

J.M. Pitt T. Demirel K.L. Bergeson J.R. Rohde J. Jashimuddin  
January 1987

# Optimization of Soil Stabilization with Class C Fly Ash

Sponsored by the Iowa Department of Transportation, Highway Division,  
and the Iowa Highway Research Board



Iowa Department  
of Transportation

report<sup>final</sup>

Iowa DOT Project HR-260  
ERI Project 1651  
ISU-ERI-Ames-87166

College of  
Engineering  
Iowa State University

The opinions, findings and conclusions expressed in this publication  
are those of the authors and not necessarily those of the  
Highway Division of the Iowa Department of Transportation.

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department of civil engineering  
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## INTRODUCTION AND BACKGROUND

Previous Iowa DOT sponsored research has shown that some Class C fly ashes are cementitious (because calcium is combined as calcium aluminates) while other Class C ashes containing similar amounts of elemental calcium are not (1). Fly ashes from modern power plants in Iowa contain significant amounts of calcium in their glassy phases, regardless of their cementitious properties. The present research was based on these findings and on the hypothesis that: attack of the amorphous phase of high calcium fly ash could be initiated with trace additives, thus making calcium available for formation of useful calcium-silicate cements.

Phase I research was devoted to finding potential additives through a screening process; the likely chemicals were tested with fly ashes representative of the cementitious and non-cementitious ashes available in the state. Ammonium phosphate, a fertilizer, was found to produce 3,600 psi cement with cementitious Neal #4 fly ash; this strength is roughly equivalent to that of portland cement, but at about one-third the cost. Neal #2 fly ash, a slightly cementitious Class C, was found to respond best with ammonium nitrate; through the additive, a near-zero strength material was transformed into a 1,200 psi cement.

The second research phase was directed to optimizing trace additive concentrations, defining the behavior of the resulting cements, evaluating more comprehensively the fly ashes available in

Iowa, and explaining the cement formation mechanisms of the most promising trace additives. X-ray diffraction data demonstrate that both amorphous and crystalline hydrates of chemically enhanced fly ash differ from those of unaltered fly ash hydrates. Calcium-aluminum-silicate hydrates were formed, rather than the expected (and hypothesized) calcium-silicate hydrates. These new reaction products explain the observed strength enhancement.

The final phase concentrated on laboratory application of the chemically-enhanced fly ash cements to road base stabilization. Emphasis was placed on use of marginal aggregates, such as limestone crusher fines and unprocessed blow sand. The nature of the chemically modified fly ash cements led to an evaluation of fine grained soil stabilization where a wide range of materials, defined by plasticity index, could be stabilized. Parameters used for evaluation included strength, compaction requirements, set time, and frost resistance.

### FLY ASH CHARACTERIZATION

A monitoring program was completed in Phase I to establish ranges in chemical and compound composition for fly ash sources with significant production. Table 1 is a list of nine sources where the fly ashes have been categorized both according to ASTM C 618 - 84 and by a scheme more suited to this study. The essence of the ASTM classification is an indirect measure of elemental calcium as an oxide. Categorization for this research was based on a direct measure of elemental calcium oxide combined with an assessment of cementitious qualities, a cementitious fly ash being defined as one producing a seven-day compressive strength in excess of 100 psi. Test conditions involve mixing a paste with a water/fly ash ratio equal to 0.24, molding this paste in 1.5-inch diameter by 3-inch long cylinders, and moist curing the cylinders at 70° F. Using these definitions, fly ashes were categorized as:

Category I: ASTM Class F fly ash having less than 10 percent calcium expressed as an oxide.

Category II: Non-cementitious ASTM Class F or C fly ash, having more than 10 percent calcium expressed as an oxide.

Category III: Cementitious fly ash, having calcium expressed as an oxide in excess of 10 percent.

Elemental oxide compositions used to classify these fly ashes are reported in Appendix A. Oxide composition was determined by x-ray fluorescence, according to the procedures described in reference 2. Seven of the nine plants produce Category III fly ash and are distributed throughout the state. The Category II sources are along the Missouri River.

Table 1. Source Monitoring

| <u>Source</u>  | <u>ASTM Class</u> | <u>Category</u> | <u>No. Samples</u> |
|----------------|-------------------|-----------------|--------------------|
| Neal #2        | C                 | II              | 4                  |
| Neal #3        | F                 | II              | 2                  |
| Neal #4        | C                 | III             | 4                  |
| Council Bluffs | C                 | III             | 4                  |
| Nebraska City  | C                 | II              | 1                  |
| Ottumwa        | C                 | III             | 4                  |
| Lansing        | C                 | III             | 4                  |
| Ames           | C                 | III             | 3                  |
| Louisa         | C                 | III             | 4                  |

## SECONDARY ADDITIVE SCREENING

Fly Ash

To reduce the amount of experimental effort, two fly ashes from the field of nine were selected as representative of materials available in Iowa. Physical and chemical properties of specific samples of Neal #2 (Category II) and Neal #4 (Category III) materials used for screening are in Table 2.

Table 2. Chemical and Physical Properties of  
Screening Fly Ashes

| Chemical Composition (%)  | Neal #2 | Neal #4 |
|---|---------|---------|
| Silicon Oxide ( $\text{SiO}_2$ )  | 54.06   | 34.75   |
| Aluminum Oxide ( $\text{Al}_2\text{O}_3$ )                                      | 19.28   | 15.47   |
| Iron Oxide ( $\text{Fe}_2\text{O}_3$ )  | 7.96    | 6.31    |
| Total ( $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ )        | 81.30   | 56.53   |
| Sulphur trioxide ( $\text{SO}_3$ )  | 2.76    | 3.55    |
| Calcium Oxide ( $\text{CaO}$ )  | 14.29   | 25.76   |
| Magnesium Oxide ( $\text{MgO}$ )  | 3.44    | 5.82    |
| Moisture Content  | 0.13    | 0.02    |
| Loss on Ignition  | 0.58    | 0.16    |
| Available Alkali as $\text{Na}_2\text{O}$                                       | 0.39    | 1.57    |
| <u>Physical Test Results</u>  |         |         |
| Fineness (retained on #325 sieve, %)  | 23.8    | 5.80    |
| Pozzolanic Activity Index with Portland Cement (ratio to control at 28 days, %) | 100     | 110     |
| Pozzolanic Activity Index with Lime at 7 days, psi                              | 1250    | 1400    |
| Water Requirement (ratio to control, %)   | 86      | 88      |
| Soundness (autoclave expansion, %)  | 0.06    | 0.07    |
| Specific Gravity  | 2.38    | 2.66    |

### Amorphous Composition

From glass chemistry (3), it was anticipated that the high calcium content of Category II and III fly ashes would render the amorphous phase more vulnerable to chemical attack, because calcium can distort silica or combined silica-alumina glass networks, thus making them unstable. Compounds resulting from chemical attack must be cementitious to be of value.

The amorphous or glassy phase composition of fly ash can be deduced from quantitative knowledge of the compound and its elemental composition. X-ray diffraction can be used to identify and quantify compounds. Table 3 summarizes this work for the fly ashes from the monitoring program. These data were determined with methods described in references 2 and 4. The glass in Category II fly ash is about 15 percent calcium oxide. About one-third of the Category III glass is calcium oxide. The resulting compositions of the amorphous phase are shown in Table 4. A parallel, therefore, is evident between amorphous calcium oxide and Categories II and III fly ashes.

### Chemical Additives

Chemical additives were selected on the basis of anticipated reactions, the objective being either to initiate chemical attack and breakdown of the glassy phase or to seed formation of cementitious compounds. In the 1960s, Davidson et. al. (5) investigated the use of trace compounds for secondary additives to

enhance reactions between lime and Class F fly ash. Because small amounts of alkaline compounds such as sodium and potassium hydroxide were found to be effective, they were considered in this research. Flouride compounds (because of their ability to attack glass), phosphates and nitrates (because of their ability to substitute in distorted amorphous structures), and magnesium and calcium oxides (because of their classic pozzolanic reactions) were considered as candidate chemicals. Three additive concentrations (0.1, 1.0, and 3.0 percent by weight of fly ash) were selected to provide evaluation over a wide range. A list of compounds used in screening is included in Table 5 through 8.

Table 3. Compound Composition of Fly Ashes

| Compound<br>Composition                         | Fly Ashes |         |        |                   |                  |
|---|-----------|---------|--------|-------------------|------------------|
|   | Neal#2    | Neal#3  | Neal#4 | Council<br>Bluffs | Nebraska<br>City |
| CaO   | 4.8       | 2.3     | 0.8    | 1.4               | 0.1              |
| SiO   | 15.6      | 7.0     | 8.0    | 5.3               | 7.1              |
| C <sub>3</sub> A                                | 0.0       | 0.0     | 4.9    | 6.0               | 0.5              |
| C <sub>4</sub> A <sub>3</sub> S                 | 0.3       | 0.1     | 1.1    | 0.2               | 0.0              |
| C <sub>3</sub> S                                | 0.9       | 0.3     | 1.3    | 0.9               | 0.1              |
| Al <sub>6</sub> Si <sub>2</sub> O <sub>13</sub> | 1.2       | 0.0     | 2.3    | 0.0               | 2.9              |
| MgO   | 0.6       | 1.0     | 3.2    | 2.0               | 0.4              |
| Fe <sub>3</sub> O <sub>4</sub>                  | 7.6       | 0.7     | 0.0    | 0.2               | 0.0              |
| Glass   | 69.0      | 88.6    | 78.4   | 84.0              | 88.9             |
|   | Ottumwa   | Lansing | Ames   |                   |                  |
| CaO   | 0.6       | 2.1     | 1.7    |                   |                  |
| SiO   | 8.6       | 10.1    | 12.4   |                   |                  |
| C <sub>3</sub> A                                | 6.9       | 5.2     | 2.9    |                   |                  |
| C <sub>4</sub> A <sub>3</sub> S                 | 0.4       | 2.3     | 1.1    |                   |                  |
| C <sub>3</sub> S                                | 1.2       | 1.7     | 1.4    |                   |                  |
| Al <sub>6</sub> Si <sub>2</sub> O <sub>13</sub> | 2.2       | 0.9     | 0.0    |                   |                  |
| MgO   | 1.1       | 2.8     | 2.9    |                   |                  |
| Fe <sub>3</sub> O <sub>4</sub>                  | 0.0       | 1.0     | 0.0    |                   |                  |
| Glass   | 79.0      | 73.9    | 47.6   |                   |                  |



Table 4. Amorphous Composition

| Glass*<br>Composition          | Fly Ashes |         |        |                   |                  |
|--------------------------------|-----------|---------|--------|-------------------|------------------|
|                                | Neal#2    | Neal#3  | Neal#4 | Council<br>Bluffs | Nebraska<br>City |
| CaO                            | 13.8      | 12.6    | 28.1   | 30.9              | 33.6             |
| SiO <sub>2</sub>               | 47.2      | 49.1    | 29.9   | 29.1              | 29.5             |
| Al <sub>2</sub> O <sub>3</sub> | 22.8      | 20.4    | 20.0   | 21.4              | 21.3             |
| Fe <sub>2</sub> O <sub>3</sub> | 1.4       | 8.4     | 7.4    | 5.9               | 6.2              |
| MgO                            | 1.7       | 2.4     | 5.7    | 4.5               | 6.1              |
| K,Na,Ti,SO <sub>3</sub>        | 13.1      | 7.1     | 8.9    | 8.2               | 3.3              |
|                                | Ottumwa   | Lansing | Ames   |                   |                  |
| CaO                            | 24.8      | 32.8    | 24.7   |                   |                  |
| SiO <sub>2</sub>               | 32.7      | 26.6    | 33.6   |                   |                  |
| Al <sub>2</sub> O <sub>3</sub> | 20.0      | 21.6    | 21.4   |                   |                  |
| Fe <sub>2</sub> O <sub>3</sub> | 6.7       | 7.1     | 7.2    |                   |                  |
| MgO                            | 5.1       | 4.6     | 4.0    |                   |                  |
| K,Na,Ti,SO <sub>3</sub>        | 10.7      | 7.3     | 9.1    |                   |                  |

\* Glass composition normalized to 100%

#### Test Procedures

Strength -- As a preliminary screening measure, unconfined compressive strength was selected as one indicator of additive effectiveness. Test specimens in this study were prepared using distilled water at a water/fly ash ratio of 0.24. At this ratio, the paste for both Neal #2 and Neal #4 fly ash was homogeneous and plastic. Fly ash paste mixes were prepared in compliance with ASTM method C 109 and all chemicals with exception of kiln dust and portland cement were dissolved or dispersed into a stable solution/dispersion with the mix water. Kiln dust and cement were dry blended with fly ash prior to mixing with water.

On completion of mixing, cylindrical unconfined compression samples (1.5 inches diameter by 3.0 inches long) were cast in split mold assemblies, rodded, and clamped between lucite plates. Six replicas were cast for each test variable. When molded, specimens were cured in a humid room at 70° F, removed from the molds at 24 hours and returned to the humidity room until testing. Compression testing was conducted at a controlled deformation rate of 0.05 inches per minute.

Set Time -- A Soiltest pocket penetrometer (Model CL-700) was used as a rapid method of measuring rate of early strength gain and set properties. The procedure involved casting fly ash paste in four inch diameter, three-fourths inch deep pans and pushing the penetrometer every few minutes until its capacity (60 psi) was reached. Figure 1 is typical of set time tests where a slow initial strength gain rate increases to a significantly faster rate. The time corresponding to the intersection of two straight lines fit to the strength rate data is defined as "initial set". The time required to reach 60 psi is termed "final set".

#### Screening Results

Neal #2 -- Results for strength and set time with sixteen additives are reported in Tables 5 and 6. Although eight of the additives served to enhance strength to some degree, a criteria of at least fifty percent improvement was imposed on an additive before further evaluation. The response for those additives judged most

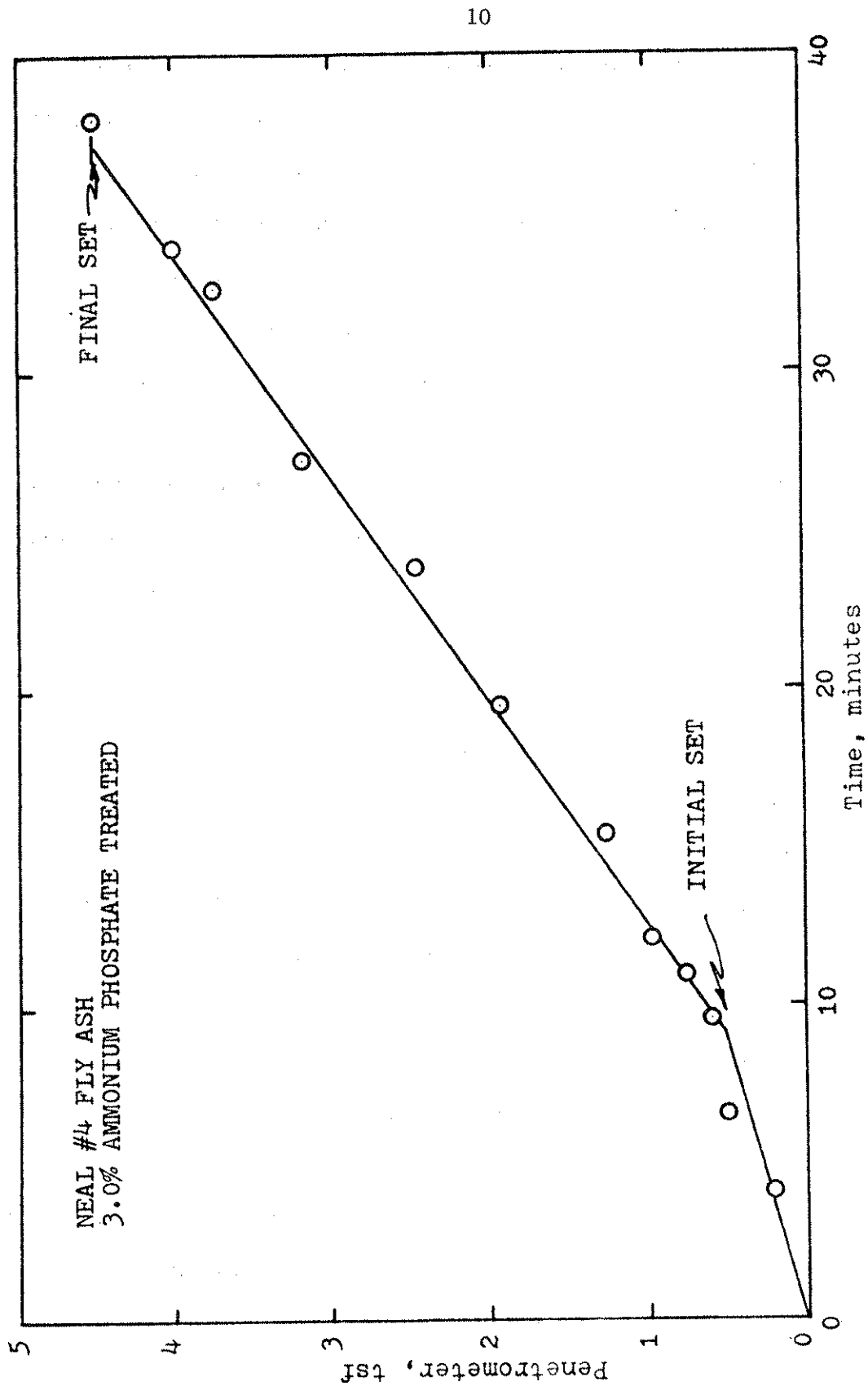


Figure 1. Setting Times of Ammonium Phosphate Treated Neal #4 Fly Ash

effective is as follows:

| Additive                               | Concentration<br>(percent) | Strength Ratio<br>(treated/control) |
|--|----------------------------|-------------------------------------|
| Ammonium Nitrate                       | 3.0                        | 2.34                                |
| Sodium Phosphate                       | 1.0                        | 1.93                                |
| Calcium Flouride &<br>Ammonium Nitrate | 3.0                        | 1.81                                |
| Magnesium Oxide &<br>Calcium Oxide     | 3.0                        | 1.62                                |
| Portland Cement                        | 1.0                        | 1.53                                |

Although set time was not considered a significant factor to application of Category II fly ashes, data in Tables 5 and 6 indicate a potential for chemical additive control. Some additives, such as phosphoric acid, can triple time to initial set while others, such as ammonium phosphate, reduce set time by a factor of two.

Neal #4 -- Results of the screening tests for Neal #4 fly ashes are in Table 7 and 8. Because of the flash set character of Category III fly ashes, set time was coupled with the 50 percent strength criteria for further additive consideration. Those additives capable of enhancing strength and at least tripling the time for final set are as follows:

| Additive                           | Concentration<br>(percent) | Strength Ratio<br>(treated/control) |
|------------------------------------|----------------------------|-------------------------------------|
| Ammonium Phosphate                 | 3.0                        | 2.48                                |
| Portland Cement                    | 3.0                        | 2.00                                |
| Magnesium Oxide &<br>Calcium oxide | 3.0                        | 1.69                                |
| Kiln Dust                          | 1.0                        | 1.52                                |
| Aluminum Sulphate                  | 3.0                        | 1.51                                |

Table 5. Screening Results for Neal #2 Fly Ash

| Additive                            | Concentration<br>% | Strength(psi) |             |     | Set(min) |       |
|-------------------------------------|--------------------|---------------|-------------|-----|----------|-------|
|                                     |                    | 1             | (Days)<br>7 | 28  | Init     | Final |
| Control                             | 0.0                | 75            | 320         | 580 | 60       | 125   |
| Zinc Oxide                          | 0.1                | 75            | 495         | 580 | 60       | 108   |
|                                     | 1.0                | 35            | 50          | 240 | 125      | 140   |
|                                     | 3.0                | 30            | 75          | 95  | 35       | 250   |
| Aluminum<br>sulfate                 | 0.1                | 105           | 510         | 740 | 35       | 85    |
|                                     | 1.0                | 80            | 190         | 425 | 100      | 145   |
|                                     | 3.0                | 40            | 230         | 330 | 90       | 120   |
| Sodium<br>flouride                  | 0.1                | 70            | 260         | 405 | 150      | 200   |
|                                     | 1.0                | 50            | 85          | 150 | 45       | 40    |
|                                     | 3.0                | 15            | 30          | 35  | 350      | 545   |
| Ammonium<br>phosphate<br>(dibasic)  | 0.1                | 75            | 410         | 635 | 50       | 125   |
|                                     | 1.0                | 105           | 545         | 585 | 50       | 125   |
|                                     | 3.0                | 20            | 80          | 85  | 95       | 115   |
| Magnesium<br>oxide                  | 0.1                | 85            | 420         | 665 | 35       | 90    |
|                                     | 1.0                | 25            | 425         | 635 | 30       | 155   |
|                                     | 3.0                | 10            | 445         | 790 | 25       | 185   |
| Magnesium<br>and calcium<br>oxide * | 0.1                | 75            | 360         | 535 | 45       | 115   |
|                                     | 1.0                | 80            | 320         | 410 | 70       | 150   |
|                                     | 3.0                | 30            | 350         | 940 | 60       | 185   |
| Kiln dust                           | 0.1                | 90            | 155         | 670 | 55       | 110   |
|                                     | 1.0                | 50            | 395         | 690 | 65       | 115   |
|                                     | 3.0                | 270           | 1035        | 750 | 30       | 110   |
| Ammonium<br>flouride                | 0.1                | 0             | 80          | 120 | 85       | 170   |
|                                     | 1.0                | 55            | 90          | 185 | 15       | 40    |
|                                     | ** 3.0             | ---           | ---         | --- | ---      | ---   |

\* MgO/CaO=0.57 by weight

\*\* Test discontinued

Table 6. Screening Results for Neal #2 Fly Ash

| Additive                                       | Concentration<br>% | Strength(psi) |             |      | Set(min) |       |
|--|--------------------|---------------|-------------|------|----------|-------|
|  |                    | 1             | (Days)<br>7 | 28   | Init     | Final |
| Control  | 0.0                | 75            | 320         | 580  | 60       | 125   |
| Aluminum<br>ammonium<br>sulphate               | 0.1                | 30            | 90          | 285  | 35       | 180   |
|  | 1.0                | 40            | 125         | 245  | 40       | 90    |
|  | 3.0                | 100           | 230         | 605  | 60       | 125   |
| Sodium<br>hydroxide                            | 0.1                | 30            | 165         | 220  | 45       | 150   |
|  | 1.0                | 210           | 450         | 550  | 160      | 415   |
|  | 3.0                | 30            | 200         | 330  | 150      | 330   |
| Ammonium<br>nitrate                            | 0.1                | 15            | 120         | 345  | 25       | 125   |
|  | 1.0                | 30            | 300         | 450  | 30       | 70    |
|  | 3.0                | 40            | 740         | 1360 | 30       | 55    |
| Phosphoric<br>acid                             | 0.1                | 70            | 210         | 665  | 60       | 125   |
|  | 1.0                | 85            | 400         | 700  | 165      | 205   |
|  | 3.0                | 20            | 395         | 520  | 20       | 160   |
| Calcium<br>fluoride +<br>Ammonium<br>nitrate * | 0.1                | 25            | 115         | 195  | 25       | 180+  |
|  | 1.0                | 20            | 80          | 225  | 25       | 75    |
|  | 3.0                | 30            | 500         | 1050 | 20       | 60    |
| Ammonium<br>bifluoride                         | 0.1                | 20            | 55          | 200  | 85       | 190   |
|  | 1.0                | 45            | 170         | 280  | 10       | 15    |
|  | 3.0                | 75            | 135         | 355  | 20       | 50    |
| Sodium<br>phosphate                            | 0.1                | 50            | 175         | 320  | 45       | 130   |
|  | 1.0                | 130           | 695         | 1120 | 60       | 100   |
|  | 3.0                | 205           | 275         | 375  | 150      | 180   |
| Cement<br>Type I                               | 0.1                | 90            | 345         | 705  | 35       | 140   |
|  | 1.0                | 85            | 410         | 890  | 30       | 120   |
|  | 3.0                | 110           | 270         | 610  | 30       | 100   |

\* CF/AN = 2.0 by weight

Table 7. Screening Results for Neal #4 Fly Ash

| Additive                            | Concentration<br>% | Strength(psi) |             |      | Set(min) |       |
|-------------------------------------|--------------------|---------------|-------------|------|----------|-------|
|                                     |                    | 1             | (Days)<br>7 | 28   | Init     | Final |
| Control                             | 0.0                | 1040          | 1195        | 1805 | <5       | 5     |
| Zinc Oxide                          | 0.1                | 1340          | 885         | 1214 | 5+       | 5+    |
|                                     | 1.0                | 450           | 1345        | 1235 | 5        | 10    |
|                                     | 3.0                | 760           | 1750        | 2050 | 5        | 10    |
| Aluminum<br>sulfate                 | 0.1                | 1225          | 1110        | 1765 | 5+       | 10    |
|                                     | 1.0                | 1510          | 1330        | 2200 | 5+       | 10    |
|                                     | 3.0                | 1180          | 1800        | 2725 | 15       | 30    |
| Sodium<br>flouride                  | 0.1                | 1055          | 1040        | 800  | <5       | 5     |
|                                     | 1.0                | 410           | 760         | 930  | <5       | <5    |
|                                     | 3.0                | 40            | 330         | 1015 | 5        | 15    |
| Ammonium<br>phosphate<br>(dibasic)  | 0.1                | 1145          | 1030        | 1670 | 10       | 15    |
|                                     | 1.0                | 250           | 480         | 1025 | 70       | 160   |
|                                     | 3.0                | 390           | 2390        | 4475 | 10       | 35    |
| Magnesium<br>oxide                  | 0.1                | 1400          | 910         | 1730 | <5       | 5+    |
|                                     | 1.0                | 825           | 2170        | 2610 | 10       | 15    |
|                                     | 3.0                | 780           | 2465        | 2405 | 10       | 15    |
| Magnesium<br>and calcium<br>oxide * | 0.1                | 1080          | 1210        | 1400 | 10       | 15    |
|                                     | 1.0                | 60            | 1420        | 2600 | 10       | 15    |
|                                     | 3.0                | 1015          | 1090        | 3055 | 10       | 20    |
| Kiln dust                           | 0.1                | 1280          | 900         | 1645 | <5       | 5+    |
|                                     | 1.0                | 960           | 1670        | 2740 | 10       | 15    |
|                                     | 3.0                | 810           | 1465        | 1755 | 10       | 20    |
| Ammonium<br>flouride                | 0.1                | 415           | 490         | 900  | 10       | 15    |
|                                     | 1.0                | 205           | 335         | 840  | 30       | 55    |
|                                     | 3.0                | 45            | 335         | 600  | 15       | 55    |

\* MgO/CaO=0.57 by weight

Table 8. Screening Results for Neal #4 Fly Ash

| Additive   | Concentration<br>% | Strength(psi) |             |      | Set(min) |       |
|------------|--------------------|---------------|-------------|------|----------|-------|
|            |                    | 1             | (Days)<br>7 | 28   | Init     | Final |
| Control    | 0.0                | 1040          | 1195        | 1805 | <5       | 5     |
| Aluminum   | 0.1                | 485           | 680         | 790  | 10       | 15    |
| ammonium   | 1.0                | 400           | 575         | 880  | 25       | 40    |
| sulphate   | 3.0                | 505           | 680         | 1010 | 30       | 80    |
| Sodium     | 0.1                | 605           | 535         | 905  | 10       | 15    |
| hydroxide  | 1.0                | 140           | 245         | 440  | 5        | 15    |
|            | 3.0                | 185           | 475         | 1335 | <5       | 5     |
| Ammonium   | 0.1                | 835           | 790         | 1100 | 15       | 20    |
| nitrate    | 1.0                | 795           | 1050        | 820  | 20       | 25    |
|            | 3.0                | 310           | 1005        | 1400 | 90       | 100   |
| Phosphoric | 0.1                | 265           | 470         | 610  | 10       | 20    |
| acid       | 1.0                | 155           | 270         | 400  | 145      | 185   |
|            | 3.0                | 20            | 735         | 755  | 10       | 45    |
| Calcium    | 0.1                | 930           | 1095        | 1165 | 10       | 15    |
| fluoride + | 1.0                | 710           | 1065        | 690  | 10       | 15    |
| Ammonium   | 3.0                | 915           | 1520        | 1375 | 25       | 30    |
| nitrate *  |                    |               |             |      |          |       |
| Ammonium   | 0.1                | 360           | 620         | 1220 | 10       | 20    |
| bifluoride | 1.0                | 440           | 595         | 815  | 15       | 20    |
|            | 3.0                | 65            | 650         | 1005 | 10       | 35    |
| Sodium     | 0.1                | 645           | 970         | 1050 | 15       | 20    |
| phosphate  | 1.0                | 180           | 220         | 550  | 25       | 45    |
|            | 3.0                | 60            | 160         | 230  | 40       | 80    |
| Cement     | 0.1                | 1790          | 1820        | 2520 | 10       | 15    |
| Type I     | 1.0                | 1670          | 1910        | 2355 | 10       | 15    |
|            | 3.0                | 1680          | 2465        | 3615 | 10       | 15    |

\* CF/AN = 2.0 by weight



Additionally, it was found that set retardation on the order of 100 to 150 minutes was achieved with ammonium nitrate and lower concentrations of ammonium phosphate, but strength was reduced to less than that of the fly ash alone.

