, would not contribute to the pozzolanic reactivity of the fly ash. Thus increasing the carbon content would tend to reduce the pore size refinement effect attributed to the pozzolanic activity. It is recognized that LOI is not the only material property in fly ashes that will influence permeability, but from the previous results presented on the mineralogical, morphological and particle size distribution are in agreement with LOI values. That is, fly ashes with higher carbon content had lower amorphous content, poorly spherical particles, higher contamination and coarser particle size distribution.

Figure 27: Variation of Concrete Permeability with LOI



Expansion Measurements:

Another test used in assessing the effect of blending ashes on concrete durability was to monitor length changes as a function of exposure time to a sodium sulfate environment. The specimens used for this test were concrete and mortar prisms. The concrete and mortar prisms were placed in a 5 % sodium sulfate solution. The length change profiles for the concrete and mortar mixes are depicted in Figure 28 and 29 respectively. With the exception of the control mix, none of the concrete mixes showed any significant deterioration up to 735 days. On the other hand, for the mortar bars, signs of deterioration were observed at earlier age. This is to be expected due to the higher aspect ratio of the mortar bars.

In the analyses for mortar expansion data, a 0.05 % failure criteria will be adopted [1]. Measured expansion of 0.05%, if taken to represent elastic strains, would indicate an average stress of 1900 psi [13 Mpa]. Mortar tensile strength is much lower than this value; therefore those strains can not be considered elastic in nature and would be indicative of cracking. In Figure 29, the trends are shown for all the mortar mixes. The control mix has reached the 0.05% limit at 180 days. It was followed by 11.9%, 10.8% and 3.3% LOI at 200, 250 and 300 days respectively. These were followed by mixes with LOI of 14 and 7% LOI which reached the 0.05% limit at 550 and 700 days respectively. It is interesting to notice from these results that almost all of the mortar mixes incorporating blends of FA3 have reached the 0.05 % criteria. In addition, none of the pure ash mortar mixes (FA1 and FA2), with LOI below the Florida Department of Transportation Specifications, has experienced the 0.05% failure criteria at an age of 735 days. However, one of the blended fly ash mortar mixes with an LOI of 3.3% has reached deterioration limits within 300 days of exposure to sodium sulfate environment.

In conclusion, it can be seen from the durability tests that, in general, all of the mixes containing FA3 whether in pure form or as part of a blend, did not perform equally to the acceptable fly ashes FA1 and FA2 in this investigation. The blended 1.2 % LOI fly ash mortar had close durability performance to the unblended 0.1% LOI fly ash mortar. It is to be remembered that this ash was generated through blending FA1 and FA2, with both passing the current FDOT specifications on Class F fly ashes. However, the 3.3%

LOI blended fly ash mortar mix did not perform as well, though its LOI is well within FDOT specifications. It is believed that the reason for the rapid deterioration of the 3.3% mix is due to the quality of the original ashes used in generating it; namely, FA3. Considering the particle size distribution data for the 3.3% LOI fly ash, it is clear that this ash has retained, at least partially, the characteristics of the particle size distribution of FA3.

Figure 28: Expansion Profiles for Mortar Bars (Sodium Sulfate)

□ LOI=0 ■ LOI=0.1 ◆ LOI=1.2 ▲ LOI=3.3 X LOI=4.8 * LOI=7 + LOI=9.4 - LOI=10.8 ◇ LOI=11.9 Δ LOI=14

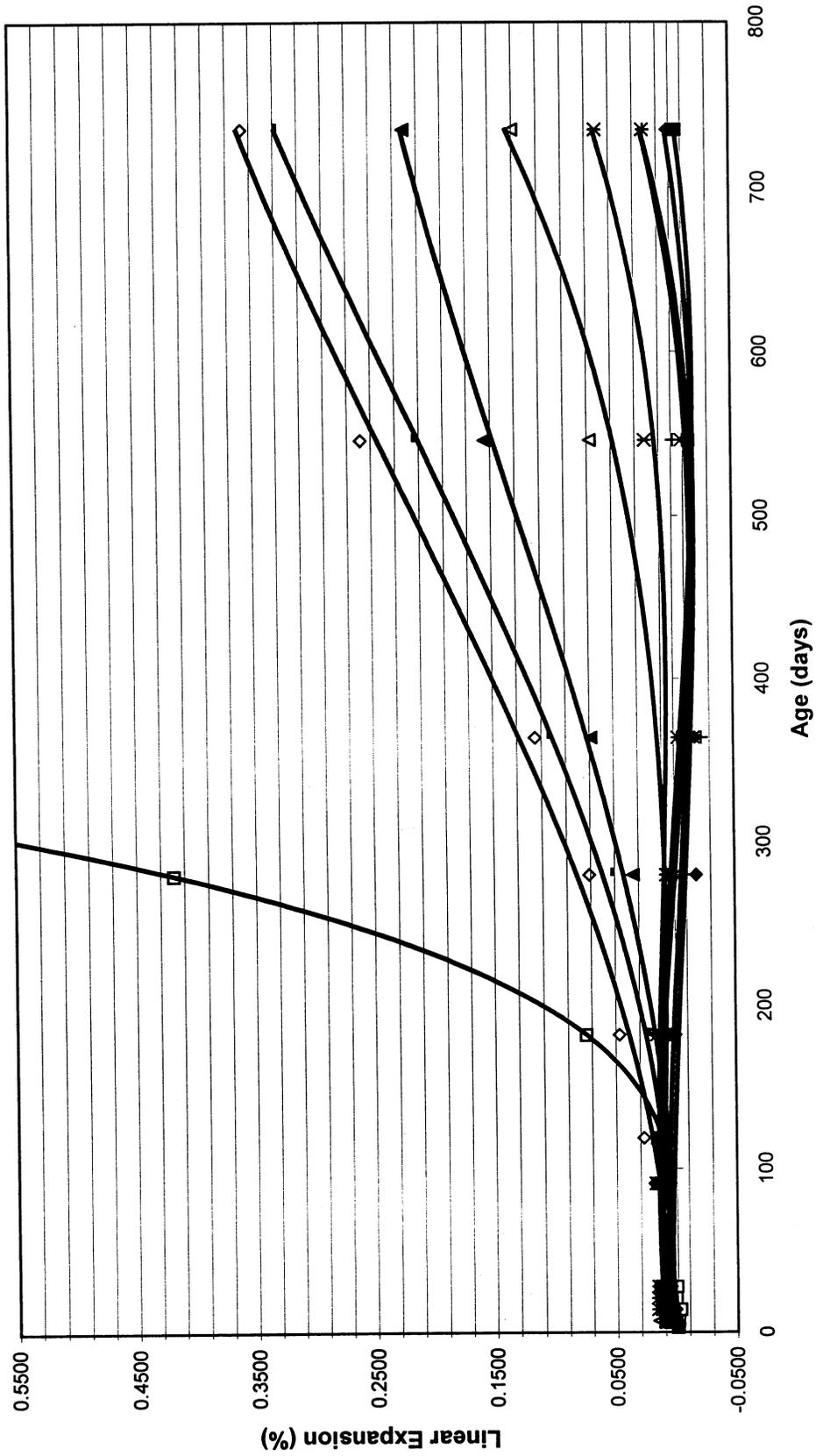
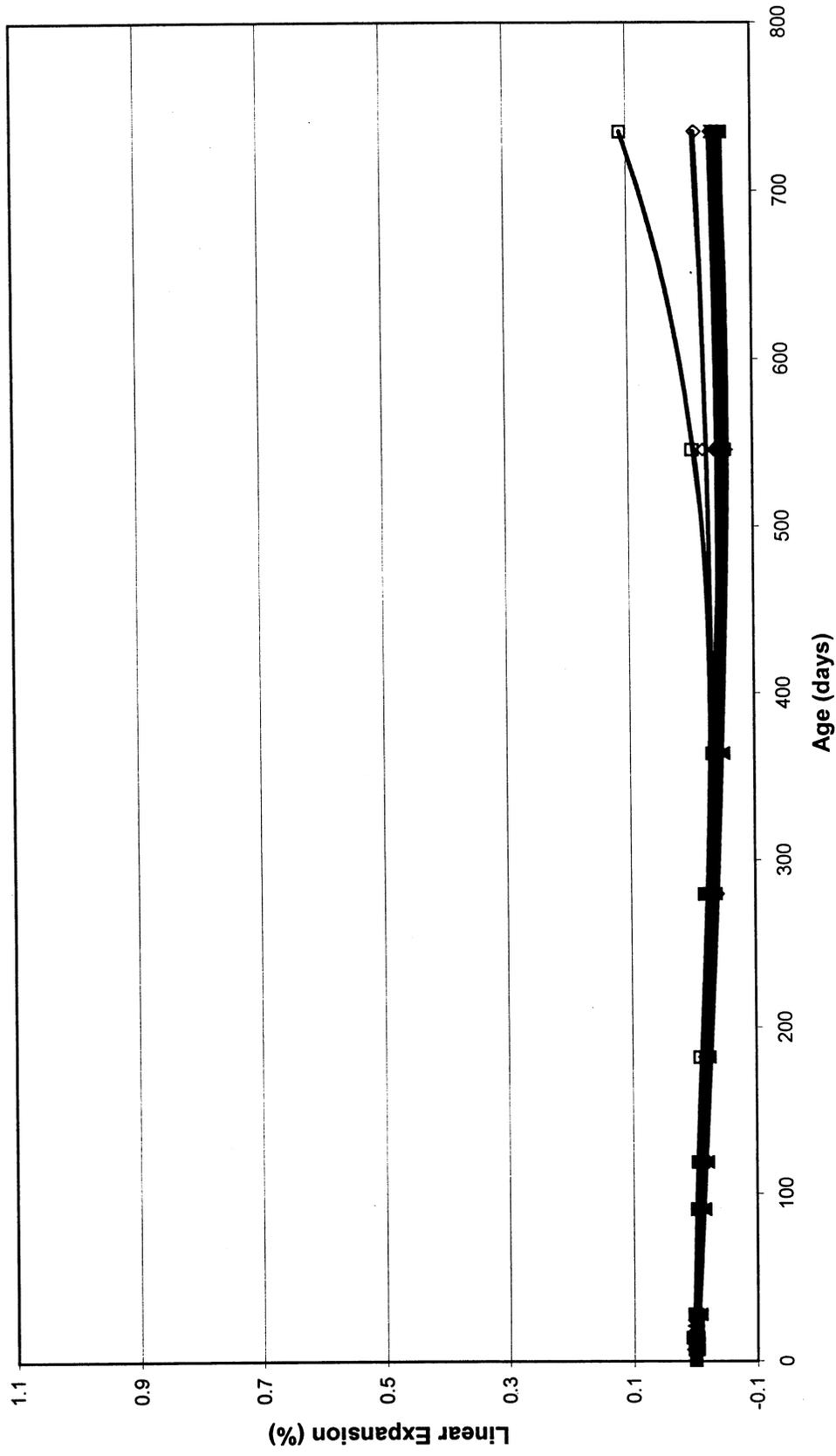