## ADDITIVE OPTIMIZATION

Because the screening program was designed only to identify promising additives, further work was necessary to: determine optimum additive concentration, evaluate the influence of reagent versus commercial additive grades, evaluate the influence of water content, and verify the screening results. In addition to Neal #2 and Neal #4 fly ashes, materials from Ottumwa, Lansing, Louisa, and Ames generating stations were also included to expand the data base to other Category III fly ashes. Test procedures are the same as those used in the screening process but additive concentrations were extended to include integer additive levels ranging from 1 to 8 percent by weight of fly ash. Water/fly ash ratios of 0.20, 0.24 and 0.30 were initially used, but this parameter was a variable in later studies. A detailed presentation of these results are presented in Appendix B but can be summarized as follows:

## Neal #4 (Category III fly ash requiring retardation)

- \* Type I portland cement -- no strength enhancement or retardation
- \* Calcium oxide (lime) -- no strength enhancement; slight but ineffective retardation
- \* Calcium-magnesium oxide (dolomitic lime) -- some strength reduction; minor but ineffective retardation
- \* Kiln dust -- maximum 700 psi strength improvement; however, the extremely fast set probably would result in unworkable field mixes.

- \* Sodium phosphate -- insignificant reduction in strength; however, could serve as a retarder.
- \* Reagent grade ammonium nitrate -- reduced strength but retarded set in concentrations in excess of 3 percent.
- \* Fertilizer grade ammonium nitrate -- reduced strength but retarded set in concentrations in excess of 3 percent.
- \* Reagent grade ammonium phosphate - produced a maximum two-fold strength increase, along with an additional hour to final set at 3 percent additive concentration; a four hour delay in set was observed at 2 percent concentration, but this was at the expense of strength.
- \* Fertilizer grade ammonium phosphate - produced the same strength increase as the reagent chemical and provided an additional hour to final set; seven percent additive concentration was required to achieve two-fold strength increase.

Neal #2 (Category II fly ash not requiring retardation)

- \* Type I portland cement -- no strength enhancement.
- \* Calcium oxide (lime) -- no strength enhancement.
- \* Calcium-magnesium oxides (dolomitic lime) -- no strength enhancement.
- \* Sodium phosphate -- increased strength from 200 to 750 psi at a 2 percent concentration.
- \* Reagent grade ammonium nitrate -- increased compressive strength from 200 to 1300 psi at 3 percent concentration; also caused a somewhat faster set a desirable feature with this ash.
- \* Fertilizer grade ammonium nitrate -- increased strength from 200 to 1000 psi at 2 percent concentration; also caused a somewhat faster set, a desirable feature with this ash.
- \* Urea fertilizer (ammonia) -- no strength improvement.
- \* Reagent grade ammonium phosphate -- increased strength from 200 to 600 psi at 1 percent concentration.

- \* Fertilizer grade ammonium phosphate -- increased strength from 200 to 600 psi at 1 percent concentration.

Ottumwa (Category III fly ash which may require retardation)

- \* Fertilizer grade ammonium nitrate -- reduced compressive strength; increased set time.
- \* Fertilizer grade ammonium phosphate -- increased compressive strength from 700 to 1100 psi at 4 percent concentration; 30 minutes final set retarded to two hours set time.

Lansing (Category III fly ash requiring retardation)

- \* Fertilizer grade ammonium nitrate -- Increased set time from 2 minutes to 10 minutes at 1 percent concentration; increased 7-day compressive strength from 2400 to 3600 psi at 5 percent concentration.
- \* Fertilizer grade ammonium phosphate -- increased set time from 4 minutes to 40 minutes at 2 percent concentration; increased 7-day compressive strength from 2200 to 3800 psi at 4 percent concentration.

Louisa (Category III fly ash which may require retardation)

- \* Fertilizer grade ammonium nitrate -- increased set time from 10 minutes to 240 minutes at a 5 percent concentration; increased compressive strength from 1700 psi to 2100 psi at 3 percent concentration.
- \* Fertilizer grade ammonium phosphate -- Increased set time from 10 minutes to 116 minutes at 1 percent concentration; decreased compressive strength significantly.

Ames (Category III fly ash requiring retardation)

- \* Ammonium phosphate -- Increased strength from 650 to 1700 psi at 3 percent concentration; time of set was increased from 32 minutes to 72 minutes at 2 percent concentration.

\* Ammonium nitrate -- Increased strength from 650 to 1900 psi at 5 percent concentration; increased set time from 32 minutes to 44 minutes at 1 percent concentration.

Patterns emerging from this study are that ammonium nitrate works best with Category II fly ash while ammonium phosphate enhances strength and assists in retardation of Category III materials. These experiments also suggest that the more conventional additives (such as portland cement, lime, and dolomitic lime, for the concentrations studied) have little or no influence on the fly ashes with which they were combined. This supports the fundamental hypothesis for this work in that additional calcium should have little or no effect on an amorphous material already rich in calcium. As a chemical, sodium phosphate has potential as a retarder for Category III and a strength enhancer for Category II fly ash, but may not be practical because of cost and availability. The fact that fertilizer grades of ammonium nitrate and ammonium phosphate are effective makes chemical enhancement of these high-calcium fly ashes possible.

## REACTION MECHANISMS

Although physical testing demonstrates ammonium phosphate and ammonium nitrate are capable of reacting in a positive manner with different fly ashes, the ability to predict performance of fly ashes from several sources (each of which may produce a variable product) depends on knowing something about reactions causing the phenomena. To this end, two Category III fly ashes with ammonium phosphate and one Category II fly ash with ammonium phosphate were selected for evaluation with x-ray diffraction.

Diffraction techniques involved use of copper K alpha radiation at 50 Kv and 25 ma with a slow scanning rate of three seconds per 0.03 degree step. This procedure was adapted to enhance crystalline peaks for identification.

Reagent Grade ChemicalsNeal #2

X-ray diffraction results for fly ash hydrated with water alone and with three percent ammonium nitrate are in Figure 2. This ammonium nitrate concentration increased the strength 2.3 times that of untreated specimens and favorably decreased final set time from 125 to 55 minutes.

From the x-ray diffraction trace with water alone, the principal crystalline reaction product is ettringite (E), a calcium-aluminum-sulfate hydrate which forms from tricalcium aluminate and gypsum.

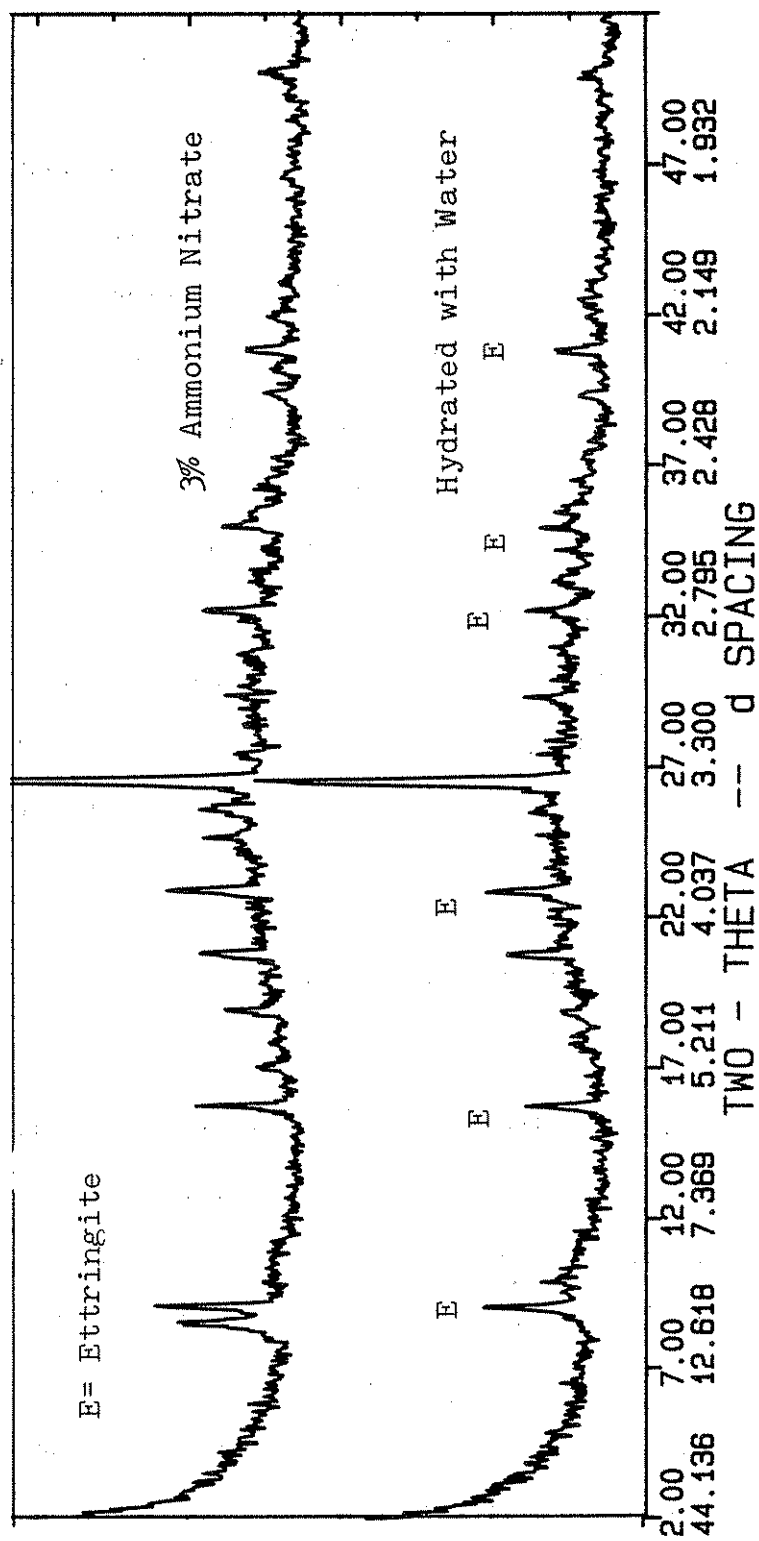


Figure 2. X-ray diffractograms, Neal 2 fly ash, untreated and ammonium nitrate treated

Ettringite is one of several products formed from hydration of portland cement. In terms of crystalline products, ammonium nitrate appears to have produced additional ettringite and a calcium-aluminum-silicate hydrate (indicated by a well defined peak at 10.4 angstroms) not present in the untreated specimen.

Poorly crystalline and amorphous materials can be evaluated from the halo (a gentle hump or rise in background response) on a diffraction trace. Other researchers (6) have shown that poorly crystalline calcium-aluminum hydrates and calcium-aluminum-silicate hydrates show as a halo in the 8 to 12 degree range. Calcium-silicate hydrates are displayed on the diffraction trace from 26 to 36 degrees. The areas beneath the halos for treated and untreated specimens appear to be the same, suggesting that increased strength derived from ammonium nitrate treatment of this fly ash is primarily due to the formation of crystalline products.

#### Neal #4

Diffraction studies were performed at three ammonium phosphate concentrations to facilitate correlation between additive concentration and hydration products. In Figure 3 diffraction traces for 0, 0.1, 1.0, and 3.0 percent ammonium phosphate concentrations are shown. These concentrations correspond to strength responses ranging from a slight decrease to a two-fold increase in strength (Figure 4).

Comparison of x-ray traces for the non-treated test and those



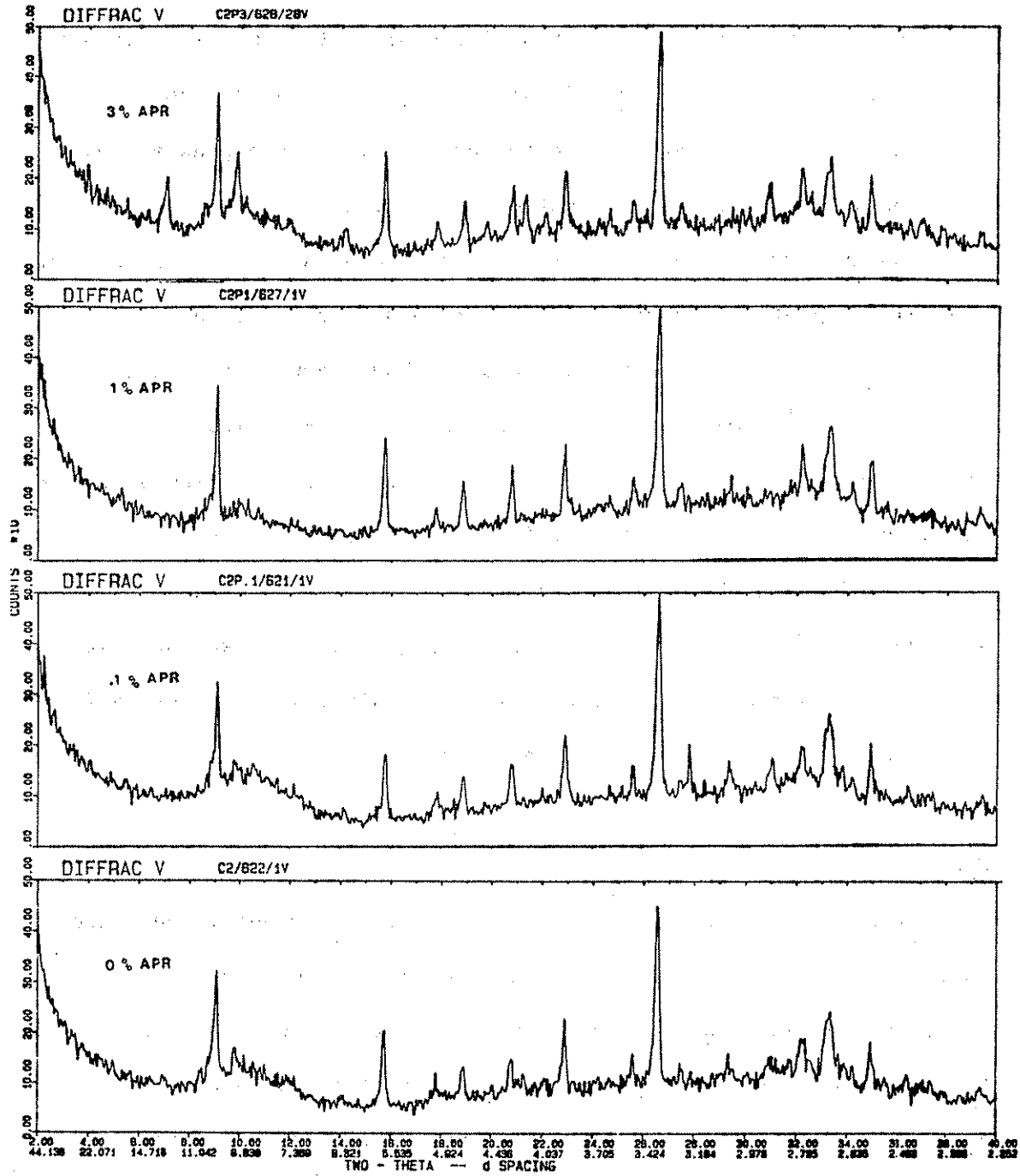


Figure 3. Xray diffractograms of untreated and dibasic ammonium phosphate treated Neal 4 fly ash

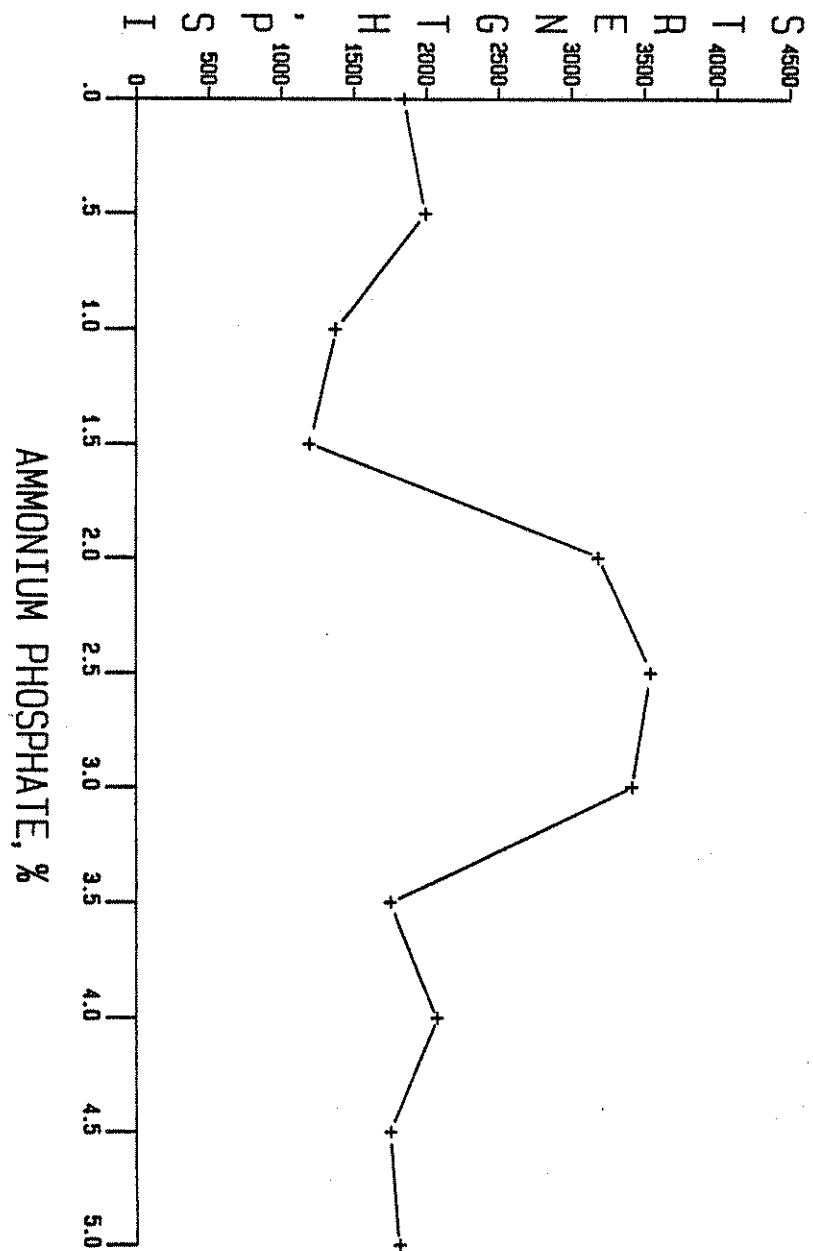


Figure 4. Compressive strength development of ammonium phosphate treated Neal 4 fly ash

for 0.1 and 1.0 percent additive indicates increased ettringite intensity and reduction in amorphous calcium-aluminum hydrates. In contrast, traces for the 3.0 percent ammonium phosphate concentration show formation of monosulfoaluminate (8.92 angstroms) and stratlingite (12.5 angstroms). The 3 percent additive level corresponds to significant strength enhancement; and diffraction tests suggest these compounds are responsible. It is interesting to note that phosphate compounds are not detectable from diffraction traces, a finding which supports the idea that phosphate tetrahedra may substitute for silica tetrahedra in these hydrates.

Set control is thought to be achieved from ammonium temporarily occupying and blocking tricalcium aluminate hydration sites. With time, the ammonium radical disassociates to gaseous ammonia, leaving a hydrogen ion at the hydration site. This mechanism has been postulated for retardation of portland cement (7).

### Fertilizer Grade Chemicals

#### Ammonium Nitrate

Fertilizer grade ammonium nitrate (produced by N-REN Corporation-St. Paul Ammonia Products, South St. Paul, Minnesota) was evaluated and compared with the reagent grade chemical. Strength and set results with Neal #2 fly ash (Appendix B) are essentially the same for both chemical grades. X-ray diffraction traces (Figure 5) for the two qualities of chemical are also the same, suggesting that the

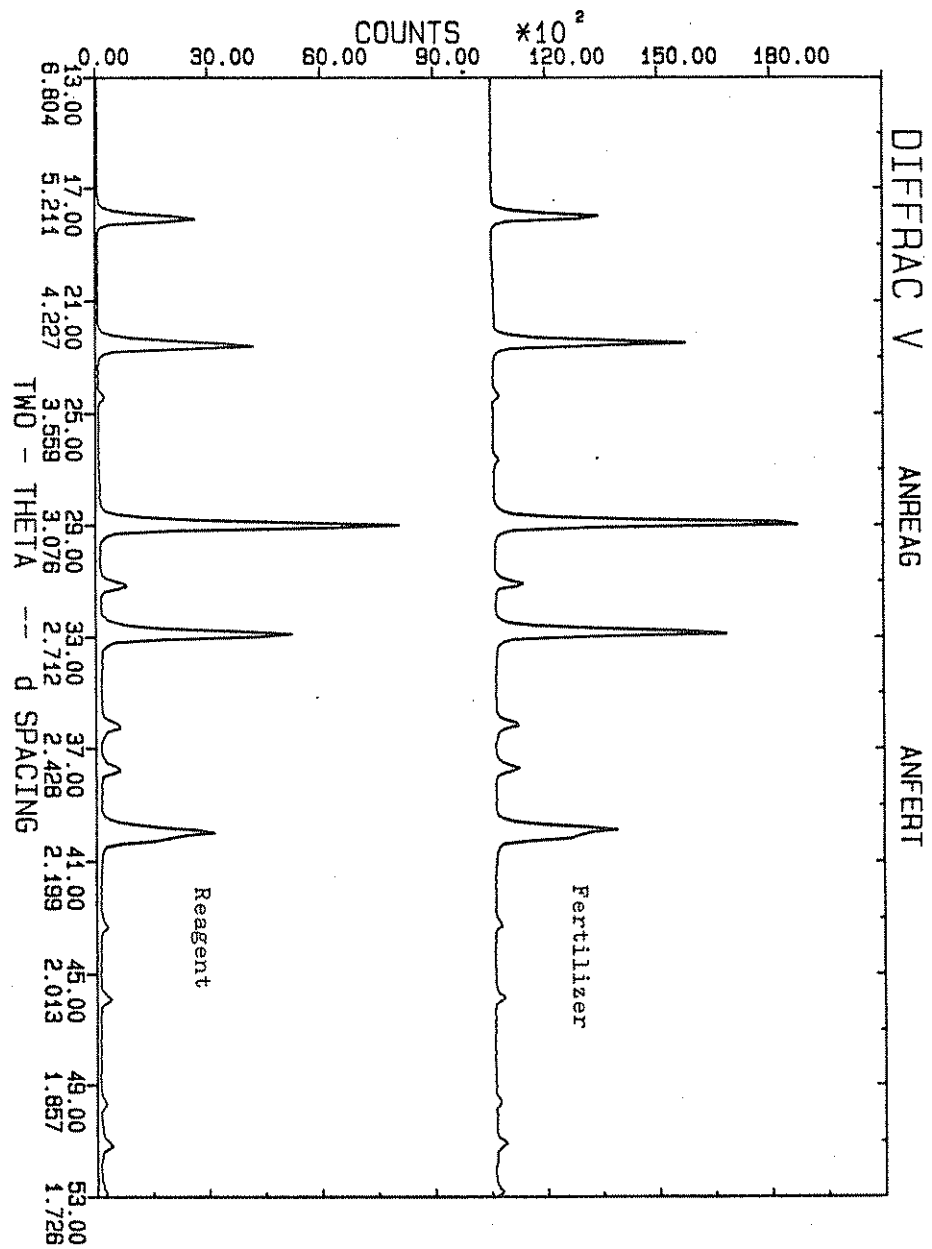


Figure 5. X-ray diffractograms of ammonium nitrate chemicals

commercial source is adequate.

#### Ammonium Phosphate

A sample of fertilizer grade dibasic ammonium phosphate (DAP) was obtained from the Farmers Grain Cooperative, Colo, Iowa. X-ray diffraction traces in Figure 6 indicate that with exception of minor amounts of aluminum phosphate (AL), the fertilizer and reagent grade ammonium phosphates are the same.

During the course of this study it was necessary to obtain additional samples of Neal #4 fly ash. The screening evaluation was done on a sample taken June 1, 1983. Test results for this sample and samples taken July 8, 1983 and July 18, 1984 are combined in Figure 7. Here it can be seen that both strength enhancement and an optimum amount of chemical additive may depend on the time of fly ash production and the additive grade. Table 9 serves to evaluate the phenomena, in that the ratio of strengths of treated to untreated specimens remains nearly constant. The outcome may not always be equally dramatic; however, significant improvement appears to be regular and may be predicted by evaluation of untreated fly ash.