Figure 29: Expansion Profiles for Concrete Prisms (Sulfate Exposure)

LOI=0
 LOI=1.2
 LOI=3.3
 LOI=4.8
 LOI=7
 LOI=9.4
 LOI=10.8
 LOI=11.9
 LOI=14



CONCLUSIONS AND RECOMMENDATIONS

Based on the data presented in this study it becomes apparent that the performance of blended Class F fly ash is dependent on the blended ash source. The following is a list of summaries and conclusions:

1. Blended ashes generated from blending ashes, from different source or the same source, and that incorporated a rejected source, had shown long term durability problems as assessed by mortar expansion measurements in sulfate environment.
2. Even if the blended ash had an LOI within the current Florida Department of Transportation specifications for Class F fly ash, the blended ash had significantly lower durability if compared to the unblended passing fly ashes. Both length and strength measurements in sulfate environments on mortar cubes confirm this finding.
3. Deterioration in blended ashes containing an unapproved source seems to be observed at variable contents of the unapproved source within the blended ashes.
4. Particle size distribution analysis up to 1000 microns together with morphological analysis were found to be excellent tools in assessing the materials properties of the as-received ash and blended ashes.

The following are suggested recommendations based on the findings of this study:

1. Prior to allowing the incorporation of blended Class F fly ashes in concrete construction practices, a study has to be initiated whereby several blended ashes with LOI below 6% have to be carefully investigated for their materials properties and durability. The indicators found in the current study would also be verified.
2. Applicability of the established indicator to current concrete production, guidelines and standards has to be examined.

3. Current FDOT specifications will have to be amended if blending of fly ashes is to be allowed.

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