Table 9. Effect of production time /Neal #4 fly ash

| Sample<br>Date | Untreated<br>Strength<br>(psi) | Treated<br>Strength<br>(psi) | Optimum<br>Additive<br>Rate % | Strength<br>Ratio |
|----------------|--------------------------------|------------------------------|-------------------------------|-------------------|
| 6/1/83         | 1850                           | 3250                         | 3-4                           | 1.8               |
| 7/8/83         | 1240                           | 2400                         | 10                            | 1.9               |
| 7/18/84        | 1030                           | 1740                         | 5-6                           | 1.7               |

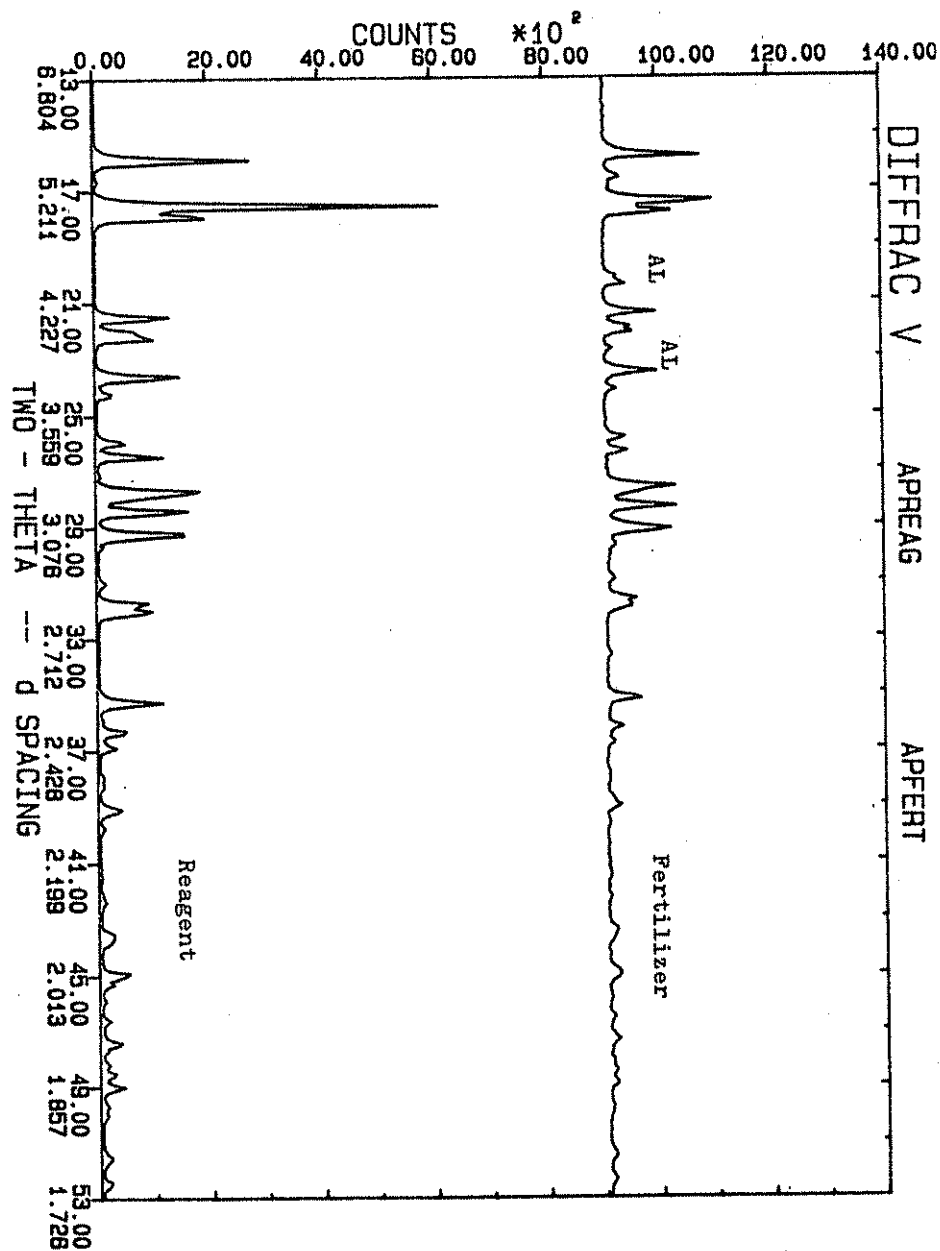
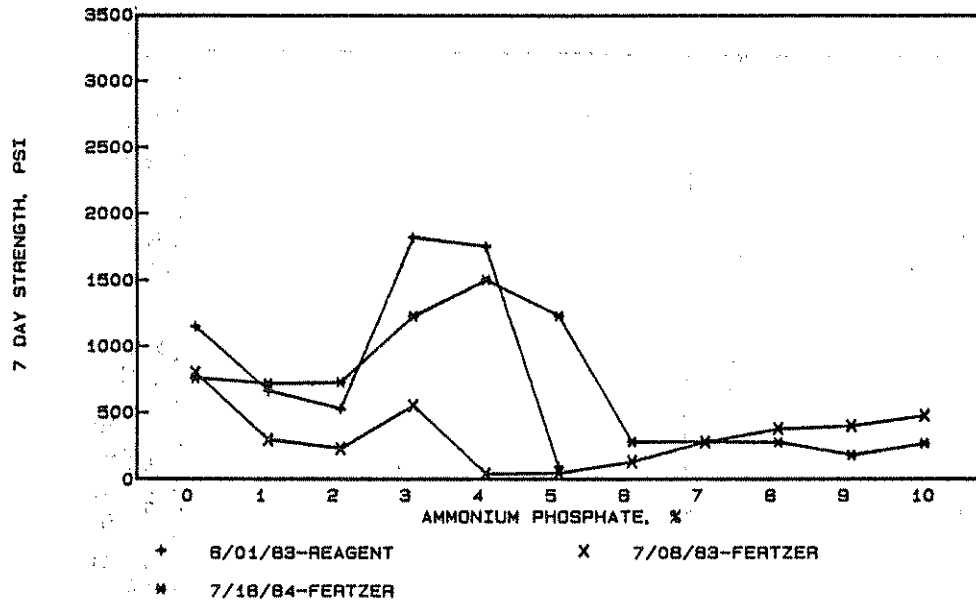


Figure 6. X-ray diffractograms of dibasic ammonium phosphate chemicals

## NEAL#4 - W/FA=0.24



## NEAL#4 - W/FA=0.24

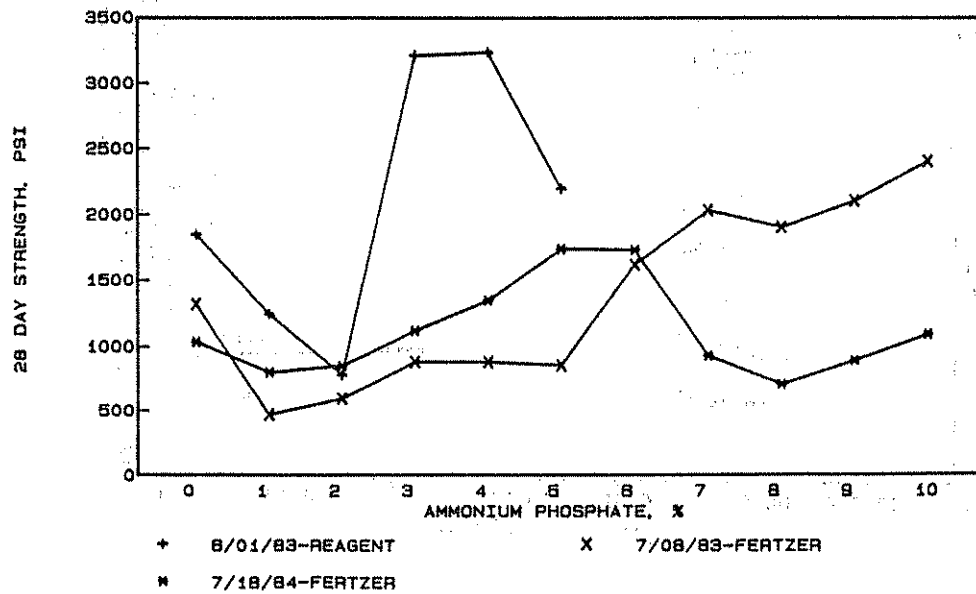


Figure 7. 7 day and 28 day compressive strength development of dibasic ammonium phosphate treatment of Neal 4 fly ashes

Evaluation of elemental and compound composition offers a partial explanation for variation in treated and untreated strength. Table 10 summarizes this data; it can be seen that total elemental composition and the elemental composition of the amorphous phase remained relatively constant. However, a direct correlation between tricalcium aluminate content and strength and the potential for an inverse relationship between percent glass and strength is observed. This suggests, at least for short term strength, crystalline composition of fly ash is a significant factor to reactions and the character of chemically modified fly ash cement.

This evaluation also is also useful for application. Although it is possible to categorize fly ash sources as to their general behavior, within such categorization there is considerable variation. The causes of such variation have yet to be identified and success in application will ultimately depend on experimentation for specific jobs.



Table 10. Elemental and compound composition of Neal #4 fly ash

|                           | Dates Sampled  |                |                |
|---------------------------|----------------|----------------|----------------|
|                           | <u>6/01/83</u> | <u>7/08/83</u> | <u>7/18/84</u> |
| <u>Elemental Analysis</u> |                |                |                |
| Silicon Oxide             | 36.09          | 39.91          | 34.66          |
| Aluminum Oxide            | 15.49          | 16.59          | 16.61          |
| Iron Oxide                | 6.22           | 5.37           | 5.88           |
| Total                     | 57.80          | 61.87          | 57.15          |
| Magnesium Oxide           | 5.98           | 5.71           | 6.00           |
| Calcium Oxide             | 26.16          | 24.01          | 25.73          |
| Titanium Oxide            | 1.07           | 1.05           | 1.13           |
| Sulphur Trioxide          | 3.65           | 2.23           | 3.21           |
| Phosphorous Pentoxide     | 0.69           | 0.71           | 1.29           |
| Potassium Oxide           | 0.28           | 0.43           | 0.30           |
| Sodium Oxide              | 2.09           | 2.19           | 2.38           |
| Total                     | 97.72          | 98.20          | 97.20          |
| <u>Compound Analysis</u>  |                |                |                |
| Mullite                   | 0.6            | 2.7            | 1.4            |
| Quartz                    | 9.9            | 10.1           | 7.2            |
| Ca. Alum. Sulphate        | 1.3            | 0.2            | 1.4            |
| Calcium Sulphate          | 3.1            | 1.6            | 3.2            |
| Magnetite                 | 5.7            | 2.4            | 4.0            |
| Calcium Oxide             | 1.1            | 0.5            | 0.7            |
| Magnesium Oxide           | 4.6            | 2.6            | 2.8            |
| Tri Calcium Alum.         | 4.3            | 3.4            | 1.0            |
| Total                     | 30.6           | 23.4           | 21.7           |
| Percent Glass             | 69.4           | 76.6           | 78.3           |
| <u>Glass Composition</u>  |                |                |                |
| Silicon Oxide             | 26             | 29             | 27             |
| Aluminum Oxide            | 13             | 13             | 14             |
| Calcium Oxide             | 21             | 21             | 23             |
| Magnesium Oxide           | 1.4            | 3.1            | 3.2            |

## BASE STABILIZATION

### Purpose and Scope

This portion of the research was aimed at obtaining a representative evaluation of the behavior of chemically modified fly ash cements (hereafter "cement"). The intent was to identify parameters significant to design and construction specifications. Research guidance was taken from classical work in portland cement stabilized bases, lime fly-ash bases, and portland cement concrete. Whenever possible, test controls and design criteria were adapted from existing technology.

Preliminary investigation was initiated with Neal #2 and Neal #4 fly ashes with crusher fines, a low value limestone product with a maximum 3/8 inch particle. After the fly ash base behavior was characterized, the technology was expanded to include fly ash from other sources.

### Additive Concentration

The first step of the experimental design was to select the appropriate concentrations based on optimization of strength and set requirements as may apply to a particular batch of fly ash. Test results in Figures 8 and 9 are from strength and set tests of Neal #4 fly ash at varying fertilizer grade ammonium phosphate concentrations. In this case, three percent ammonium phosphate was selected as a compromise between strength and set time. This led to cement strengths of 2500 psi with 40 minutes until final set.

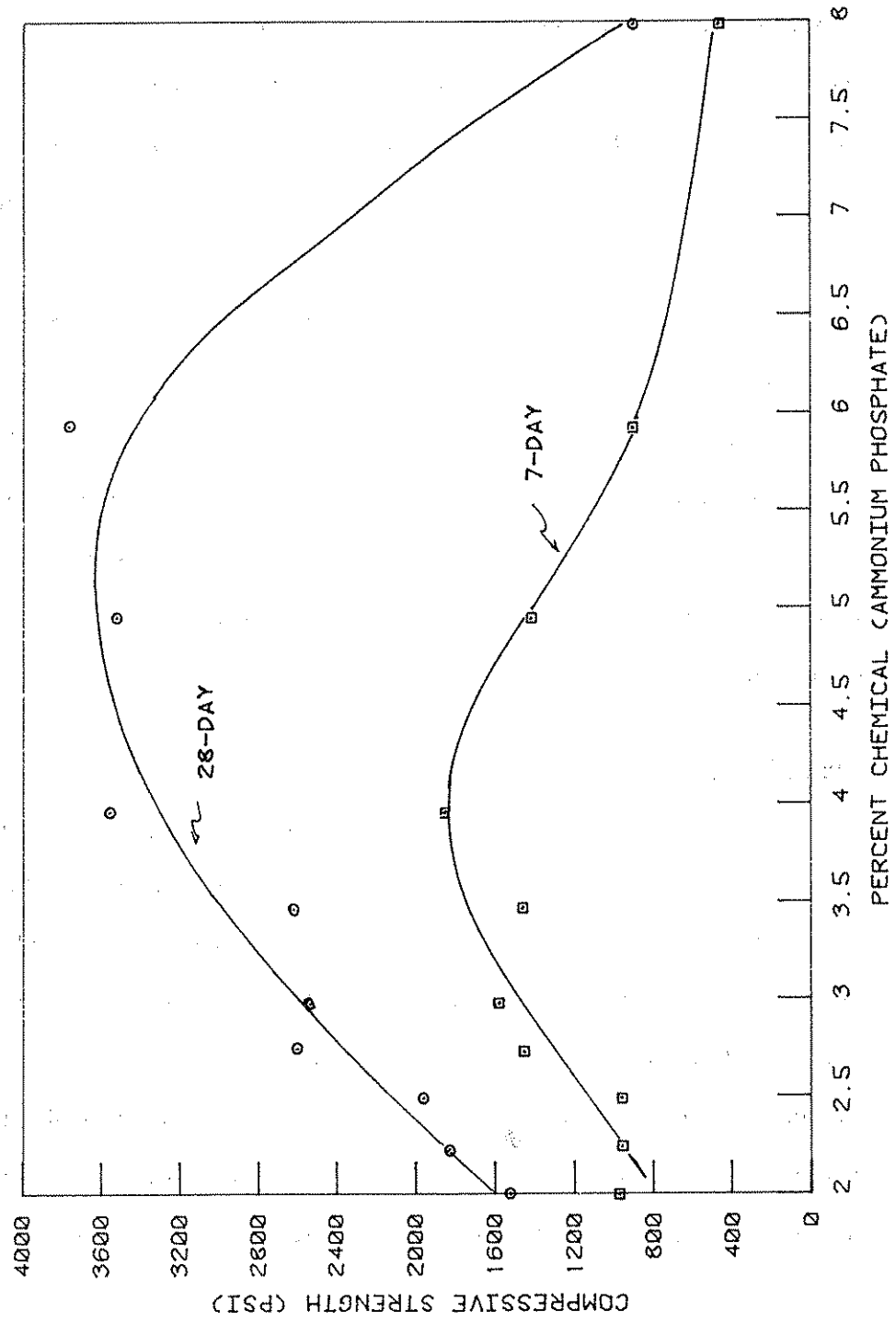


FIGURE 8 : COMPRESSION STRENGTH VERSUS PERCENT CHEMICAL (NEAL #4)

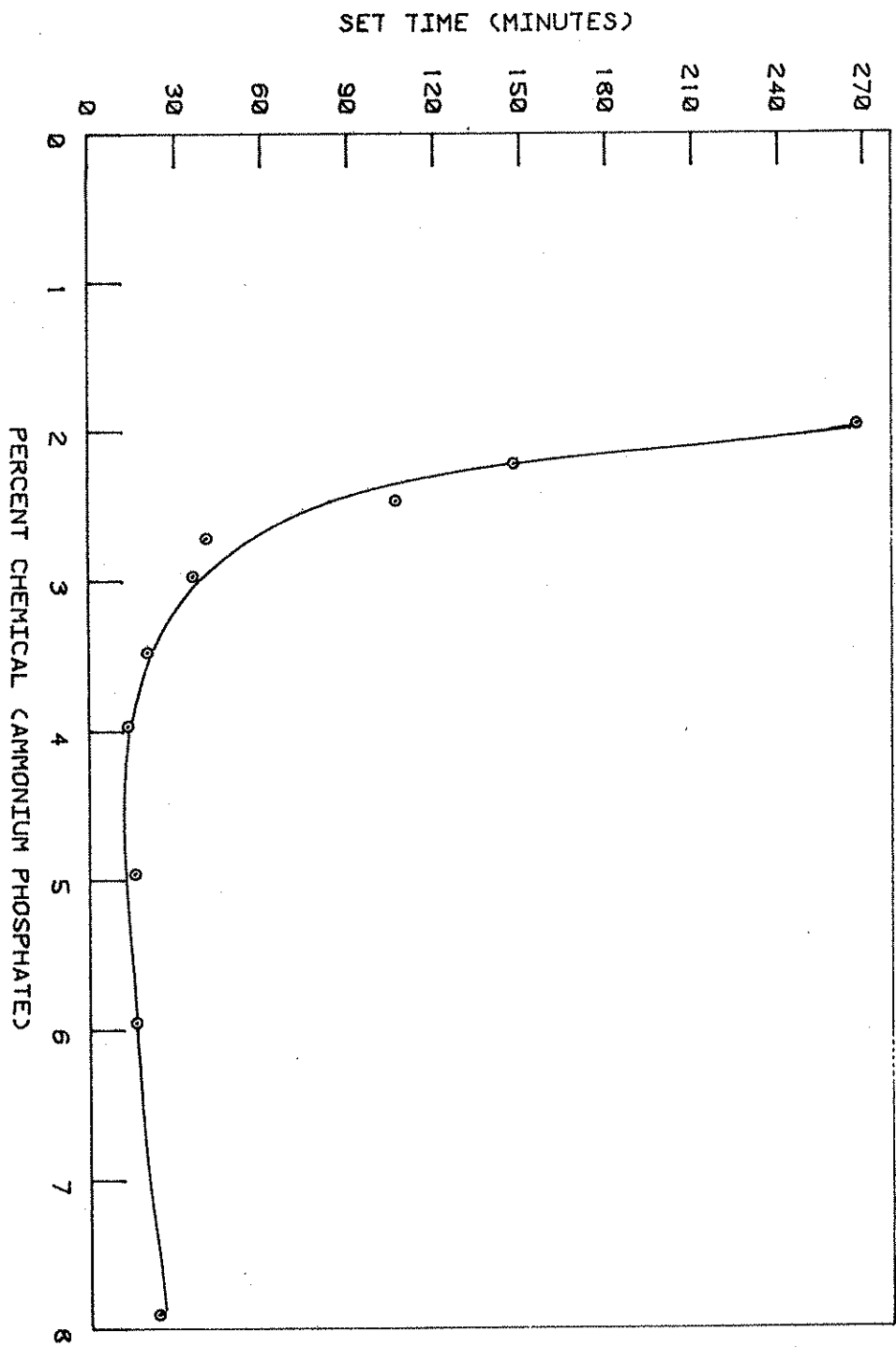


FIGURE 9 : SET TIME VERSUS PERCENT CHEMICAL (NEAL #4)

Similar tests were performed with the other fly ashes included in this study and the results are included in Appendix C. The fly ash sources and selected additive concentrations are summarized in Table 11.

Table 11. Fly ashes and additives

| <u>Fly-ash</u> | <u>Additive &amp; Concentration</u>                               |
|----------------|---|
| Neal #2        | 2 percent ammonium nitrate  |
| Neal #4        | 3 percent ammonium phosphate                                      |
| Lansing        | 2 percent ammonium phosphate                                      |
| Louisa         | 1 percent ammonium phosphate                                      |
| Ottumwa        | no additive (additive concentrations were beyond economic reason) |

#### Aggregate

Three-eighths inch limestone crusher fines from Martin-Marietta quarry in Ames, Iowa were selected as being typical of such materials available throughout the state. Mixes designated as "composite" for aggregate gradation were as received from the quarry. The composite gradation shown in Figure 10 represents an A-1-a soil. The composite aggregate also was broken into six uniform sieve sizes designated "A" through "F" (Table 12) to facilitate evaluation of gradation, particle size, and particle surface area on fly ash treated base material.

Dry rodded unit weight (ASTM C 29) and saturated surface dry moisture content and specific gravity (ASTM C 127 and C 128) tests

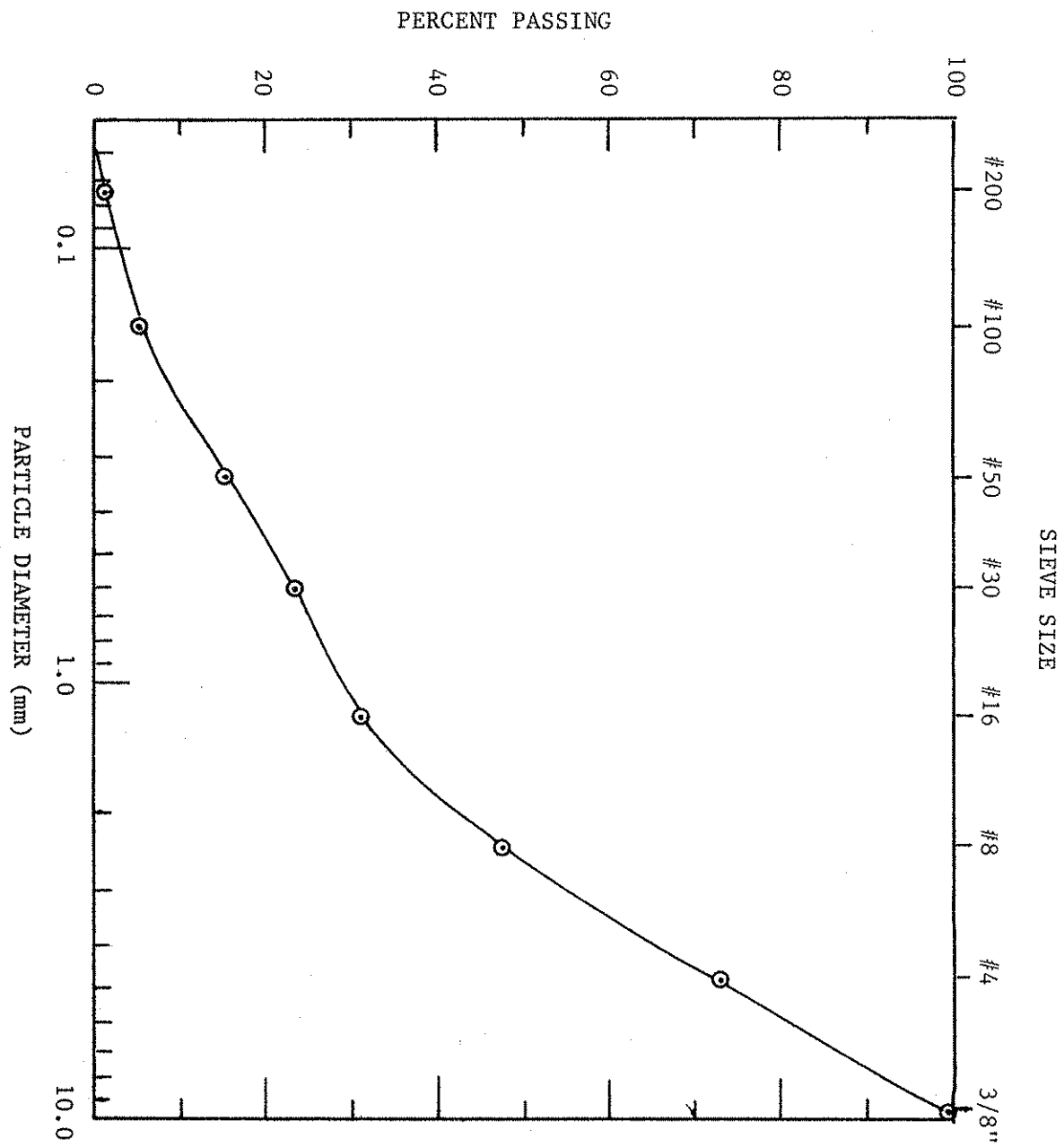


FIGURE 10. SIEVE ANALYSIS (COMPOSITE AGGREGATE)

Table 12. Aggregate Description.

| Aggregate Designation      | Sieve Fraction | Dry Rodded Unit Weight (pcf) | SSD Moisture Content (%) | Relative Surface area |
|----------------------------|----------------|------------------------------|--------------------------|-----------------------|
| A                          | 3/8" to 4+     | 91.1                         | 2.64                     | 4                     |
| B                          | 4- to 8+       | 91.4                         | 2.22                     | 8                     |
| C                          | 8- to 16+      | 95.2                         | 3.41                     | 16                    |
| D                          | 16- to 30+     | 100.6                        | 4.79                     | 32                    |
| E                          | 30- to 50+     | 100.9                        | 0.438                    | 64                    |
| F                          | 50- to 100+    | 96.7                         | 0.127                    | 128                   |
| COMPOSITE<br>(as received) | --             | 115.7                        | 2.20                     | 30                    |

Table 13. Calculation of Relative Surface Area for Composite.

| Sieve | % Retained | Relative Surface                     | % Ret. Rel. Sur. |
|-------|------------|--------------------------------------|------------------|
| 3/8   | 0.57       | 2                                    | 0.01             |
| 3/16  | 26.52      | 4                                    | 1.06             |
| 8     | 25.23      | 8                                    | 2.02             |
| 16    | 17.00      | 16                                   | 2.72             |
| 30    | 7.24       | 32                                   | 2.32             |
| 50    | 8.79       | 64                                   | 5.63             |
| 100   | 9.87       | 124                                  | 12.63            |
| 100-  | 4.78       | <100                                 | 4.8              |
| TOTAL | 100.00%    | Surface area of 3/8" composite: 31.2 |                  |

were performed as they relate to voids available for cement filling. Specific gravity was found to be 2.68. These results are in Table 12.

Aggregate surface area is a parameter known to be important to the behavior of portland cement concrete. It has been correlated to cement demand, workability and strength. Thus aggregate surface area is suspected to be of equal or greater importance to granular base stabilization than it is to portland cement concrete, because of potential for greater variation in aggregate gradation. Direct measurement of aggregate surface area is difficult, but the equally useful relative surface area can be determined from a gradation curve. The last column of Table 12 is surface area of specific sieve fractions as related to a standard, in this case, that for surface area of particles between the 1 1/2" to 3/4" sieves.

Relative surface area for a composite can be computed as a weighted mean. Results of such a computation are shown in Table 13.

Void space of the aggregate must be considered in terms of the water and fly ash contents required to fill available space. Based on the dry rodded unit weights, Table 12, and the aggregate specific gravity of 2.68, the available free space (AFS) can be computed as:

$$AFS = 1 - \frac{\text{Dry Rodded Unit Weight}}{(2.68)(62.4 \text{ lb/cu.ft.})}$$

For this work, fly ash cement reaction products are assumed to



equal the volume of the constituents. This approximation should yield insight to the effects of the volume of fly ash and water in a mix for comparison with available void spaces. Figure 11 shows the necessary combinations of water/cement ratio and cement content to fill voids in the compacted aggregate. Combinations falling below this line would have inadequate water and fly ash to fill voids. Cement content is defined as percentage of fly ash based on the weight of aggregate.

#### Neal #2 and Neal #4 Fly Ash Cement Base

An in-depth study of stabilized base courses from Neal #2 and Neal #4 fly ashes was performed to define and evaluate several design considerations.

Water/cement ratio -- The strength of additive treated fly ash cements was determined at water/cement ratios ranging from 0.1 to 0.5 to see how chemically modified cements behaved with respect to water available for hydration. Figure 12 shows a typical effect of water/cement ratio on strength properties of the 3 percent ammonium phosphate treated Neal #4 fly ash. Results for other fly ashes are presented in Appendix D. It can be seen that for each fly ash there is an optimum water/cement ratio for maximum strength. Below it cement is too dry and there is not enough water for hydration. Above optimum water/cement ratio, strength is reduced due to higher porosity caused by excess water. Results in Figure 12 show a predictable response essential to a rational design procedure. Since

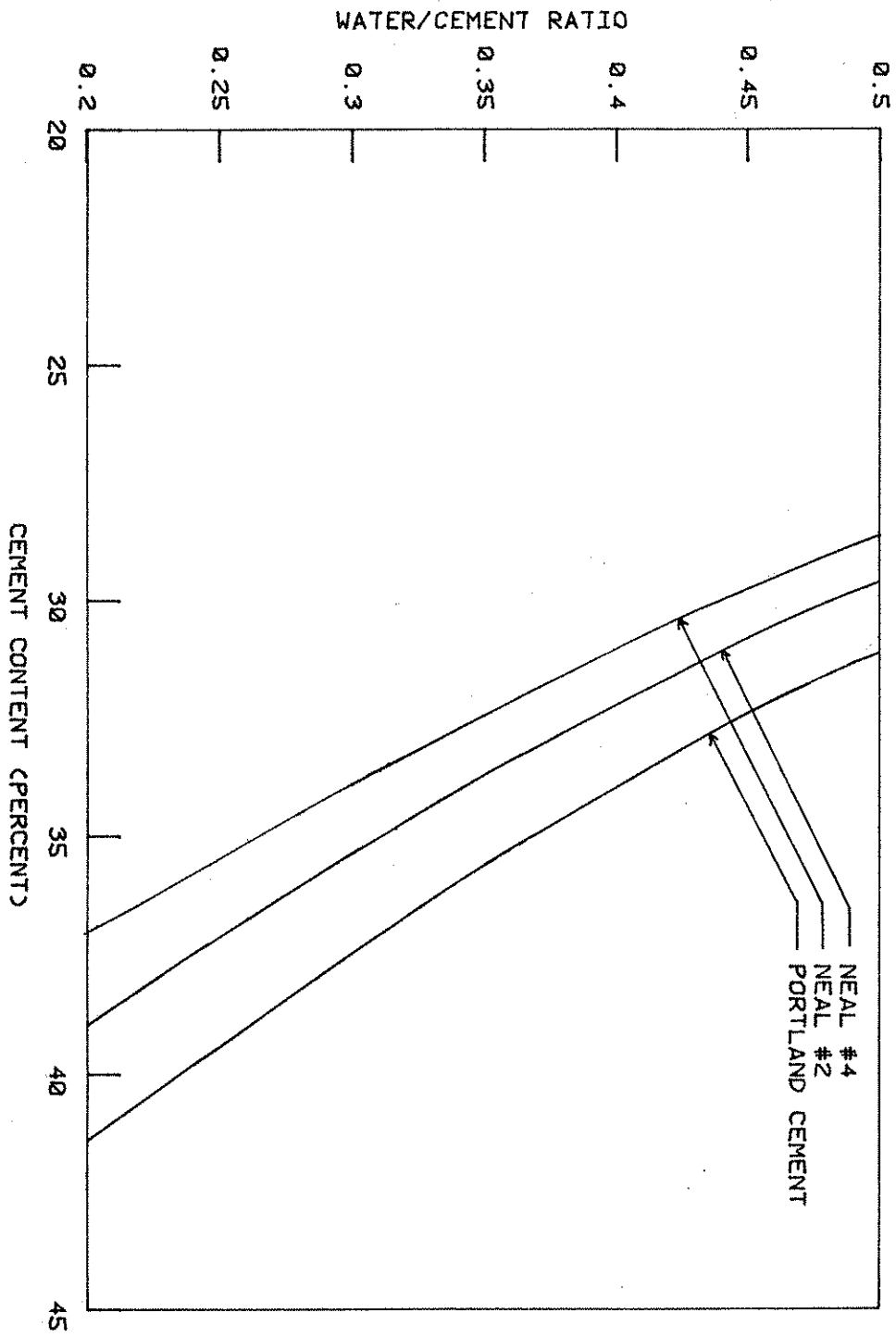


FIGURE 11. REQUIRED MIX PROPORTIONS TO FILL VOIDS IN AGGREGATE

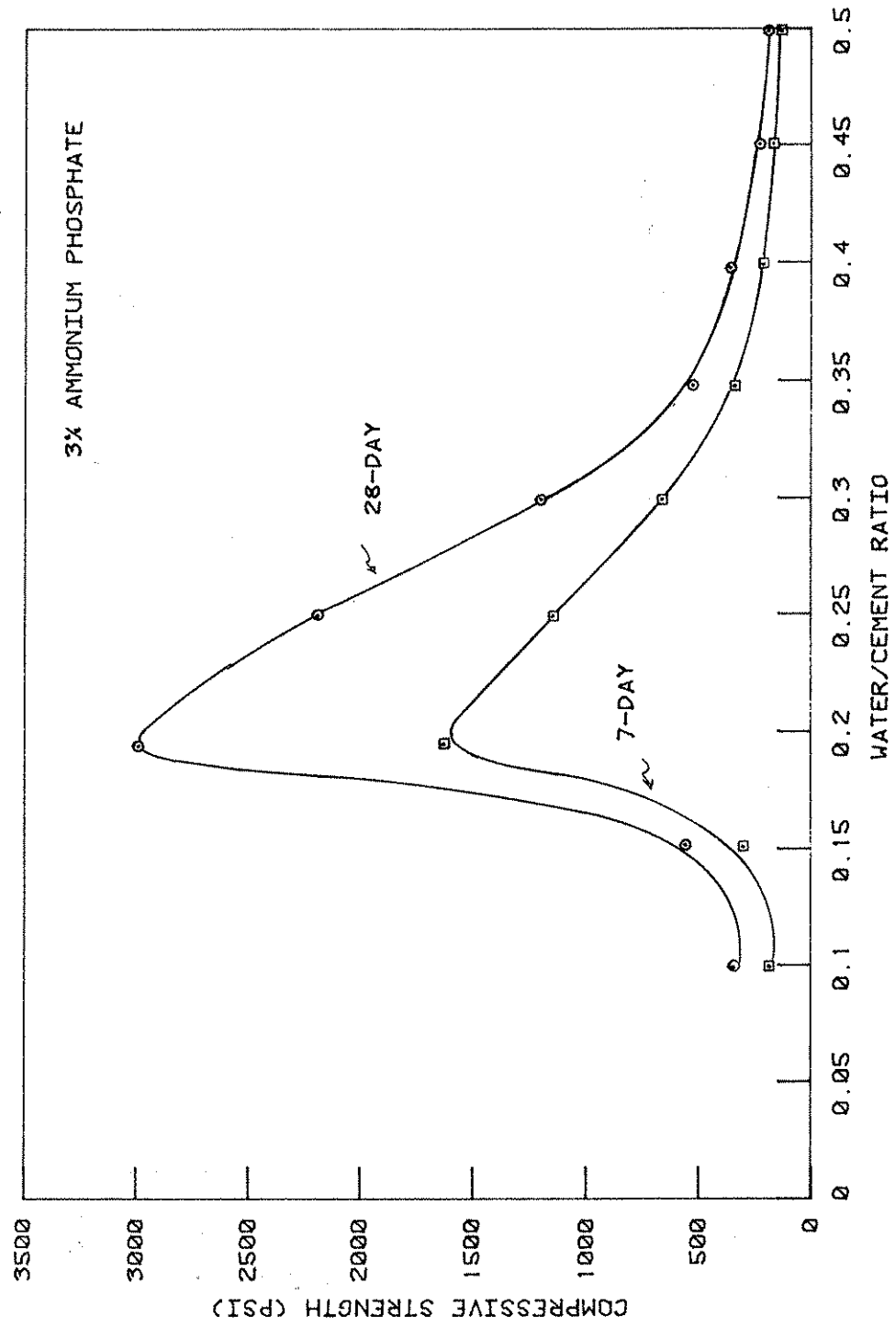


FIGURE 12. COMPRESSIVE STRENGTH OF FLYASH PASTE VERSUS WATER/CEMENT RATIO (NEAL #4)

the descending leg represents paste fluidity capable of allowing a compactible aggregate mix, it is of particular importance. Obviously, design water contents should be held as near the optimum as possible while allowing placement.

A significant feature of the water/cement ratio response is that it is similar to that of portland cement. Design criteria appropriate for portland cement may also be the same for chemically modified fly ash.

To support a preliminary design method, the response in Figure 12 is represented by the classical water/cement ratio versus strength relationship often used for portland cement. The relations are as follows:

$$f_c' (7 \text{ days}) = \frac{10,487}{e^{9.3(w/c)}}; \quad f_c' (28 \text{ days}) = \frac{23,380}{e^{10(w/c)}}$$

where  $w/c$  = water/cement ratio

**Moisture-density** -- To evaluate the compaction properties of fly ash stabilized base courses, a series of standard Proctor tests (ASTM 698) were performed with the composite aggregate and the two fly ashes. As previously determined, 2% ammonium nitrate was added to Neal #2 mix water and 3% ammonium phosphate was added to Neal #4 mix water. Five proctor specimens were made at each of several cement contents to determine optimum moisture contents.

Aggregate was brought to SSD moisture content before compaction so molding water was available for hydration. Density measurements

for Neal #2 and Neal #4 fly ash are shown in Figures 13 and 14 respectively. The reduction in density as cement content increases can be attributed to the low specific gravity of the fly ash hydrate.

Since the aggregate used for these tests was at SSD moisture, the water added to the mix was available for hydration and can be considered in terms of the amount of cement added, or the water cement ratio. This ratio is identical to that conventionally used for portland cement mix design.

In Figures 15 and 16, the relationships at maximum density between water/cement ratio and percent cement for Neal #2 and Neal #4 fly ashes show increasing cement decreases the water required to achieve maximum density. This reduction in water requirement can be attributed to the spherical shape of fly ash particles, which eases or "lubricates" the movement of angular limestone thus requiring less water to achieve compaction.

Strength -- After determination of optimum water/cement ratios (analogous to optimum moisture contents), a set of 2" diameter by 4" long cylindrical specimens was compacted at optimum water/cement ratios. Batches were made of several cement contents and specimens were tested in compression and tension after 14 and 28 days curing.

Compression test results, shown in Figure 17 and 18, show maximum 28 days strengths of about 1200 psi for Neal #2 cement and 2400 psi for Neal #4. For both cements, limiting strength is reached at cement content near 30%; this is in agreement with calculated void filling requirements.

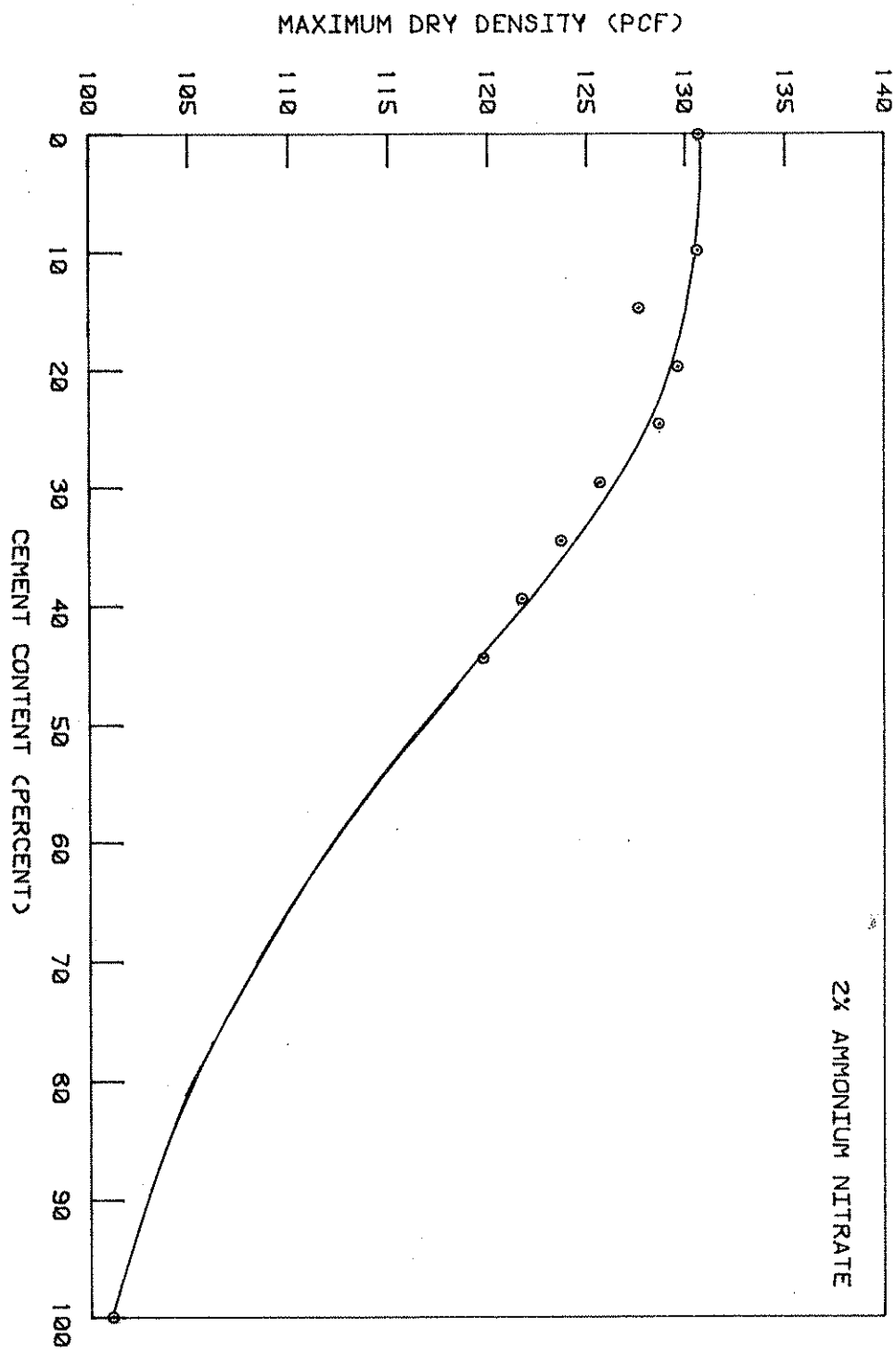


FIGURE 13. MAXIMUM DRY DENSITY VERSUS CEMENT CONTENT (NEAL#2)

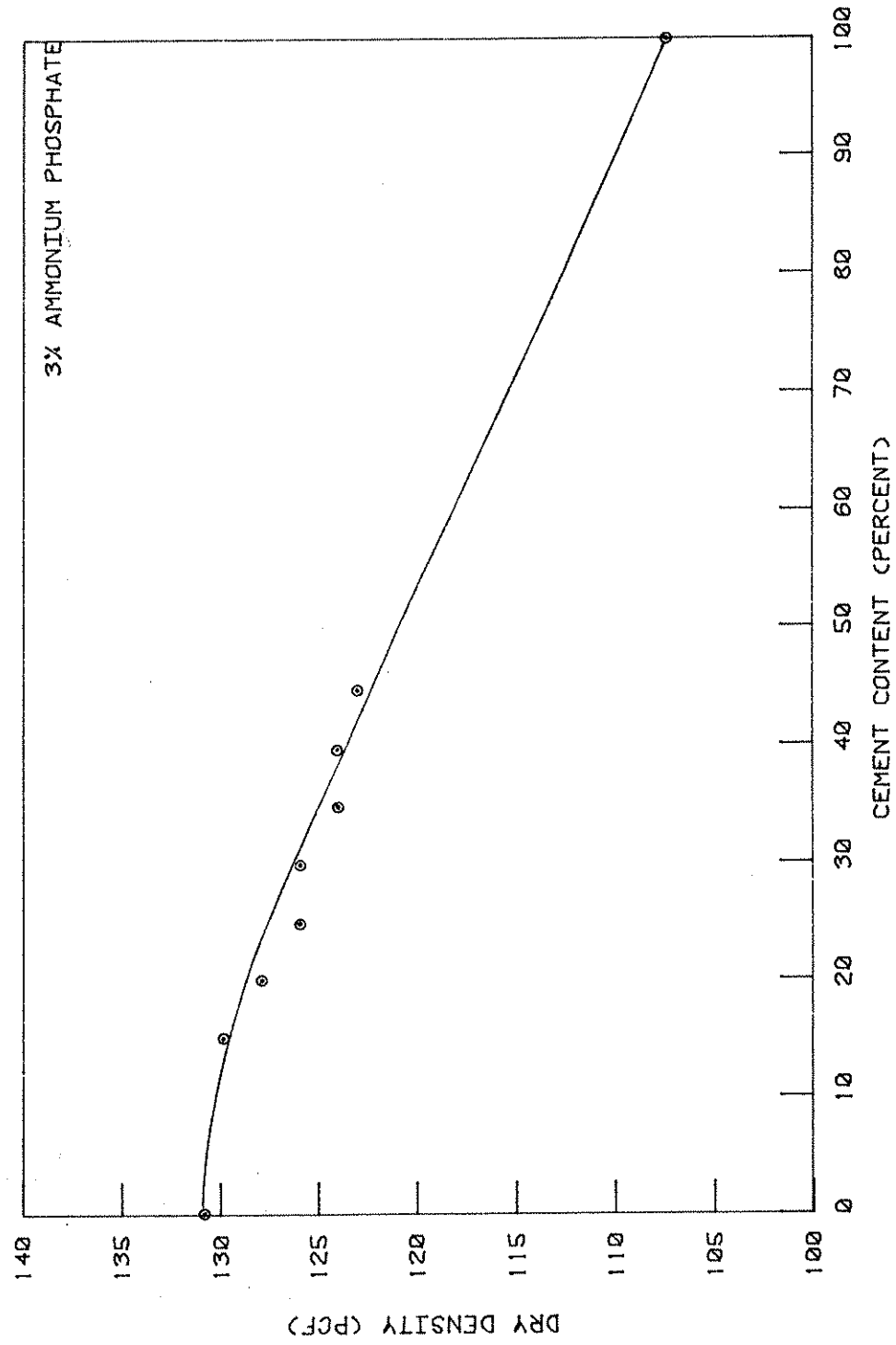


FIGURE 14. MAXIMUM DRY DENSITY VERSUS CEMENT CONTENT (NEAL #4)

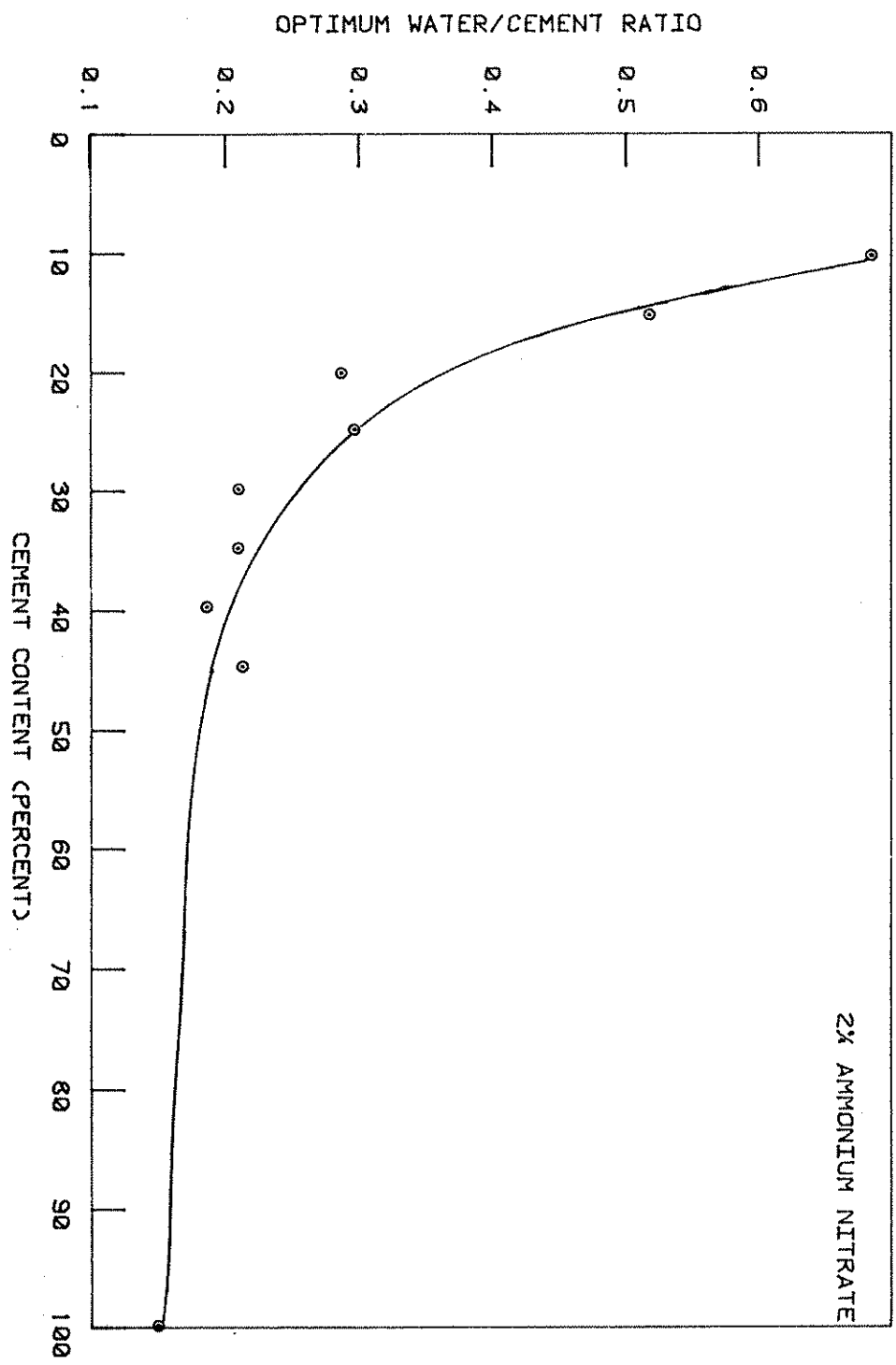


FIGURE 15. OPTIMUM WATER/CEMENT RATIO VERSUS CEMENT CONTENT (NEAL #2)



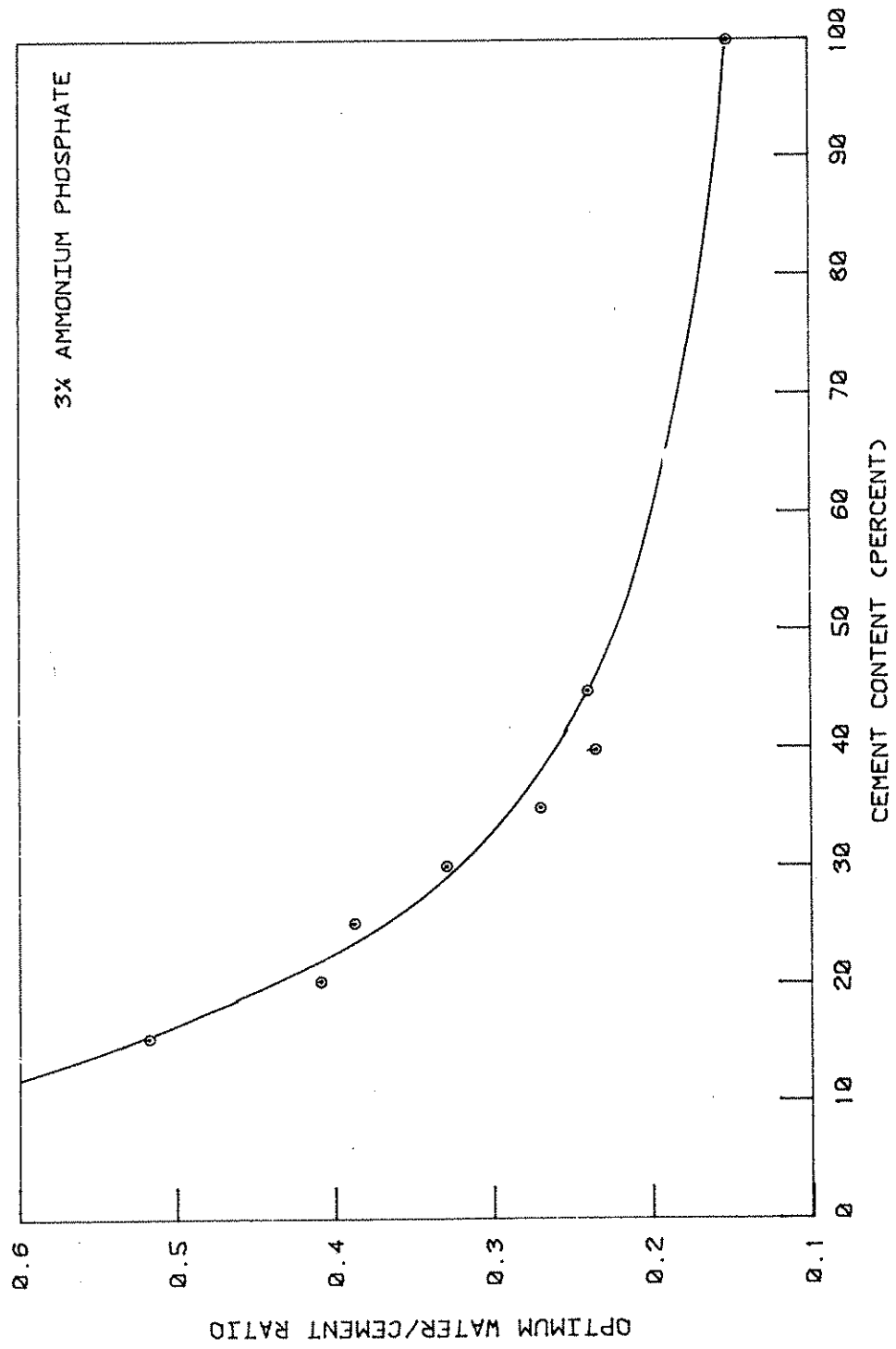


FIGURE 16. OPTIMUM WATER/CEMENT RATIO VERSUS CEMENT CONTENT (NEAL #4)

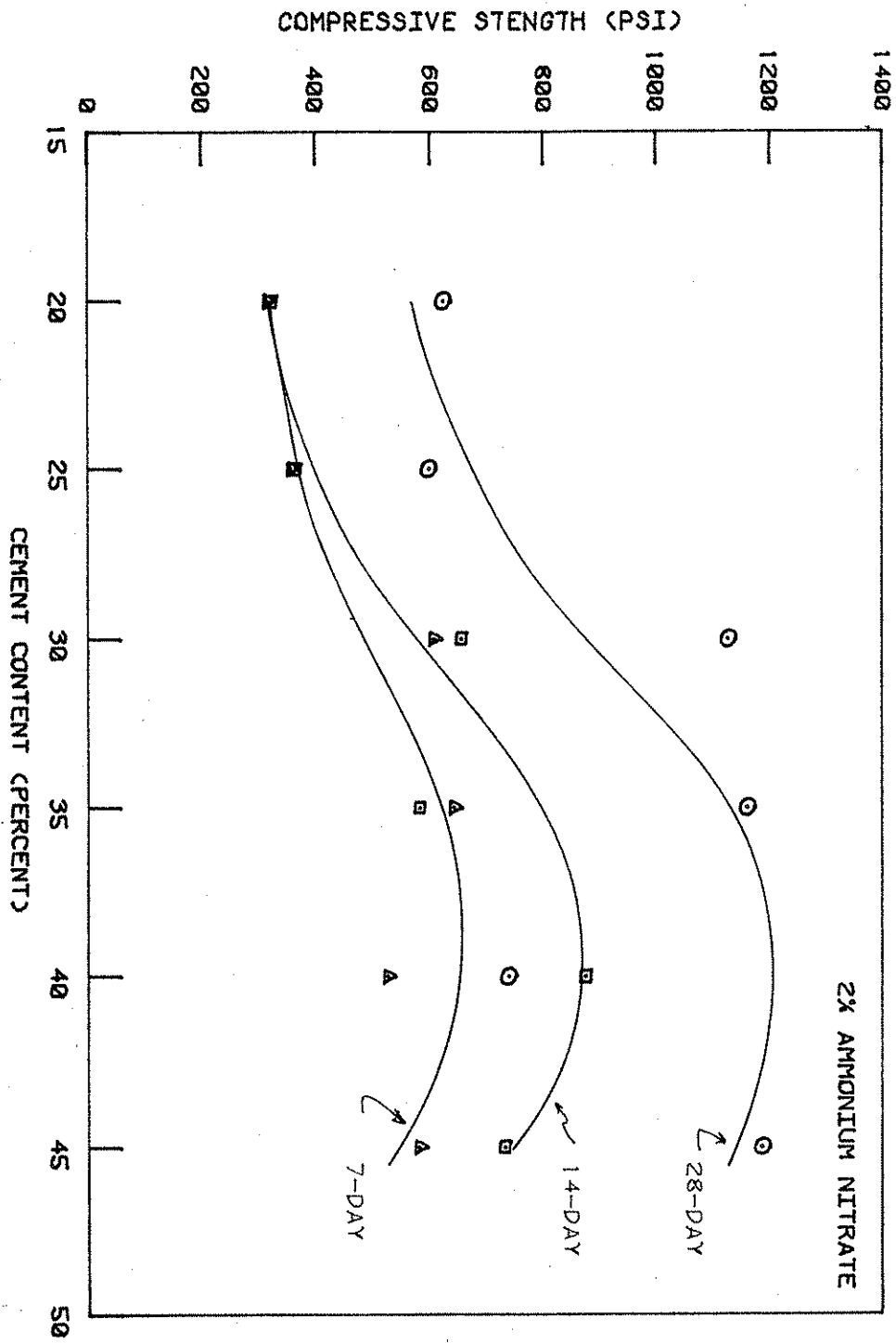


FIGURE 17. COMPRESSIVE STRENGTH VERSUS CEMENT CONTENT AT OPTIMUM  
MOISTURE CONTENT (NEAL #2)

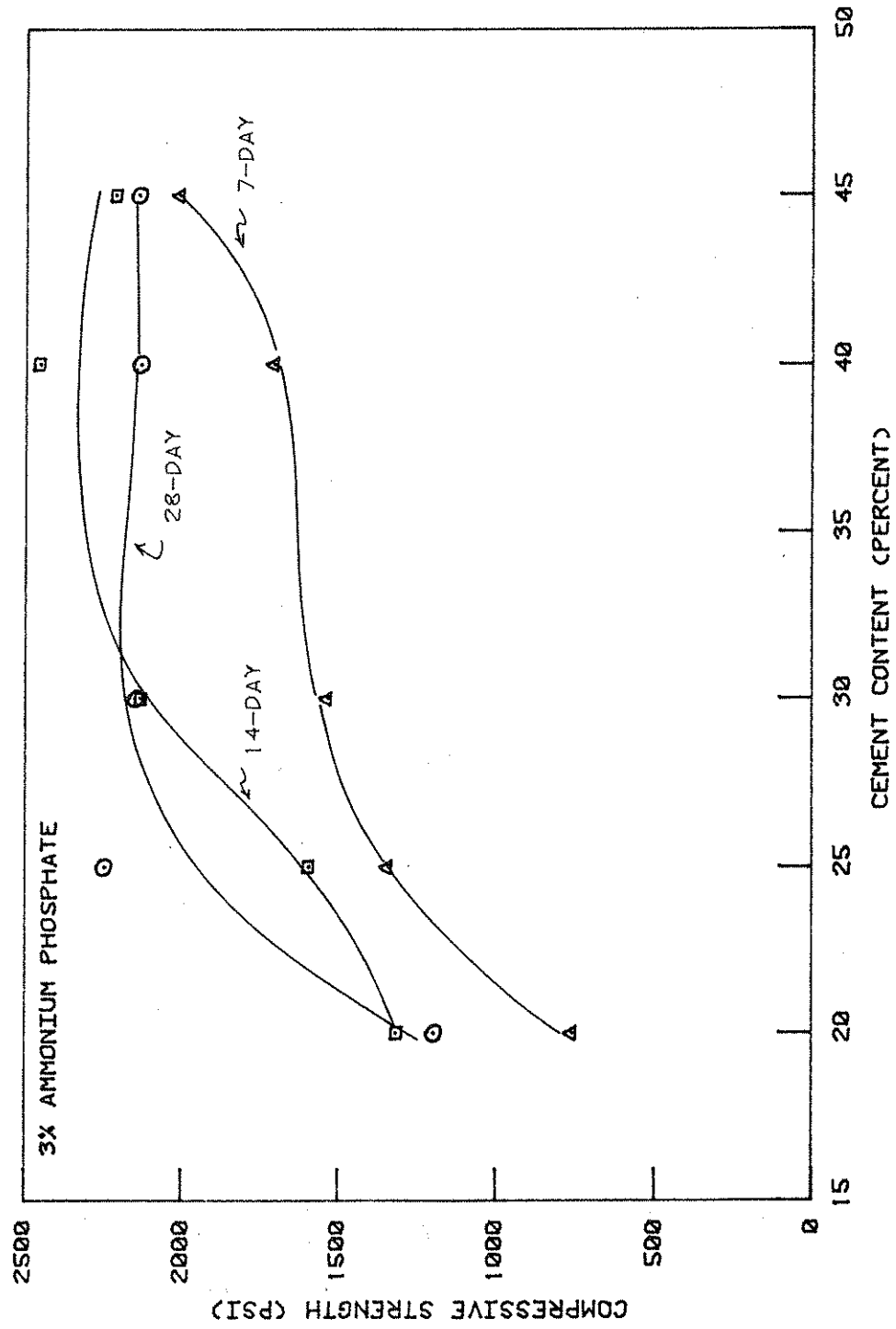


FIGURE 18. COMPRESSIVE STRENGTH VERSUS CEMENT CONTENT AT OPTIMUM MOISTURE CONTENT (NEAL #4)

Tensile test results, shown in Figure 19 and 20, demonstrate the same relationship as for compressive strength. Regardless of age, strength of fly ash cement stabilized gravel appears to be about 16% of compressive strength. This is somewhat more than the 10% common for portland cement stabilized materials.

Rates of strength gain for cements derived from Neal #2 and Neal #4 fly ashes are given in Table 14. About 60 percent of compressive strength for both cements is realized in 7 days, while 75 to 85 percent is available at 14 days.

Table 14. Strength Over Time Changes

|         | Percent 28-day strength |         |             |         |
|---------|-------------------------|---------|-------------|---------|
|         | 7 day                   |         | 14 day      |         |
|         | Compression             | Tension | Compression | Tension |
| Neal #2 | 60%                     | 60%     | 75%         | 75%     |
| Neal #4 | 60%                     | 65%     | 85%         | 75%     |

Consistency measurement -- Proctor moisture-density testing provides the consistency information necessary to determine whether laboratory mixes can be compacted in the field. However, running a complete series of five tests to determine optimum water content, or water/cement ratio, for several water/cement levels is a costly process. Additionally, the Proctor compaction mechanism bears little resemblance to vibratory compaction, which has been found to be more

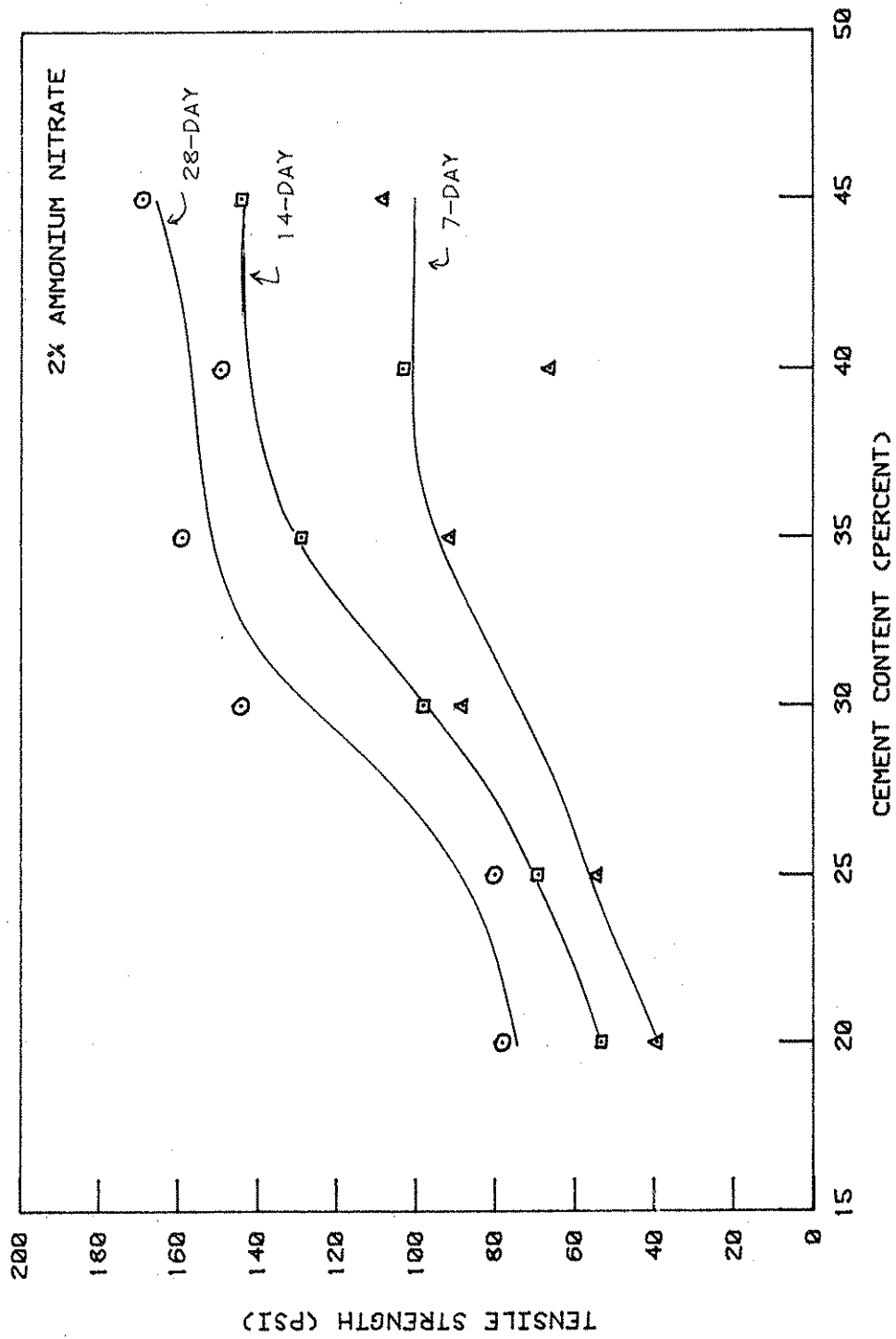


FIGURE 19. TENSILE STRENGTH VERSUS CEMENT CONTENT AT OPTIMUM MOISTURE CONTENT CNEAL #20

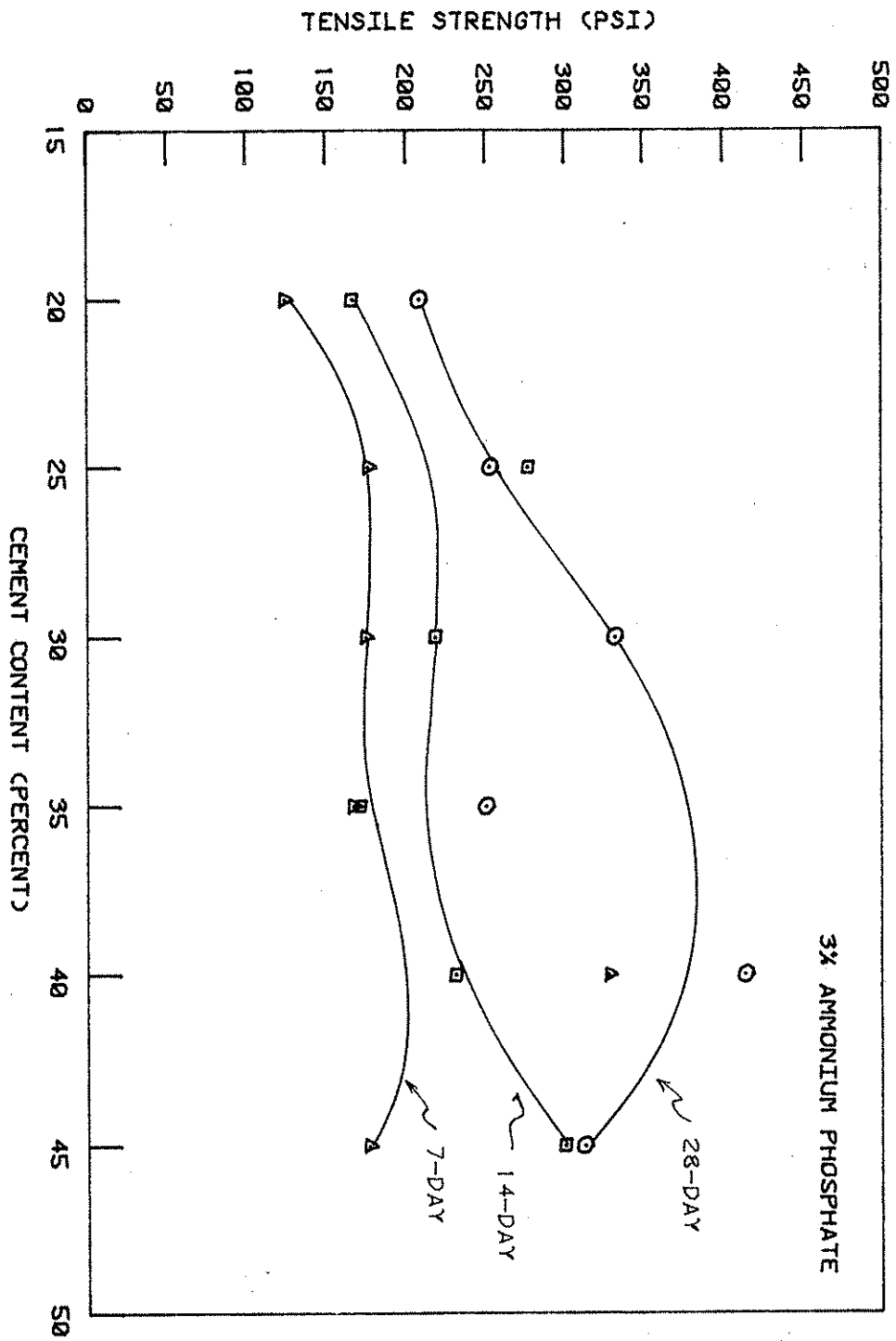


FIGURE 20. TENSILE STRENGTH VERSUS CEMENT CONTENT AT OPTIMUM MOISTURE CONTENT (NEAL #4)

efficient with cement-stabilized granular materials. Thus the Vebe test, an ACI standard for lean, no-slump concrete, was selected as a means for consistency measurement. This Vebe test was originally developed in Sweden and has recently been applied to rolled concrete in the U.S., as well as to lime-fly ash base stabilization in Britain.

The Vebe apparatus consists of a vibrating table supported by rubber shock absorbers which are connected to a heavy concrete base. A removable, cylindrical metal bucket is secured to the vibrating table top. A schematic of the apparatus is shown in Figure 21.

A standard slump cone was used to mold a mix in the bucket according to ASTM C 143-78. After the metal slump cone is removed, a transparent plastic disk with a steel rod threaded at the center is placed on top of the cone and allowed to slide up and down inside the bucket. After the vibrator is switched on, the time (in seconds) required to deform the cone into a cylinder is recorded as the "Vebe Time". Complete deformation is defined as the time at which the entire surface of the plastic disk is in direct contact with the mixture.

The Vebe test permits rapid determination of workability and is capable of yielding consistent mixes, but also requires calibration for correlation with the Proctor Density Test. To calibrate the Vebe test, mixes for Neal #2 and Neal #4 were duplicated at optimum moisture content and their Vebe times determined. Vebe times of  $43 \pm 3$  seconds were obtained for the Neal #2 mix and  $40 \pm 4$  seconds for the Neal #4 mix. These tests indicate that for variable fly ash contents

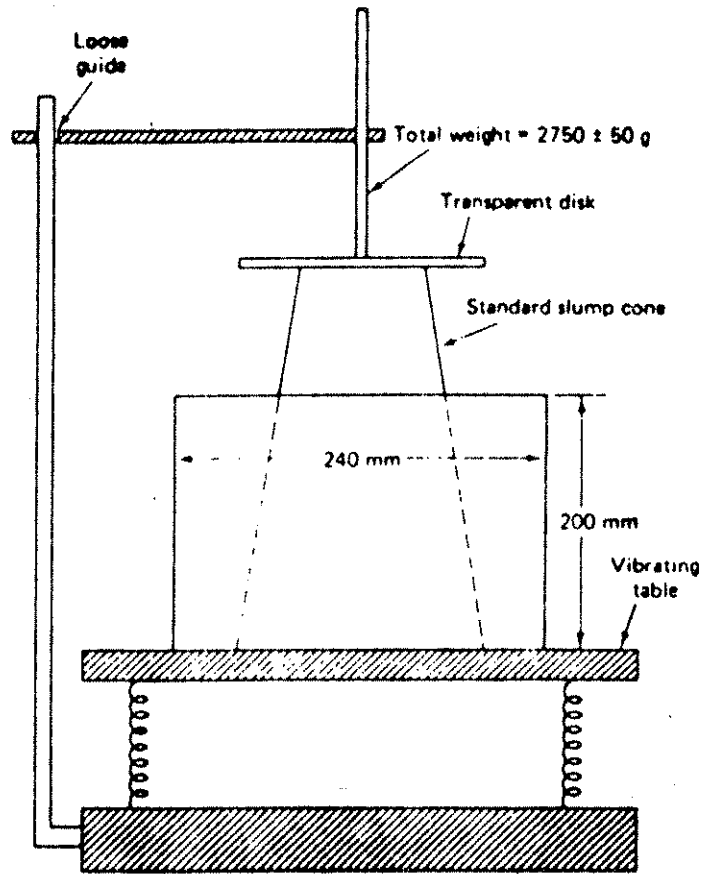


Figure 21. Vebe apparatus.



at optimum moisture content, the Vebe time was constant.

For the remainder of the experimental work, a Vebe time of  $43 \pm 3$  seconds was used for control of consistency.

#### Fly Ash Base Behavior

To generalize design of fly ash stabilization beyond an empirical determination (i.e., test batches for all potential materials), it is necessary to identify and analyze variables pertinent to performance. The performance of a mix can be characterized by compressive and tensile strength, workability or compactibility, density, and resistance to freeze-thaw shrinkage. The design variables evaluated in this phase of the work include cement content, aggregate surface area, and water/cement ratio.

Cement content -- To evaluate the effects of cement content on strength, trial batches were made with Neal #2 fly ash (using the separate sieve size aggregates detailed in the materials section) with six cement contents ranging from 20 to 45 percent. Five hundred and four specimens were made; 12 samples were made for each of the seven aggregates at each of the six cement contents. The water/cement ratio of all mixes produced was adjusted to maintain 42 seconds Vebe time. Four samples of each cement content were tested at 7, 14, and 28 days, two in compression and two in tension.

In Figure 22 and 23 are 28-day strengths for compression and tension tests, respectively. With one exception (aggregates E and F), a notable trend for both compression and tension was observed;

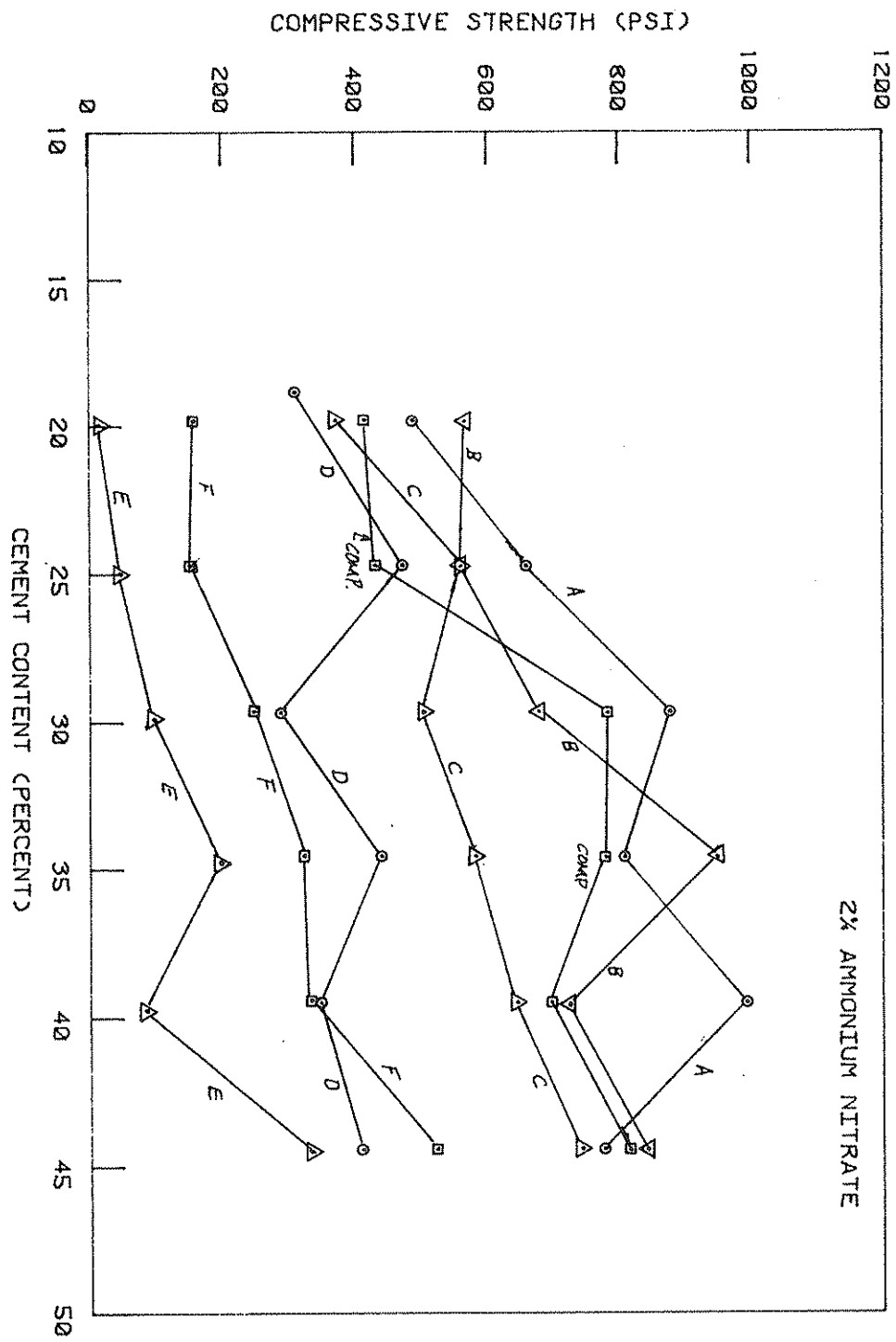


FIGURE 22. COMPRESSIVE STRENGTH VERSUS CEMENT CONTENT (NEAL #2)

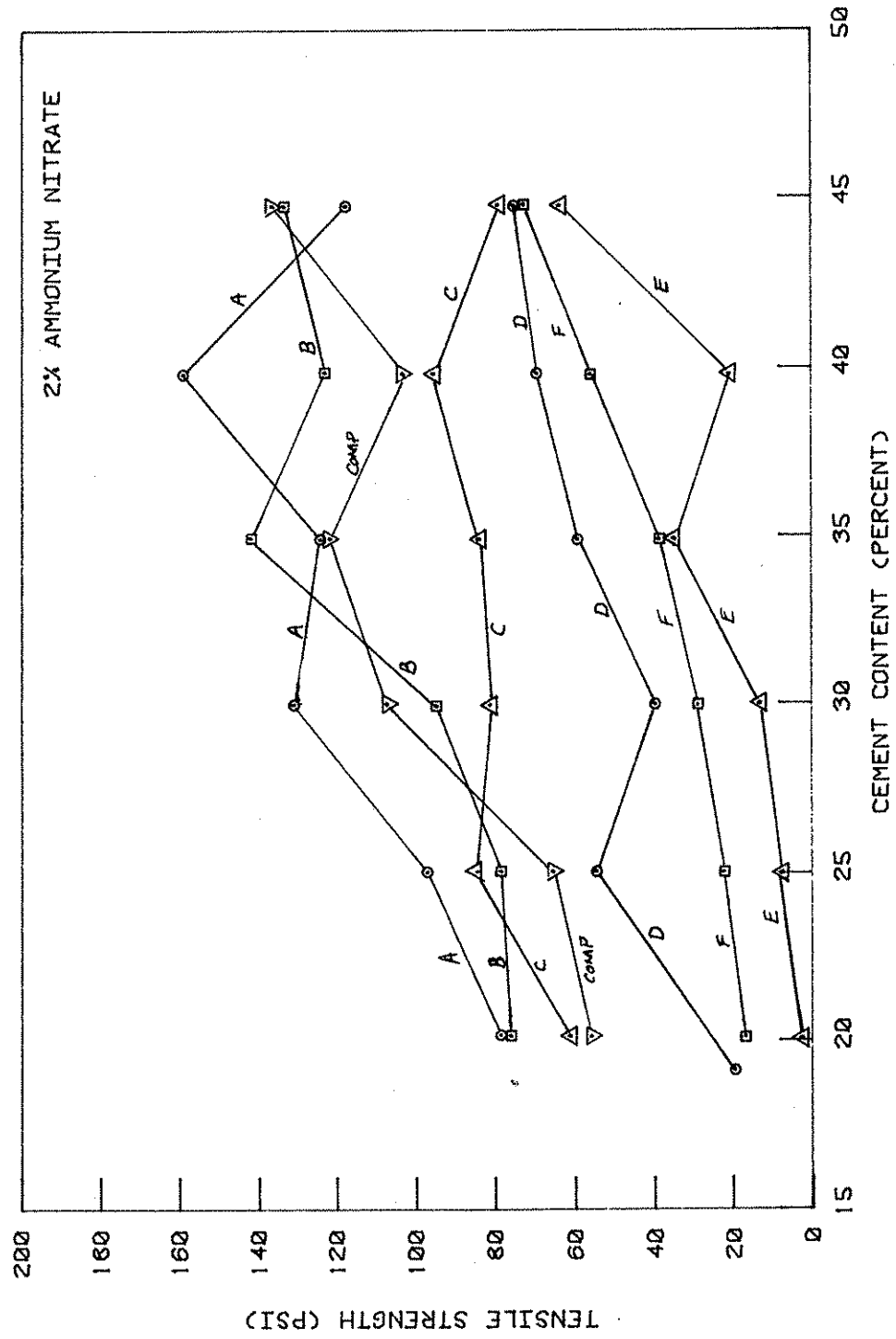


FIGURE 23. TENSILE STRENGTH VERSUS CEMENT CONTENT (NEAL #2)

increased strength with increasing aggregate size. Additionally the composite aggregate generally fell between aggregates B and C.

It must be remembered that all of these mixes are at optimum consistency, and water/cement ratio is not constant. As cement content was increased, water/cement ratio was decreased to maintain the same level of compactibility. This increase in strength at cement contents in excess of those required for void filling is probably due to increase in cement strength from reduced water/cement ratio.

Surface area -- In theory for concrete strength, it is accepted that bond strength (the strength at the aggregate/cement paste interface) is the weakest link of the composite. Thus, for a given bonding capacity between a cement and an aggregate, greater surface area results in more sites of weakness and lower composite strength.

The same Neal #2 results presented in Figures 22 and 23 were replotted (Figures 24 and 25) to illustrate the importance of a strength relationship. The tendency is for decreasing strength with increasing surface area, down to a relative surface area of 64 (30- to 50+ material). A slight and possibly insignificant increase in strength was observed at a relative surface area of 128 (50- to 100+ material).

Strength for the composite aggregate samples is also plotted in Figures 24 and 25 at its relative surface area of 13.8. It is encouraging that this fits into the patterns established by the

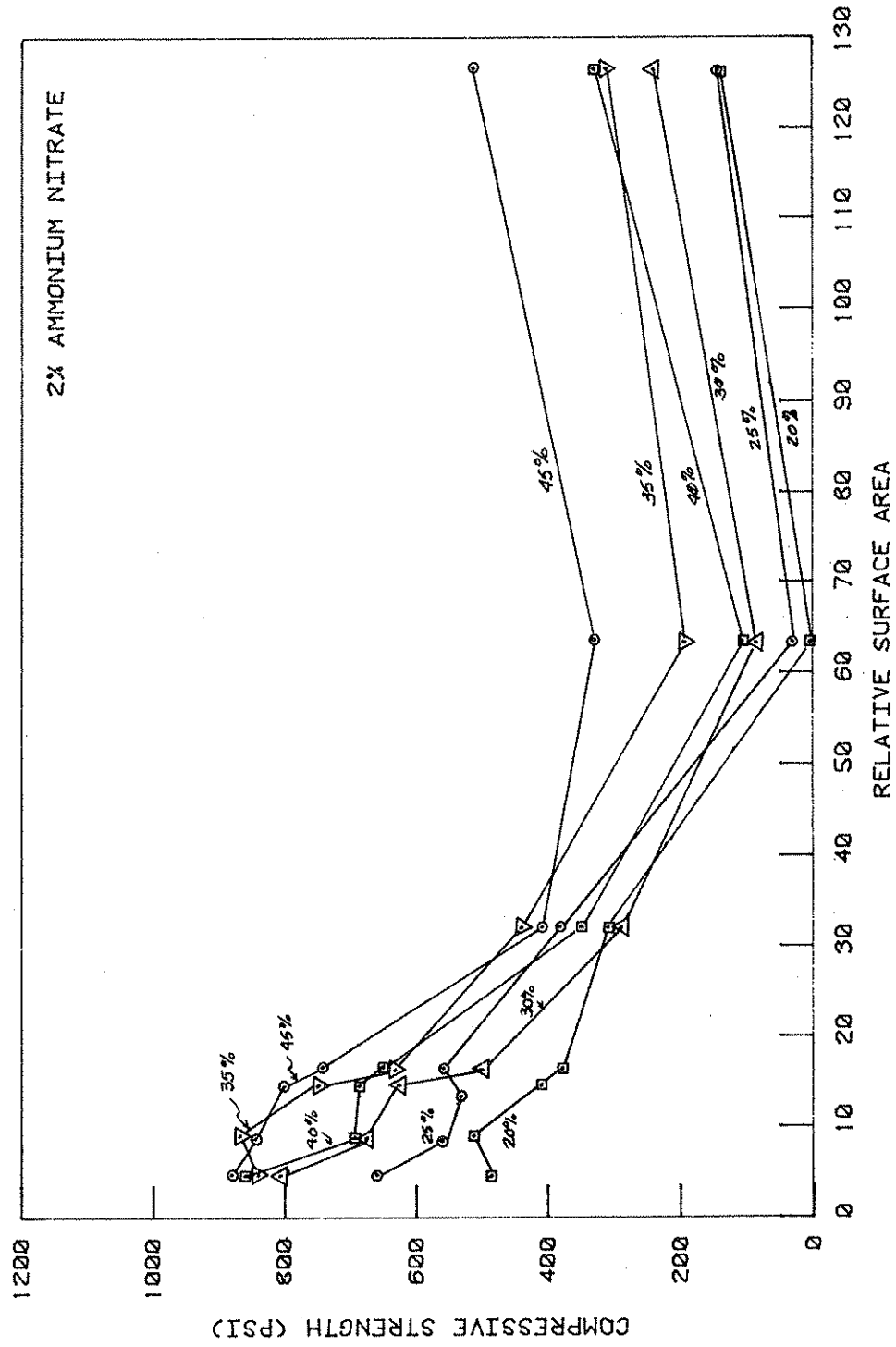


FIGURE 24. COMPRESSIVE STRENGTH VERSUS RELATIVE SURFACE AREA (NEAL #2)

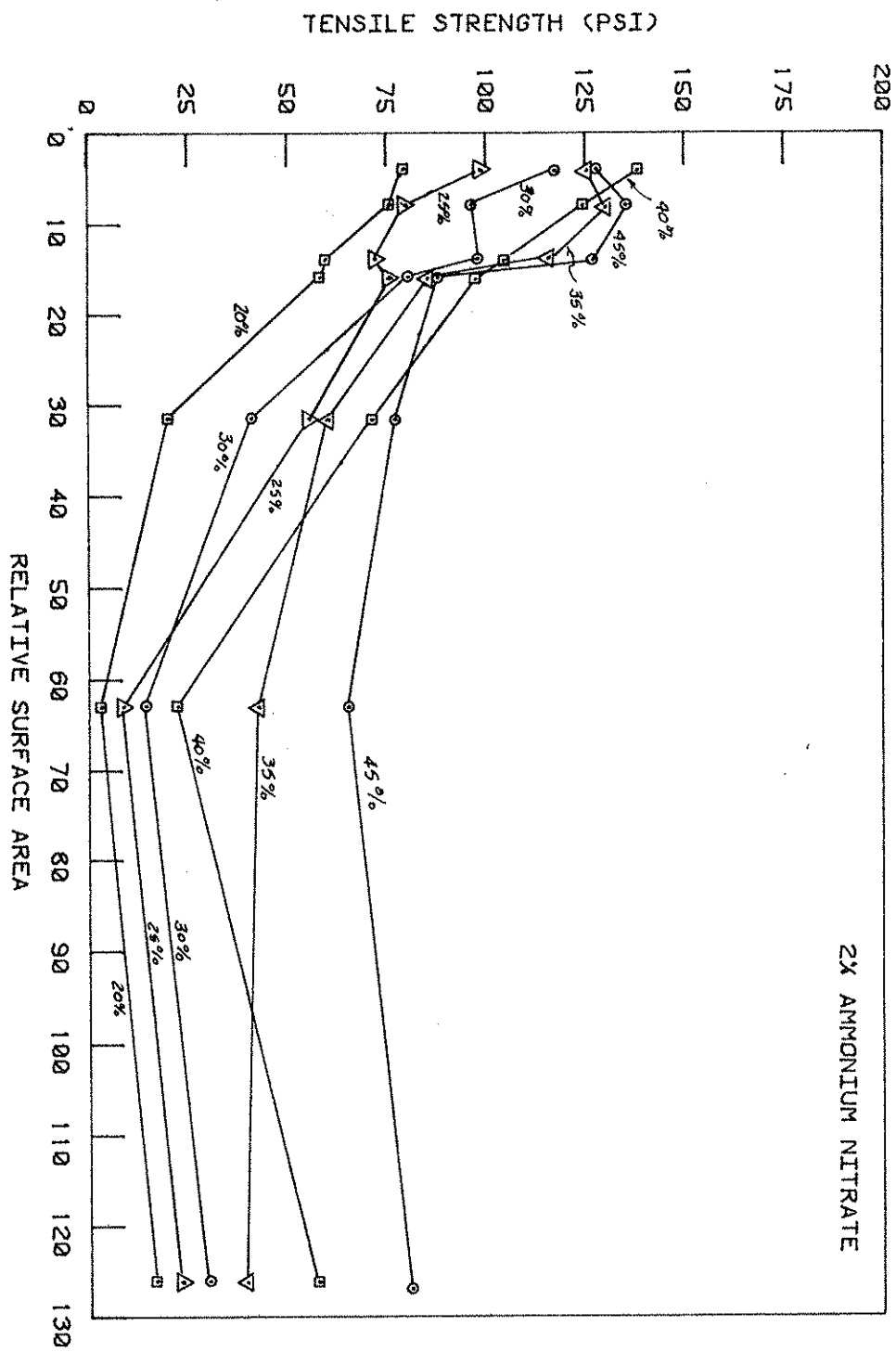


FIGURE 25. TENSILE STRENGTH VERSUS RELATIVE SURFACE AREA (CNEAL #2)

uniformly graded aggregate. Strength of cement stabilized granular materials appears to be sensitive to relative surface area. As relative surface area is easy to measure, it could serve as an important design parameter.

Consistency -- Because all mixes produced with the Neal #2 fly ash had the same consistency, or  $43 \pm 2$  seconds Vebe times, it is possible to consider the influence of water/cement ratio relative to fly ash content. Figure 26 shows an envelope representing the workable mixes for seven aggregate types. The fact that very little change in water/cement ratio with respect to cement content implies that a workable mix could be easily designed for practical water/cement ratios of 0.2 to 0.3.

Constant water/cement ratio -- After seeing potential in a surface area relationship at constant consistency, the Neal#4 fly ash was used to evaluate changes in strength relative to surface area with a constant strength cement. Figure 27 is the result which reinforces the significance of relative surface area as a design parameter. Obviously, high relative surface area aggregates should be avoided, even though some compensation may be possible with reduction in water cement ratio.

Frost action -- In evaluating the overall performance of fly ash as a construction material, it is important to consider its ability to withstand the rigors of freezing and thawing. In this research the procedure described by ASTM test method C 666-84, Method

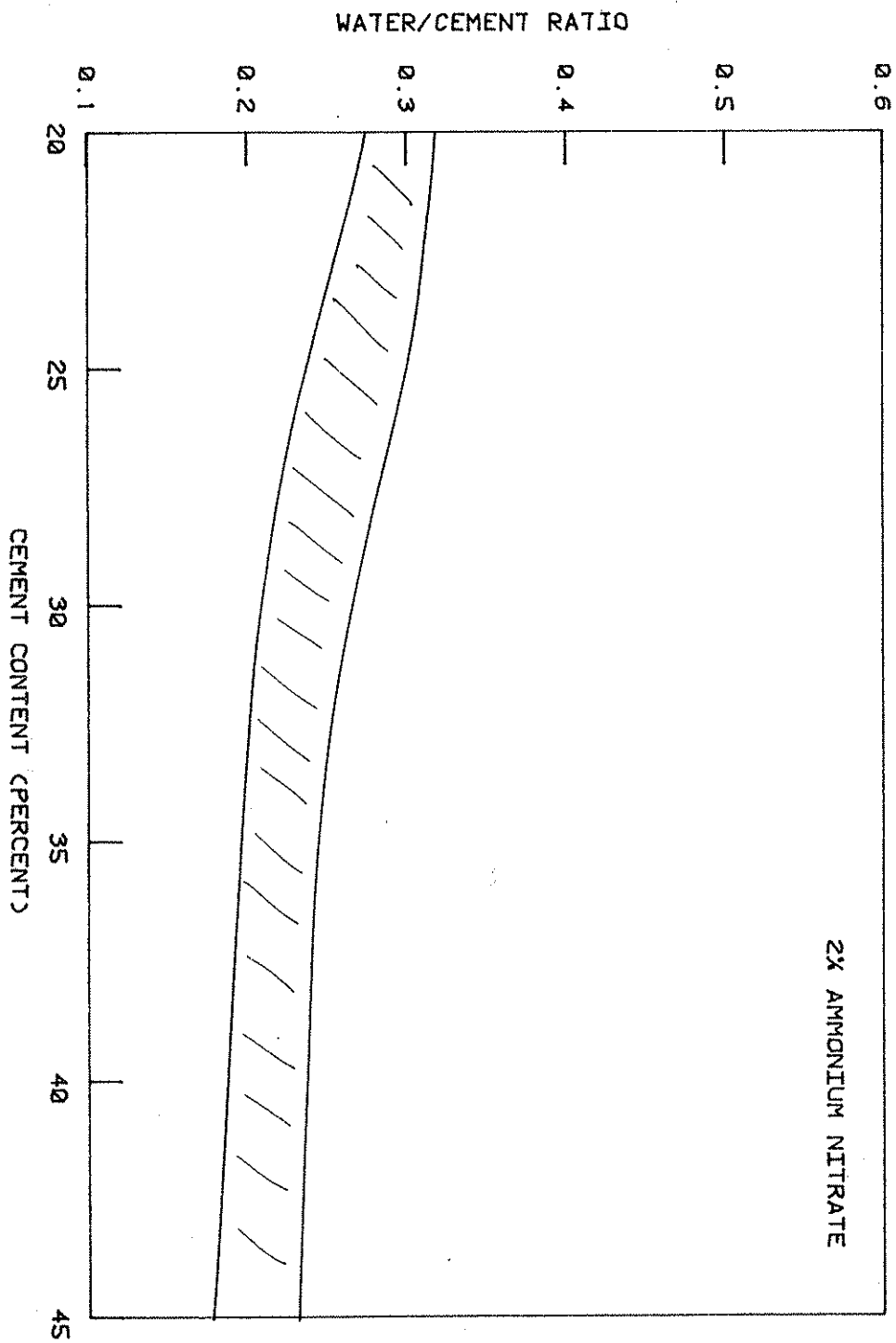


FIGURE 26. PLOT OF CONSTANT CONSISTENCY (VEBE TEST) (NEAL #2)



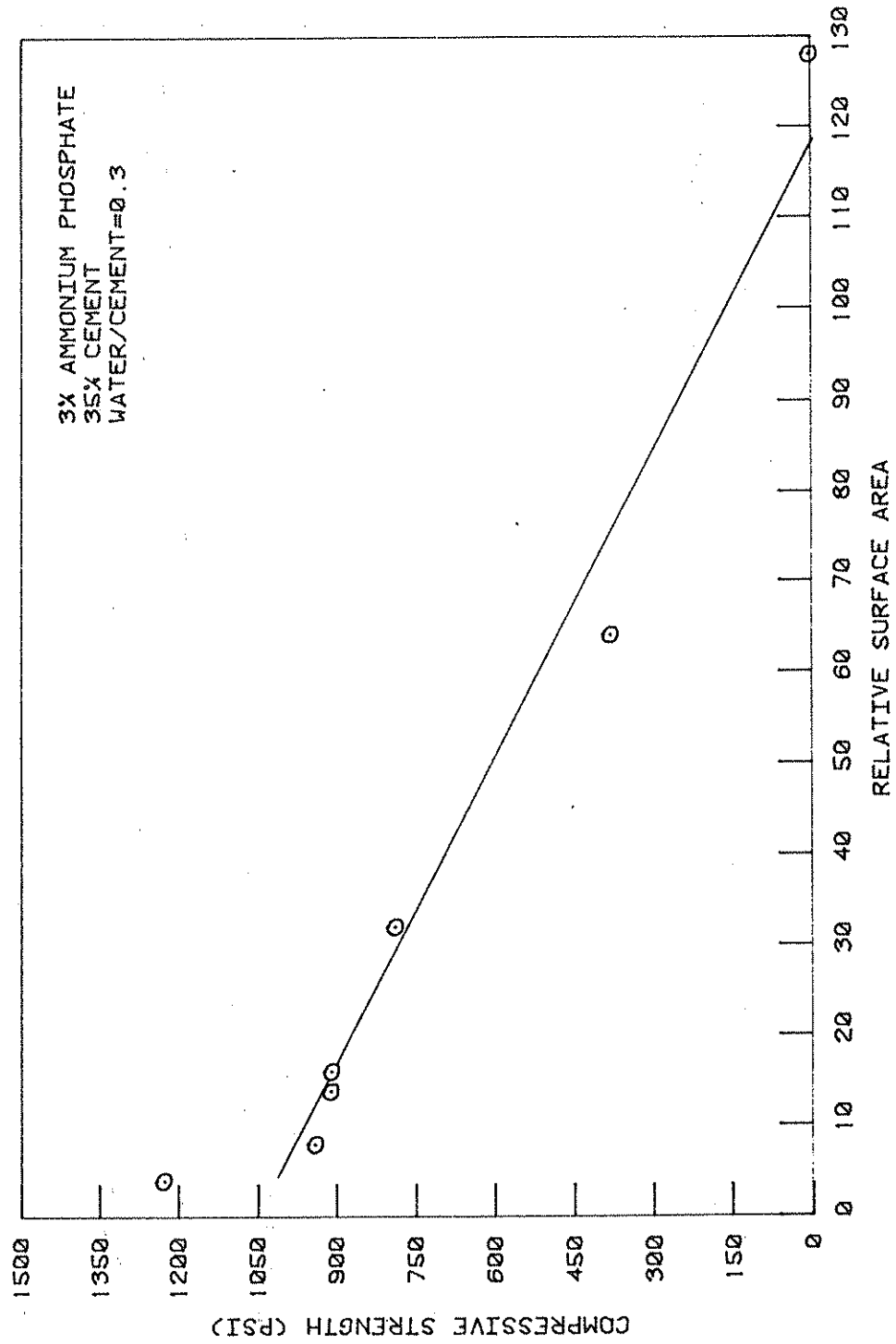


FIGURE 27. COMPRESSIVE STRENGTH VERSUS RELATIVE SURFACE AREA (NEAL #4)

A, was used to subject specimens to rapid freeze thaw action. This test, intended for concrete, is extreme in terms of the actual conditions existing for a stabilized base. Tests performed in this research are therefore a conservative measure of freeze-thaw performance. The results of the 12-cycle rapid freeze-thaw test (simulating an average Iowa winter) were measured in terms of residual compressive strengths and weight loss and are presented in Tables 15 (a) and (b).

Several criteria have been established for acceptable freeze-thaw performance of portland cement and lime-fly ash stabilized base courses. For example, ASTM C 593 requires a minimum 400 psi compressive strength after freeze-thaw. The Portland Cement Association criteria and AASHTO standards T 135-70 for freeze-thaw performance are based on weight loss limitations of 7 to 14 percent. Comparing these standards to the data in Table 15 (a) and (b) indicates that Neal #4 fly ash has little difficulty meeting all standards. However, none of the Neal #2 mixes, with the exception of the 45 percent cement mix, meets these established standards. Finally, there appears to be a correlation between initial strength and freeze-thaw performance. All samples with strengths 1200 to 1500 psi and higher met the minimum strength required by freeze-thaw specifications. This minimum initial compressive strength may be a key to design.

Shrinkage -- Shrinkage of portland cement stabilized subbases

Table 15. Freeze-thaw Performance.

## a. Neal #2

| Cement<br>(%) | Initial<br>Strength,<br>(psi) | Strength After<br>Freezing,<br>(psi) | Weight Loss,<br>(%) |
|---------------|-------------------------------|--------------------------------------|---------------------|
| 20            | 623                           | 0                                    | 59                  |
| 25            | 597                           | 92                                   | 25                  |
| 30            | 1126                          | 61                                   | 35                  |
| 35            | 1160                          | 271                                  | 21                  |
| 40            | 925                           | 110                                  | 37                  |
| 45            | 1190                          | 543                                  | 0                   |

## b. Neal #4

| Cement<br>(%) | Initial<br>Strength,<br>(psi) | Strength After<br>Freezing,<br>(psi) | Weight Loss,<br>(%) |
|---------------|-------------------------------|--------------------------------------|---------------------|
| 20            | 1502                          | 652                                  | 0                   |
| 25            | 1556                          | 1004                                 | 6                   |
| 30            | 2609                          | 1048                                 | 0                   |
| 35            | 1992                          | 1520                                 | 0                   |
| 40            | 2488                          | 1484                                 | 0                   |
| 45            | 2528                          | 1657                                 | 0                   |

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Table 16. Shrinkage Results, strain % x 10

| Cement Content, % | PC    | Neal #2 | Neal #4 |
|-------------------|-------|---------|---------|
| 100               | 351.0 | 98.2    | 34.7    |
| 20                | 66.8  | 15.6    | 35.2    |
| 25                | 75.3  | 17.8    | 37.3    |
| 30                | 77.9  | 31.1    | 29.8    |
| 35                | 120   | 35.6    | 32.5    |
| 40                | 109   | 24.6    | 59.6    |
| 45                | 81.1  | 48.9    | 70.7    |

has been acknowledged as a problem; for this reason, shrinkage evaluation for fly ash mixes was performed. Shrinkage samples were produced (ASTM C 157) for all composite aggregate mixes of Neal #2 and Neal #4 fly ashes evaluated in the strength study. Samples were allowed to cure in a humidity room for 14 days at 77° F, then were placed in a 100° F oven for 14 days. Measurements on all samples were taken every two days. In all cases, the 14-day cycle was sufficient time for stabilization of the specimen length.

Table 16 summarizes the results of fly ash and comparable portland cement concrete specimens. Cumulatively, fly ash demonstrated a factor of two reductions in shrinkage in comparison with portland cement. This reduction in shrinkage could be a significant improvement.

#### Other Fly Ash Cement Base

Lansing, Louisa and Ottumwa fly ashes were used to generalize relative surface area relationships and evaluate the influence of fly ash type on density (expressed as bulk specific gravity). These three fly ashes were selected to provide a range of cement strengths. Lansing fly ash with 2 percent ammonium phosphate yielded the strongest cement, while Louisa fly ash with 1 percent ammonium phosphate represented an intermediate strength, and Ottumwa fly ash without an additive yielded the lowest strength cement. Strengths of these cements for three different water/cement ratios are shown in Table 17.

Table 17. Cement Strength

| Fly Ash | 7-day Compressive Strength (psi) |      |      |
|---------|----------------------------------|------|------|
|         | Water/Cement Ratio               |      |      |
|         | 0.18                             | 0.23 | 0.28 |
| Lansing | 1740                             | 1650 | 1025 |
| Lousia  | 380                              | 395  | 295  |
| Ottumwa | 190                              | 185  | 120  |

Relative Surface Area -- The results shown in Figures 28 through 30 show relative surface area is significant to stabilized base strength; however, such factors as cement strength, water/cement ratio, and cement content also play an important role. From this study it can be seen that high strength cements offer significantly stronger bases until the relative surface area reaches 80 or above; thereafter, similar strengths can be realized for like cement contents regardless of cement source.

Cement content also plays an important role in stabilized base strength with respect to relative surface area. As might be expected, more cement allows use of finer material.

A last consideration is water/cement ratio. Compressive strengths for bases from high-strength cements are very sensitive to water/cement ratio and construction would produce best results on the wet side of an optimum water/cement ratio. For the weakest cement

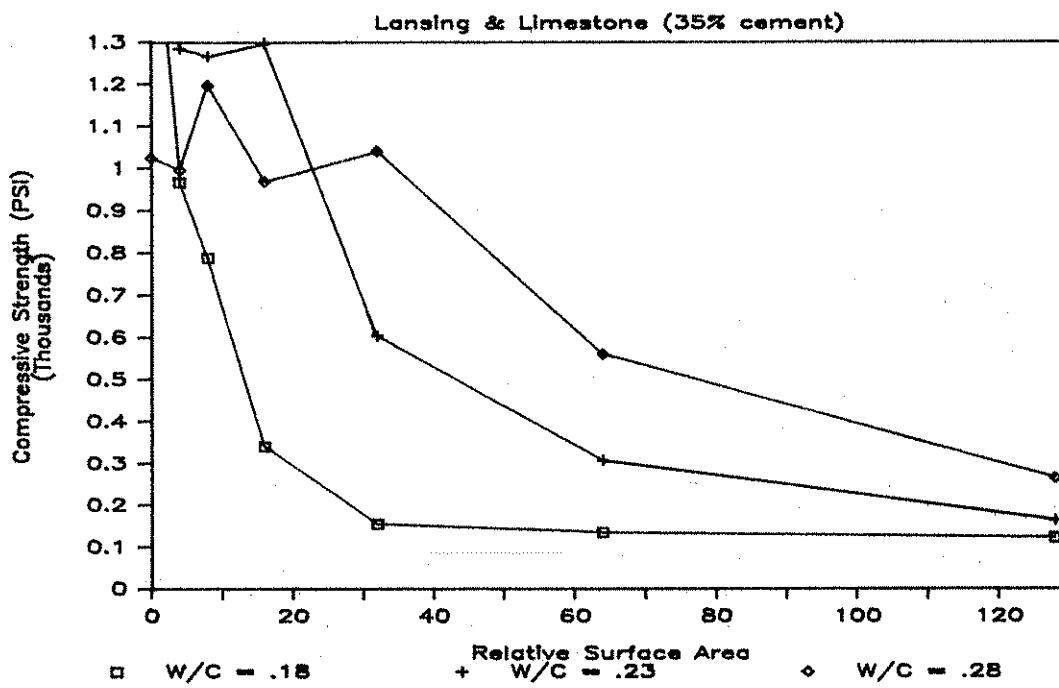
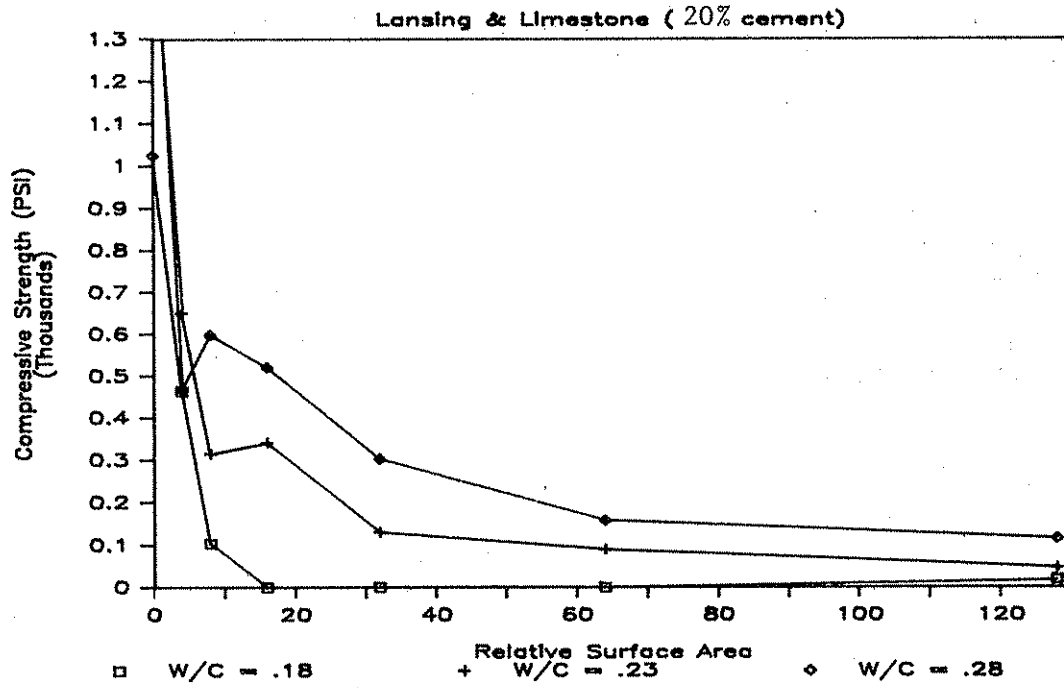


Figure 28. Compressive strength versus relative surface area (Lansing).

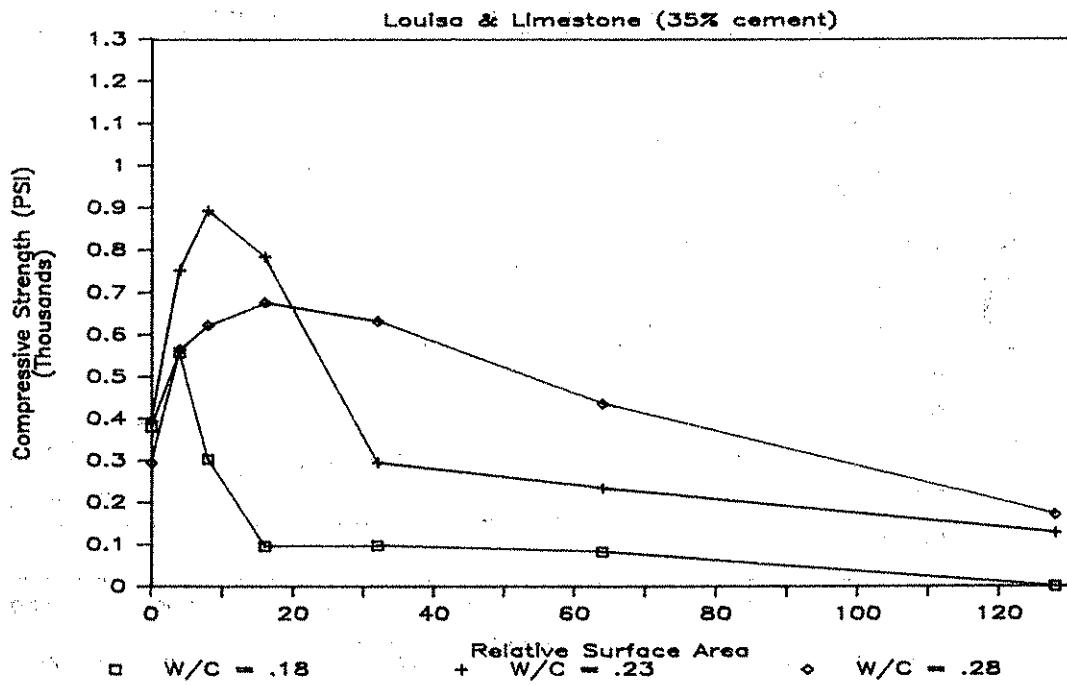
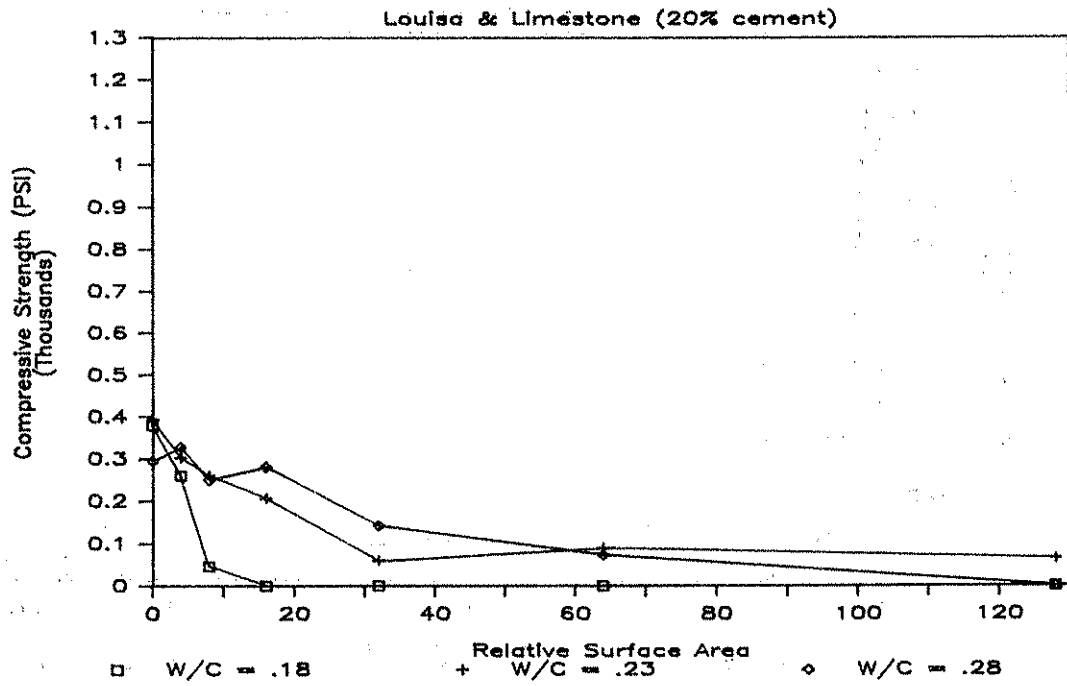


Figure 29. Compressive strength versus relative surface area (LOUISA).

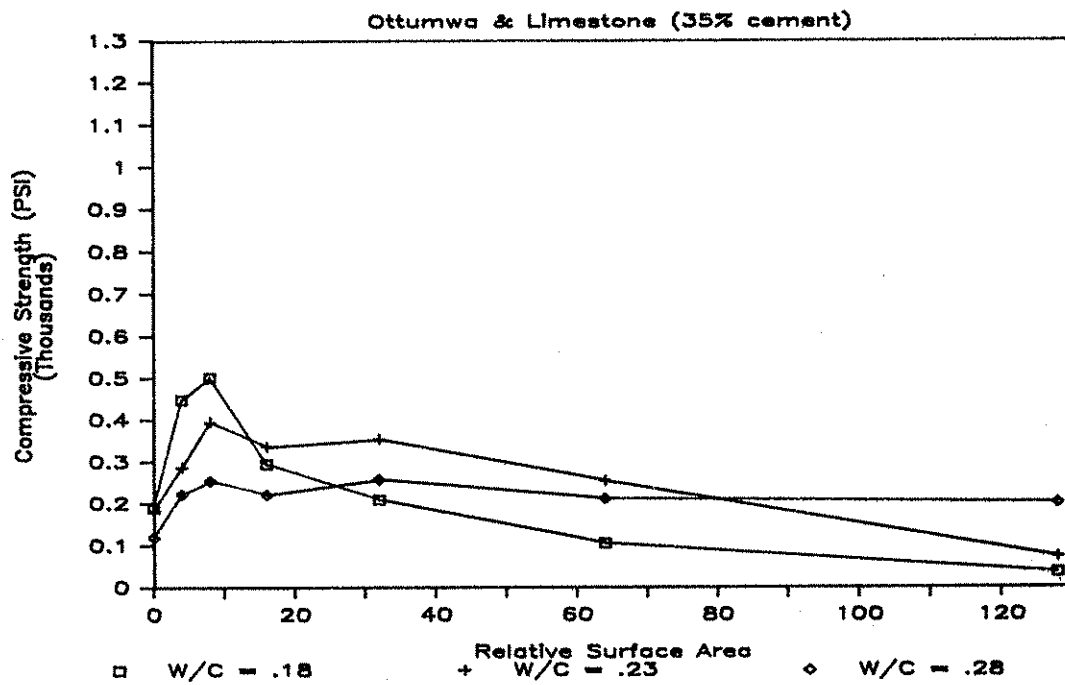
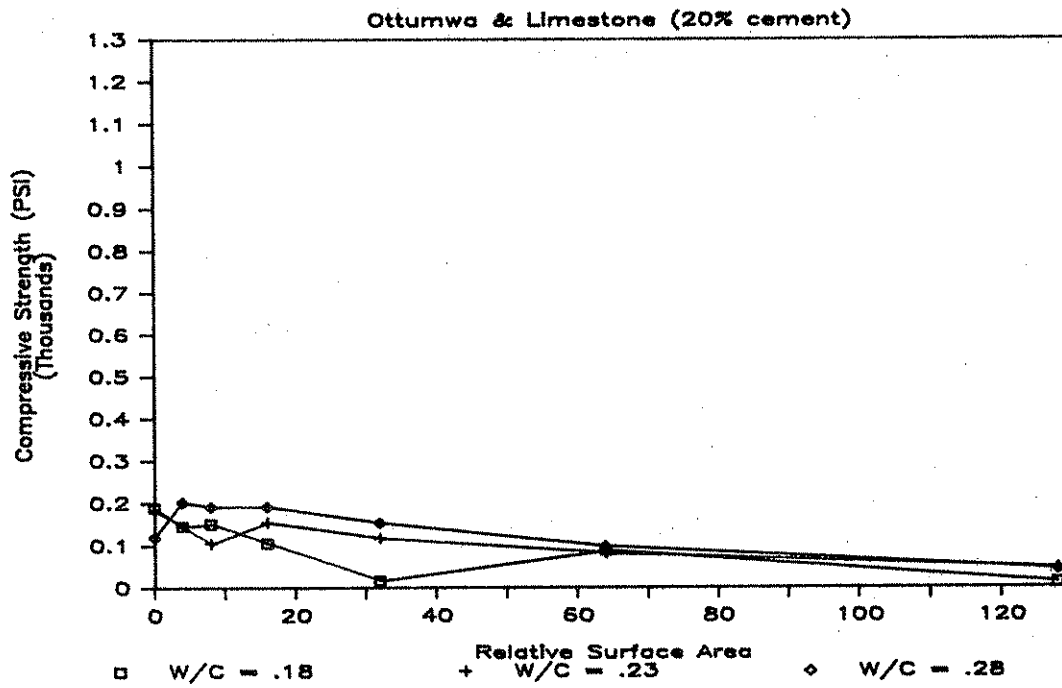


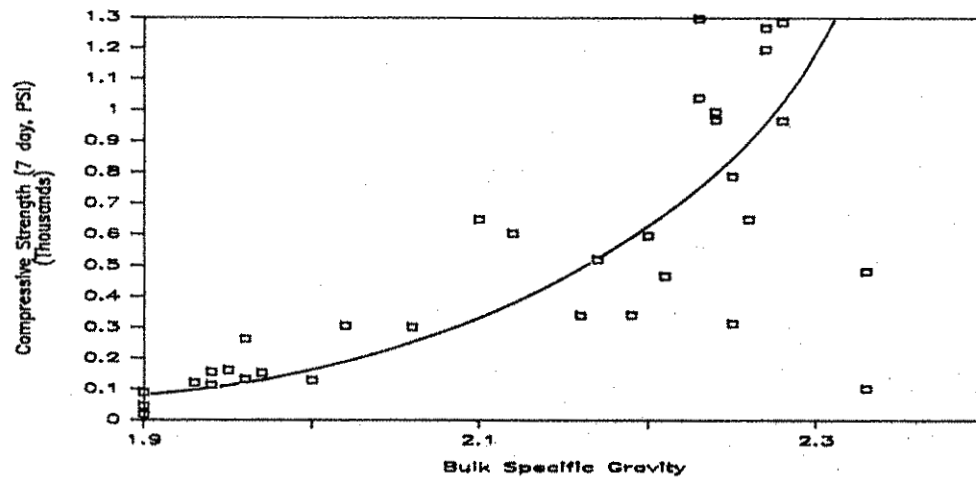
Figure 30. Compressive strength versus relative surface area (OTTUMWA).



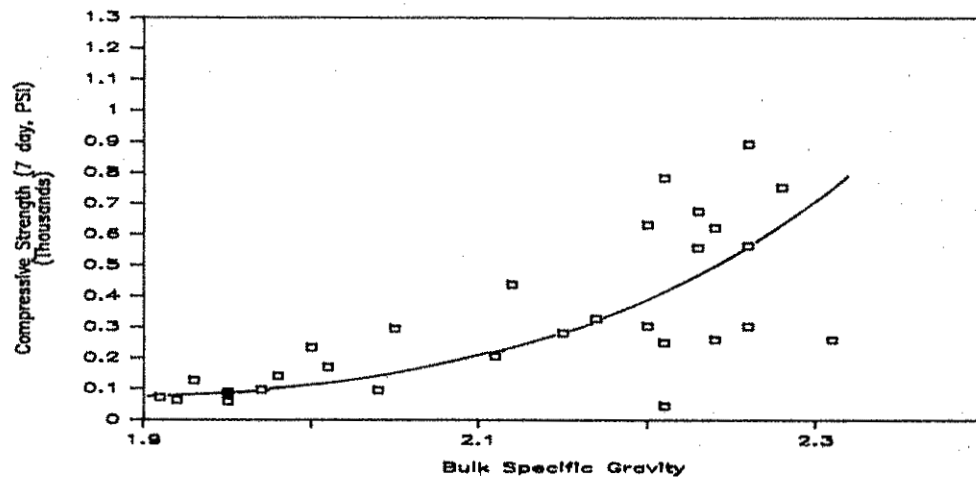
(Ottumwa), stabilized bases would not be particularly strong but quality control should be easy.

Bulk specific gravity -- Unit weight or bulk specific gravity is an important quality control parameter; therefore, it is evaluated in the context of strength for the three fly ash sources. Figure 31 shows how sensitivity between strength and bulk specific gravity increases with strength of the cement. Stronger base courses are possible with stronger cements of course, but the compaction process must be more closely supervised.

## LANSING



## LOUISA



## OTTUMWA

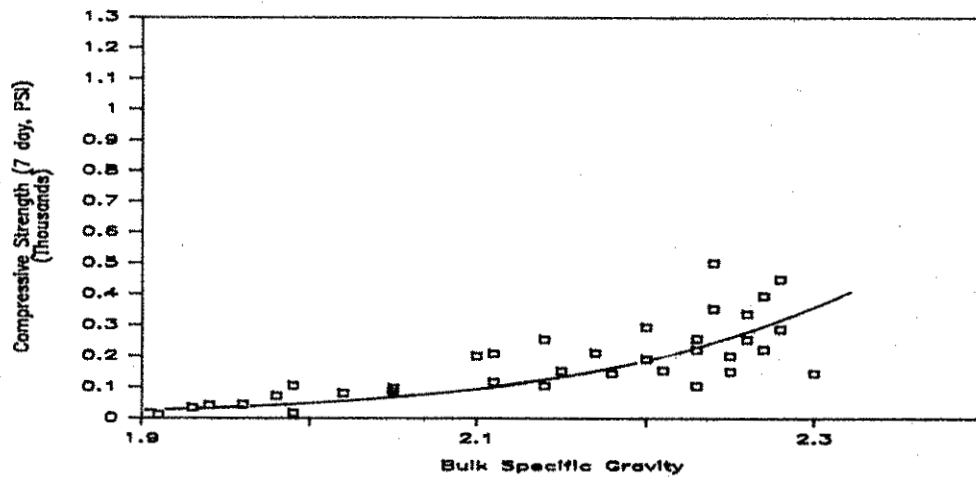


Figure 31. Compressive strength versus bulk specific gravity for Lansing, Louisa, and Ottumwa fly ashes.

## SOIL STABILIZATION

The soil stabilization phase of this research was conducted to determine how the addition of fly ash increases strength in different types of natural soils, enabling them to function as a base or a subbase. Goals were to increase strength and density, and decrease plasticity; freeze-thaw resistance was also measured.

Nine combinations of soils (categorized by their AASHTO designations) were tested to measure tensile strength, compressive strength and density over a range of moisture contents.

### Tensile Strength

Ames fly ash with 2 percent ammonium phosphate additive was used; 2 inch diameter by 1 inch long cylindrical specimens were compacted by a 10 pound drop hammer, 5 blows on each end. Specimens were moist cured for 7 days, with tensile strength determined according to ASTM C 496.

Four soils, namely Hallet Coarse Sand (HCS), Peterson Pit Sand (PPS), a clayey silt (glacial till sampled north of Ames) and a commercial bentonite, were used to blend nine soils as shown in Table 18. Particle size distributions for the same soils are shown in Table 19. Classification results of the soil blends are reported in Table 20. The nine soils were grouped into three categories: Blend #1, Blend #2 and Blend #3 are clayey soils; Blend #4, Blend #5,

Table 18. Composite Blends

| Blend # | Hallet<br>Course<br>Sand (HCS) | Clayey<br>Silt<br>(Ames) | Petersons'<br>Pit Sand<br>(PPS) | Bentonite |
|---------|--------------------------------|--------------------------|---------------------------------|-----------|
| 1       | 0%                             | 100%                     | 0%                              | 0%        |
| 2       | 0%                             | 90%                      | 0%                              | 10%       |
| 3       | 0%                             | 80%                      | 0%                              | 20%       |
| 4       | 30%                            | 50%                      | 20%                             | 0%        |
| 5       | 30%                            | 40%                      | 20%                             | 10%       |
| 6       | 30%                            | 30%                      | 20%                             | 20%       |
| 7       | 50%                            | 20%                      | 30%                             | 0%        |
| 8       | 50%                            | 10%                      | 30%                             | 10%       |
| 9       | 20%                            | 30%                      | 50%                             | 0%        |

Table 19. Particle-size Analysis (Percent Passing)

| Sieve-Size | Petersons'<br>Pit Sand | Hallet<br>Course<br>Sand | Clayey<br>Silt | Bentonite |
|------------|------------------------|--------------------------|----------------|-----------|
| 3/8"       | 100                    | 100                      | 100            | 100       |
| 4          | 100                    | 100                      | 100            | 100       |
| 10         | 99.8                   | 76.7                     | 100            | 100       |
| 20         | 94.7                   | 50.8                     | 94.6           | 100       |
| 40         | 38.9                   | 21.9                     | 85.4           | 100       |
| 60         | 11.5                   | 5.2                      | 74.2           | 100       |
| 100        | 2.8                    | 0.6                      | 63.4           | 100       |
| 200        | 1.0                    | 0.2                      | 54.5           | 100       |

Table 20. AASHTO Classification of Soil Blends.

| Blend # | Percent Passing |    |     | LL  | PL | PI  | AASHTO           |                |
|---------|-----------------|----|-----|-----|----|-----|------------------|----------------|
|         | Sieve Size      |    |     |     |    |     |                  | Classification |
|         | 10              | 40 | 200 |     |    |     |                  |                |
| 1       | 100             | 85 | 55  | 25  | 12 | 11  | A-4, Clayey Soil |                |
| 2       | 100             | 87 | 59  | 49  | 18 | 31  | A-7-5, " "       |                |
| 3       | 100             | 88 | 64  | 106 | 22 | 84  | A-7-5, " "       |                |
| 4       | 93              | 57 | 28  | 20  | 4  | 16  | A-2-6, Cly. Sand |                |
| 5       | 93              | 59 | 32  | 72  | 16 | 56  | A-2-7, Cly. Sand |                |
| 6       | 93              | 60 | 37  | 120 | 20 | 100 | A-7-5, Cly. Sand |                |
| 7       | 88              | 40 | 11  | NP  |    |     | A-1-b, Cor. Sand |                |
| 8       | 88              | 41 | 16  | 50  | 10 | 40  | A-2-7, Cly. Sand |                |
| 9       | 95              | 50 | 17  | 17  | 16 | 1   | A-1-b, Cor. Sand |                |

Table 21. Modification of Soil Plasticity by Fly Ash

| Blend # | Plain Soil |    |     | Soil + FA |    |    | Soil + FA + AP |    |    |
|---------|------------|----|-----|-----------|----|----|----------------|----|----|
|         | LL         | PL | PI  | LL        | PL | PI | LL             | PL | PI |
| 1       | 25         | 12 | 11  | 27        | 21 | 6  | 29             | 22 | 7  |
| 2       | 49         | 18 | 31  | 38        | 21 | 17 | 39             | 22 | 17 |
| 3       | 106        | 22 | 84  | 56        | 22 | 38 | 72             | 34 | 38 |
| 4       | 20         | 4  | 16  | 19        | 18 | 1  | 23             | 20 | 3  |
| 5       | 72         | 16 | 56  | 29        | 23 | 6  | 43             | 23 | 10 |
| 6       | 120        | 20 | 100 | 42        | 20 | 22 | 68             | 19 | 49 |
| 7       | 0          | 0  | 0   | 0         | 0  | 0  | 0              | 0  | 0  |
| 8       | 50         | 10 | 40  | 22        | 20 | 2  | 22             | 18 | 4  |
| 9       | 17         | 16 | 1   | 0         | 0  | 0  | 0              | 0  | 0  |

## NOTE:

LL = Liquid Limit; PL = Plastic Limit; PI = Plasticity Index

FA = Fly Ash; AP = Ammonium Phosphate

Blend #6, and Blend #8 are silty to clayey sands; Blend #7 and Blend #9 are sandy and gravelly soils.

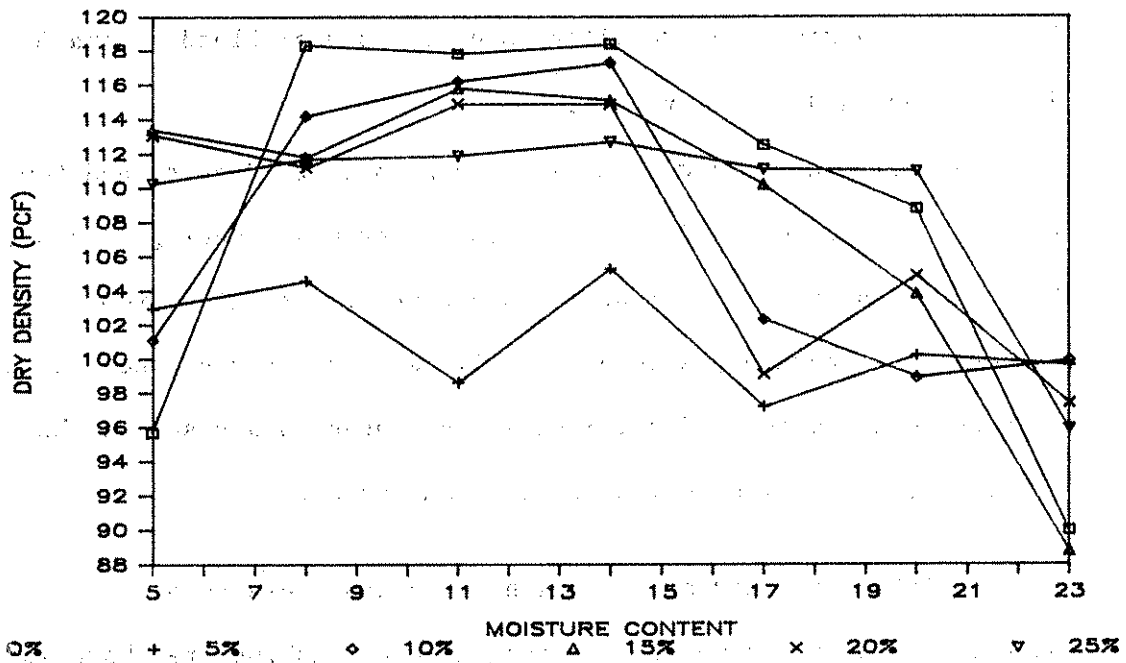
Relationships between moisture content, dry density and tensile strength were determined. Six moisture contents (ranging from 5 to 23 percent) at five different fly ash contents (ranging from 5 percent to 25 percent) were used. Fly ash content was based on the percentage dry weight of soil; moisture content was based on the percentage of combined weight of fly ash and soil.

Moisture-density and strength -- Typical results of moisture-density and moisture-strength relationships are shown in Figure 32. The complete set of data is presented in Appendix E.

The most significant observation from these tests is that the addition of chemically modified fly ash significantly increased soil strength. Earlier studies on Neal #4 cement indicated that tensile strength is approximately 15 percent of compressive strength. Therefore, it may be deduced that compressive strength will also increase with the addition of fly ash. A brief summary of fly ash cement performance with all the nine soil blends is as follows :

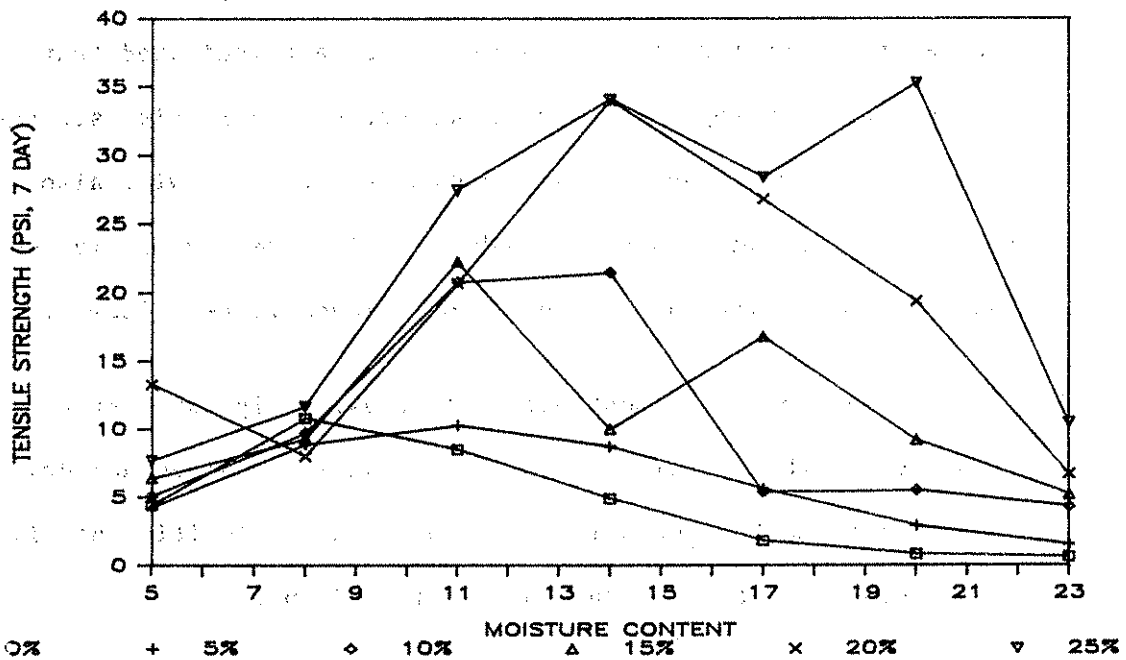
Blend #1 -- A-6, clay, 0% bentonite. Increasing fly ash content increases strength, but also increases the water requirement; dry density is reduced by increasing fly ash content. Addition of fly ash increased tensile strength from 5 psi to 35 psi.

Blend #2 -- A-7-5, clay, 10% bentonite. Fly ash increases the



NOTE: Symbols indicate percent fly ash.

BLEND #1: Graph (A)--Dry density versus moisture content.



NOTE: Symbols indicate percent fly ash.

BLEND #1: Graph (B)--Tensile strength versus moisture content.

Figure 32. Relationships of dry density (A) and tensile strength (B) to moisture content in Blend #1.

strength from 5 psi to 30 psi. Increasing moisture content reduces dry density; however, increasing fly ash content did not reduce density.

Blend #3 -- A-7-5, clay, 20% bentonite. Addition of fly ash increased the strength from 7 psi to 40 psi. No noticeable change in dry density was observed with the addition of fly ash.

Blend #4 -- A-2-6, clayey sand, 0% bentonite. Strength increased from 5 psi to more than 50 psi at 11 percent water content; however strength was observed to be very sensitive to moisture content. Strength drops dramatically below and above the optimum moisture content, and at a moisture content of 20 percent and above, strength reduces to zero. Dry density did not vary significantly with respect to moisture content, although the densities were maximum at the moisture content of 11 percent.

Blend #5 -- A-2-7, clayey sand, 10% bentonite. Strength increased from 4 psi to a maximum of 37 psi, and the increase in strength was proportional to the amount of fly ash added. Maximum dry densities were observed at a moisture content of 11 percent, after which density decreased rapidly; higher densities were found with high fly ash contents, however. Maximum strength increase was observed between 11 and 17 percent moisture content.

Blend #6 -- A-7-5, clayey sand, 20% bentonite. Increasing fly ash content greatly increases strength, from 1 psi to about 85 psi; but



strength was extremely sensitive to the moisture content. Maximum dry density was found at 11 percent moisture content. Above and below 11 percent moisture content strength dropped sharply; at 17 percent moisture content and above strength reduced to zero. Again, increasing fly ash concentration also increased density.

Blend #7 -- A-1-b, coarse sand, 0% bentonite. Strength increased with increasing fly ash concentrations. The maximum strength increase (from 3 psi to 58 psi) was obtained at a fly ash content of 25 percent and 11 percent moisture content. Strength was more sensitive to moisture content at higher fly ash content. The maximum density was obtained at 11 percent moisture content. Higher densities were obtained for the soil with higher fly ash contents.

Blend #8 -- A-2-7, clayey sand, 10% bentonite. Strength increased with, and was extremely sensitive to moisture content. The maximum strength increase was from 2 psi to 83 psi for 25 percent fly ash content at a moisture content of 11 percent. Soils containing lower amounts of fly ash (0, 5 and 10 percent), exhibited no strength above a 14 percent moisture content. Strength of soils containing higher amounts of fly ash (15, 20 and 25 percent), reduced to zero at 17 percent moisture content. Maximum dry density occurred at a moisture content of 11 percent; density did not vary significantly with the fly ash content.

Blend #9 -- A-1-b, coarse sand, 0% bentonite. Tensile strengths increased with increasing fly ash contents; and at 25 percent fly ash

content maximum strength increase (from 3 psi to 64 psi) occurred at a moisture content of 11 percent. Strength and density responses to the moisture content were similar to those observed in Blend #8.

The most important finding in this part of the research is that Ames fly ash can greatly increase the strength of a broad range of soils when used with a 2 percent ammonium phosphate additive. In general, maximum density was observed around 11 percent moisture content. The role of moisture content is extremely important, in that above or below the optimum, water reduces strength. While strength generally increased with fly ash content, the most significant strength increases were observed when fly ash contents exceeded 10 percent. Density also increased with increasing fly ash content, with the exception of Blend #1; a finding contrary to that observed in limestone stabilized base courses.

Clayey soils (Blends #1, #2 and #3) showed very low strength gains, so a large quantity of fly ash (more than 20 percent) was needed. Increasing quantity of bentonite decreased strength and density, probably because it increased surface area.

The second soil category (clayey or silty sand, comprising Blends #4, #5, #6 and #8) showed a much better response for both strength gain and density. Strengths in the range of 85 psi to 90 psi and densities of up to 130 pcf were obtained. This type of soil is very sensitive to moisture content, and it was observed that water exceeding the optimum by as little 3 percent may totally

diminish strength. Twenty percent fly ash is required to produce a significant strength gain; however strength for this group of soils is twice that for clayey soils, using the same amount of fly ash.

The third soil group (coarse sand, Blends #7 and #9), was expected to show the highest strength gain, but did not. Densities were similar to those found in clayey/silty sands; strength gain was slightly lower. Higher strength gains in finer grained soils (as compared to granular soils) may indicate that some type of chemical reaction is taking place between the soil and fly ash.

#### Soil Modification

Investigations beyond strength tests were conducted to observe how fly ash (with and without ammonium phosphate) can modify soil to reduce the plasticity index. The test included measuring the liquid and plastic limits for all the soil blends; i.e., no fly ash, plain fly ash and chemically modified fly ash.

Results presented in Table 21 and Figure 33 show that the addition of fly ash significantly reduced the liquid limit and increased the plastic limit for most of these soils, resulting in a reduced plasticity index. Intriguingly, plain fly ash was found more effective in reducing soil plasticity than ammonium phosphate treated fly ash.

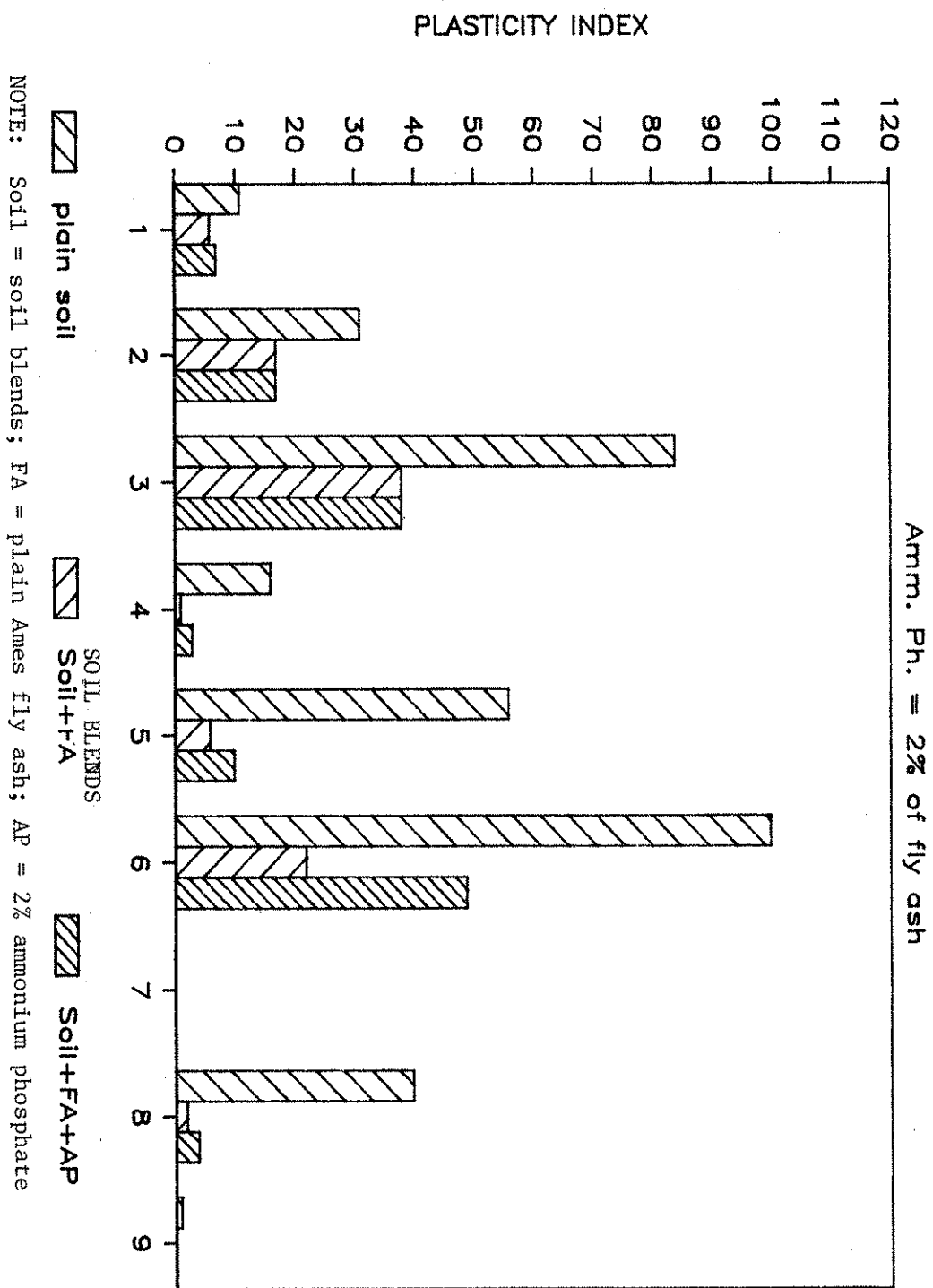


Figure 33. Plasticity index versus soil mixes for Blends #1-9.

### Compressive Strength

A study of compressive strength was conducted with soil Blends #1 through #3 and #5 through #7. Two proctor samples per mix were molded at the optimum moisture content, then 7 day compressive strength was determined. Two fly ash contents (10 and 20 percent by soil weight) were used with an additive of 0.5 percent ammonium phosphate.

Moisture-density-strength -- Unconfined compression tests are shown in Table 21. Very little change in density was due to the change in fly ash concentration; however, density depended upon soil type. Blends #7A and #7B, sandy soils, showed highest density. Finer grained soils such as Blends #2, #3 and #6, showed lower density.

All soil blends showed strength to increase as fly ash content increased from 10 to 20 percent, with the highest strength increase obtained in Blend #1. Blend #7 showed much lower strength than Blend #1; since Blend #1 is a clayey silt and Blend #7 is a sandy soil having a lower relative surface area (thus being expected to show a higher strength) this finding was rather unexpected. As observed earlier, some form of chemical reaction may be responsible.

These findings suggest the relationship between relative surface area and strength previously observed for crushed limestone may not hold true for finer grained soils. It is known that soil plasticity and finer fractions of soil particles (such as

Table 22. Compressive Strength of Soil Blends

| Blend # | Optimum<br>Moisture<br>Content (%) | Dry Density<br>(pcf) | Compressive<br>Strength (psi) |
|---------|------------------------------------|----------------------|-------------------------------|
| 1A      | 11                                 | 120                  | 108                           |
| 1B      | 11                                 | 119                  | 177                           |
| 2A      | 9                                  | 109                  | 93                            |
| 2B      | 9                                  | 109                  | 142                           |
| 3A      | 9                                  | 104                  | 60                            |
| 3B      | 9                                  | 105                  | 103                           |
| 5A      | 12                                 | 118                  | 63                            |
| 5B      | 12                                 | 117                  | 117                           |
| 6A      | 9                                  | 109                  | 38                            |
| 6B      | 9                                  | 111                  | 40                            |
| 7A      | 11                                 | 127                  | 30                            |
| 7B      | 11                                 | 125                  | 118                           |

Note :

A = 10 percent Ames fly ash with 0.5 percent ammonium phosphate

B = 20 percent Ames fly ash with 0.5 percent ammonium phosphate

the percent passing through the #200 sieve) play an important role in soil stabilization; therefore, an attempt was made to relate soil strength to these two parameters.

At maximum strength, an optimum concentration of particles passes through the #200 sieve (see Figure 34); this concentration was determined to be 50 to 60 percent. This knowledge may serve as an indicator of stabilization potential for a particular soil.

Figure 35 shows that soil strength drops as the plasticity index increases, which may serve as an additional indicator of a given soil's stabilization potential.

Wet-dry and freeze-thaw durability tests -- To investigate the wet-dry and freeze-thaw characteristics of the soil samples, tests were conducted in accordance with ASTM Standards D 559-82 and D 560-82.

In the wet-dry tests (ASTM D 559-82), specimens are molded in a proctor mold and then placed in the moisture room for 7 days, after which they were submerged in water for 5 hours. Following immersion they are placed in an oven at 160° F for a period of 42 hours. The 48 hour process of wetting and drying constitutes one cycle; the test consists of 12 such cycles. At the end of each cycle two firm strokes are applied by a wire brush and the soil loss measured.

In the freeze-thaw tests (ASTM D 560-82), specimens are molded in the proctor mold and placed in the moisture chamber for 7 days, after which they are placed in a freezing chamber at -10° F for 24

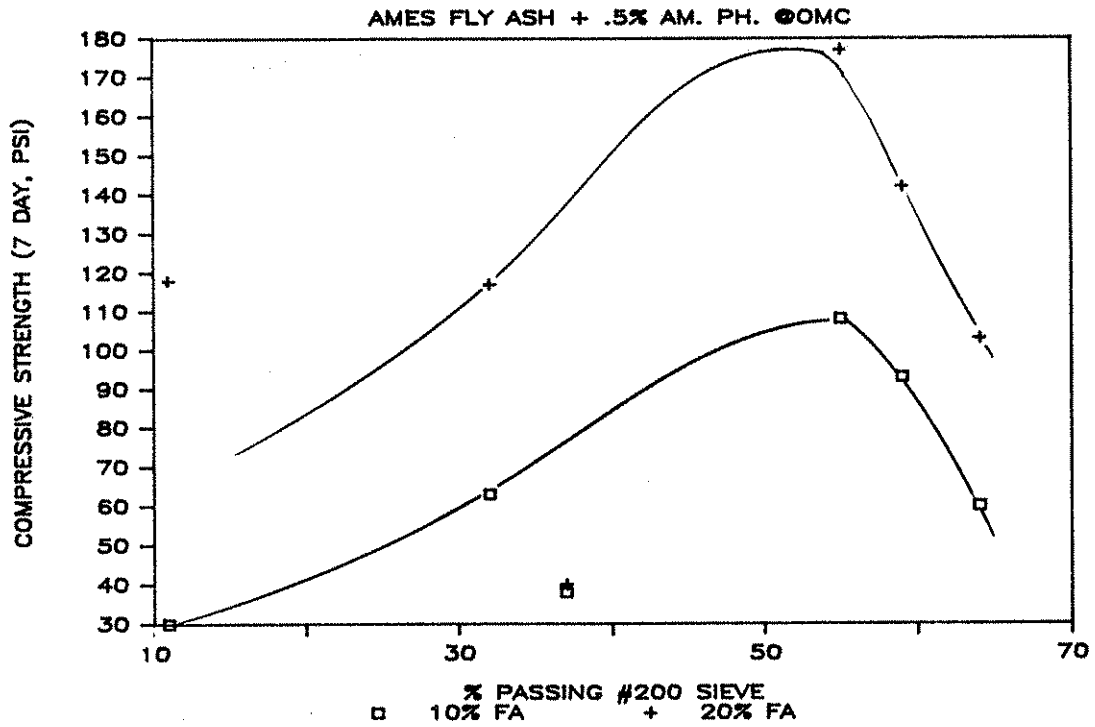


Figure 34. Compressive strength versus percent passing #200 sieve.  
 NOTE: FA = Ames fly ash with 0.5% ammonium phosphate.

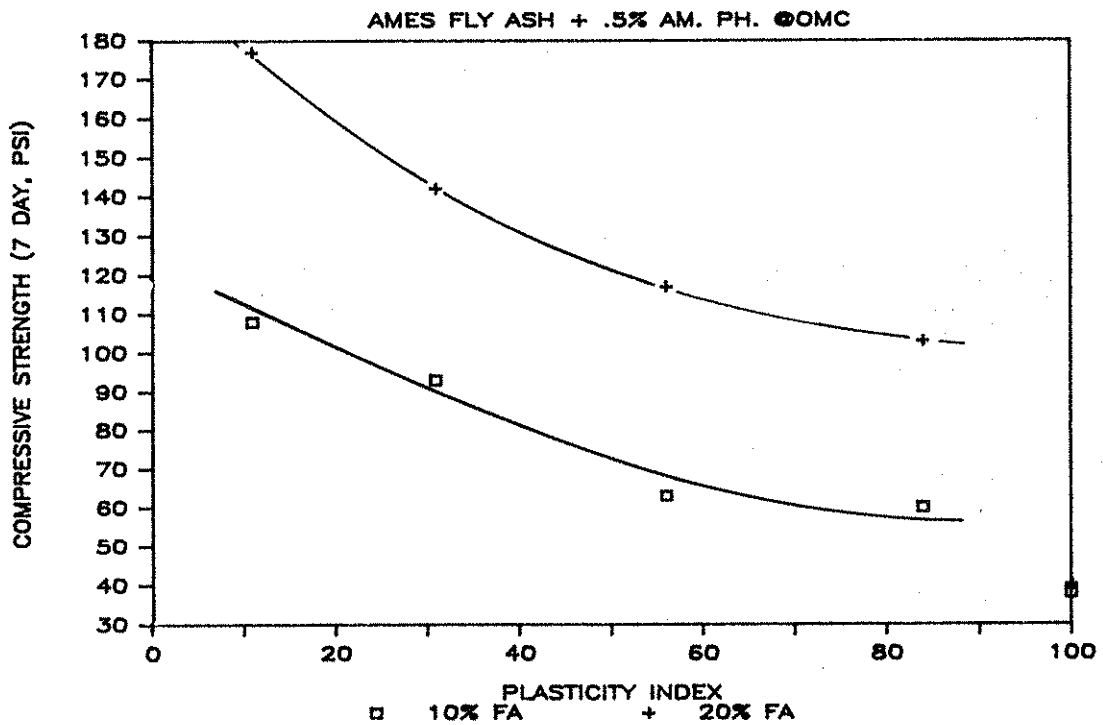


Figure 35. Compressive strength versus plasticity index. NOTE: FA = Ames fly ash with 0.5% ammonium phosphate.



hours. Freezing is followed by a period of 23 hours thawing in a humidity room having 100 percent relative humidity at 70° F. Periods of freezing and thawing constituted one cycle (48 hours) and the test consisted of 12 cycles. At the end of each cycle two firm strokes were applied with a wire brush and the soil losses measured.

Four soil samples containing fly ash were made in the automatic proctor machine for the six soil blends; also another set of soil samples was prepared using no fly ash; this latter set serving as the control. These samples were subjected to the freeze-thaw and wet-dry tests already described.

Freeze-thaw and wet-dry tests are extreme in nature; all soils became too soft and fell apart within the first cycle or immediately afterward.

This somewhat disappointing test result was largely expected. While the extremity of the test conditions can seldom be expected in the field, they nevertheless demonstrate that fine grained soils cannot withstand freeze-thaw and wet-dry conditions as well as limestone crusher fines.

## EQUIVALENT STRENGTH

Purpose and Scope

This part of the research was designed to compare the cost effectiveness of ammonium phosphate treated fly ash (APFA) stabilization with that from more conventional cements. To allow objective measurement, APFA stabilized strengths for three different soil types were correlated to costs for conventional stabilizers in quantities required to produce equivalent strengths. Ames fly ash with 0.5 percent ammonium phosphate was compared to Type I portland cement, portland cement with Ames fly ash, and kiln dust with Ames fly ash. Composition and quantities of cements are in Table 23.

The three soils used for this evaluation were blended from soils described in the previous section and are as follows:

Soil A - 100% clayey silt

Soil B - 20% clayey silt + 30% Petersons' pit sand +  
50% Hallet coarse sand

Soil C - 90% clayey silt + 10% bentonite

Procedure

The first step in the evaluation was to develop moisture-density and moisture-strength relationships from standard Proctor tests (ASTM D 698). This included three soils and fourteen variations in stabilizer and stabilizer concentration. Typical data (in this case for APFA) from which optimum moisture content and the associated strength were determined are shown in Figure 36. Table 24 is a

Table 23. Stabilizer Cement Composition

| <u>Cement</u>                | <u>Percent By Weight of Soil</u> <sup>a</sup> |
|------------------------------|---|
| APFA                         | 20 & 30                                       |
| Portland Cement              | 1, 5, 7, 11                                   |
| Portland Cement &<br>Fly Ash | 3+9, 4+12, 5+15, 6+18                         |
| Kiln Dust &<br>Fly Ash       | 2+2, 7+7, 9+9, 15+15                          |

<sup>a</sup>  
Percent for combinations are in sequence with  
cement description.

Table 24. APFA Treated Soil

| <u>Soil</u> | <u>Cement Content, %</u> | <u>Strength (7-day)</u> | <u>OMC</u> | <u>Max. Density</u><br>(PCF) |
|-------------|--------------------------|-------------------------|------------|------------------------------|
| A           | 20                       | 450                     | 9.1        | 127                          |
| A           | 30                       | 362                     | 9.2        | 124                          |
| B           | 20                       | 625                     | 7.7        | 136                          |
| B           | 30                       | 1020                    | 7.7        | 136                          |
| C           | 20                       | 202                     | 10.3       | 118                          |
| C           | 30                       | 440                     | 10.8       | 121                          |

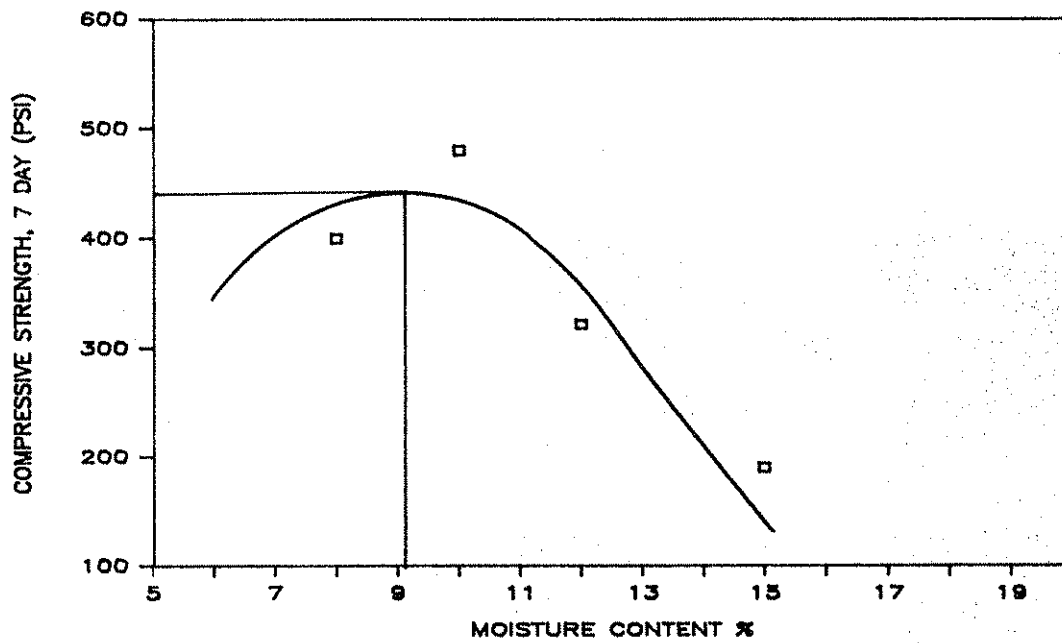
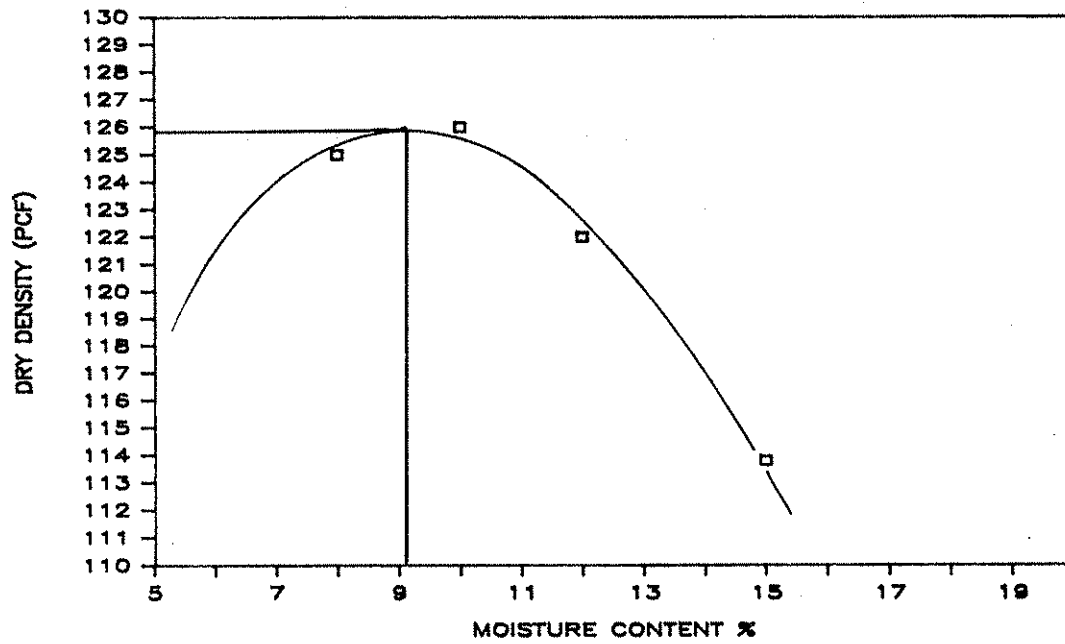


Figure 36. Compressive strength and density for soil A and ammonium phosphate treated fly ash.

summary of data for APFA stabilized soils. Strength data from a similar analysis using the conventional stabilizers are in Figures 37 to 39. These figures are used to correlate strength of APFA treated soils to that of conventional treatments to determine quantities of conventional stabilizer. This process is summarized in Table 25.

#### Cost

Reasonable material costs at point of origin for various ingredients are as follows:

|  |              |
|--|--------------|
| Untreated fly ash  | \$ 12.00/Ton |
| Chemically treated fly ash<br>(ammonium phosphate at \$ 200/Ton) | \$ 13.00/Ton |
| Portland Cement  | \$ 62.00/Ton |
| Kiln Dust  | \$ 12.00/Ton |

If it is assumed that transportation costs are not a factor, the cost for equivalent strength can be computed and are shown in parenthesis in Table 25.

Economics of chemically treated fly ash is dependent on soil type, stabilizer quantity, and is not always the least costly option. However, in most cases chemically treated fly ash could offer significant savings. Job location may also be a significant factor. For example construction near fly ash sources but distant from the three portland cement and associated kiln dust sources should have a significant advantage in transportation. Figure 40 suggests Western

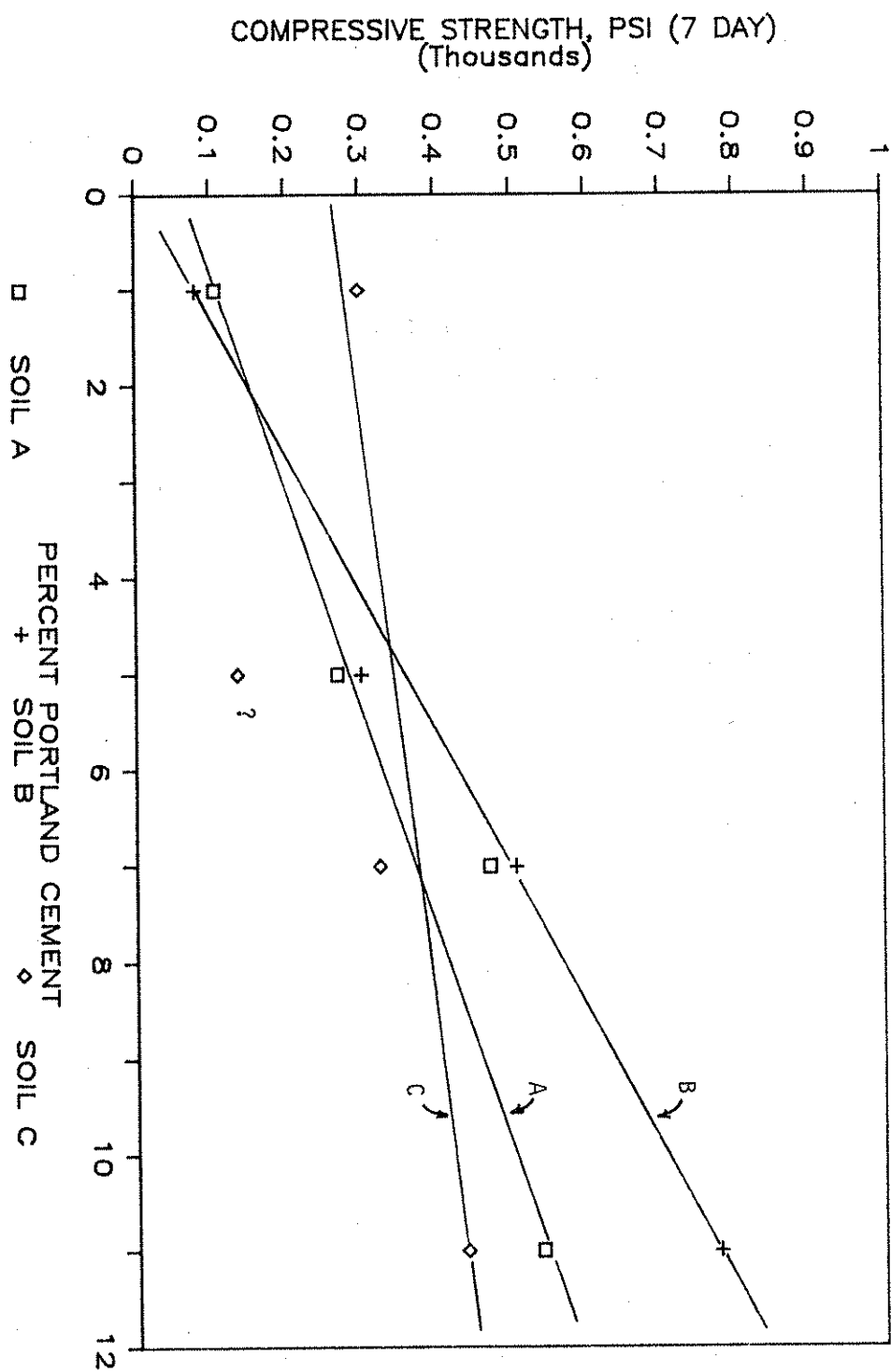


Figure 37. Compressive strength versus percent portland cement.

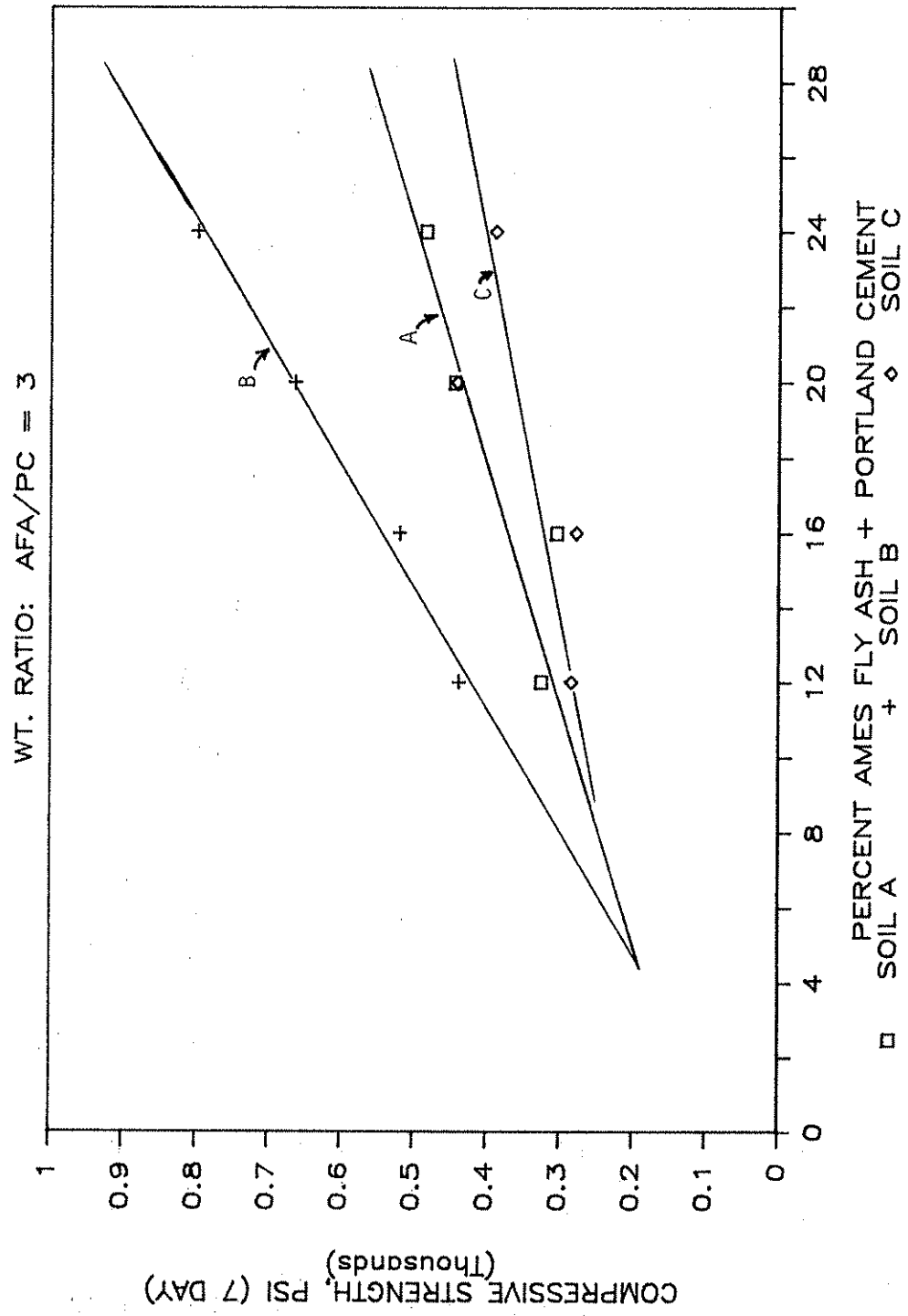


Figure 38. Compressive strength versus percent Ames fly ash and portland cement (3 parts Ames fly ash and 1 part portland cement).

Figure 39. Compressive strength versus percent Ames fly ash and kiln dust (1 part Ames fly ash and 1 part kiln dust).

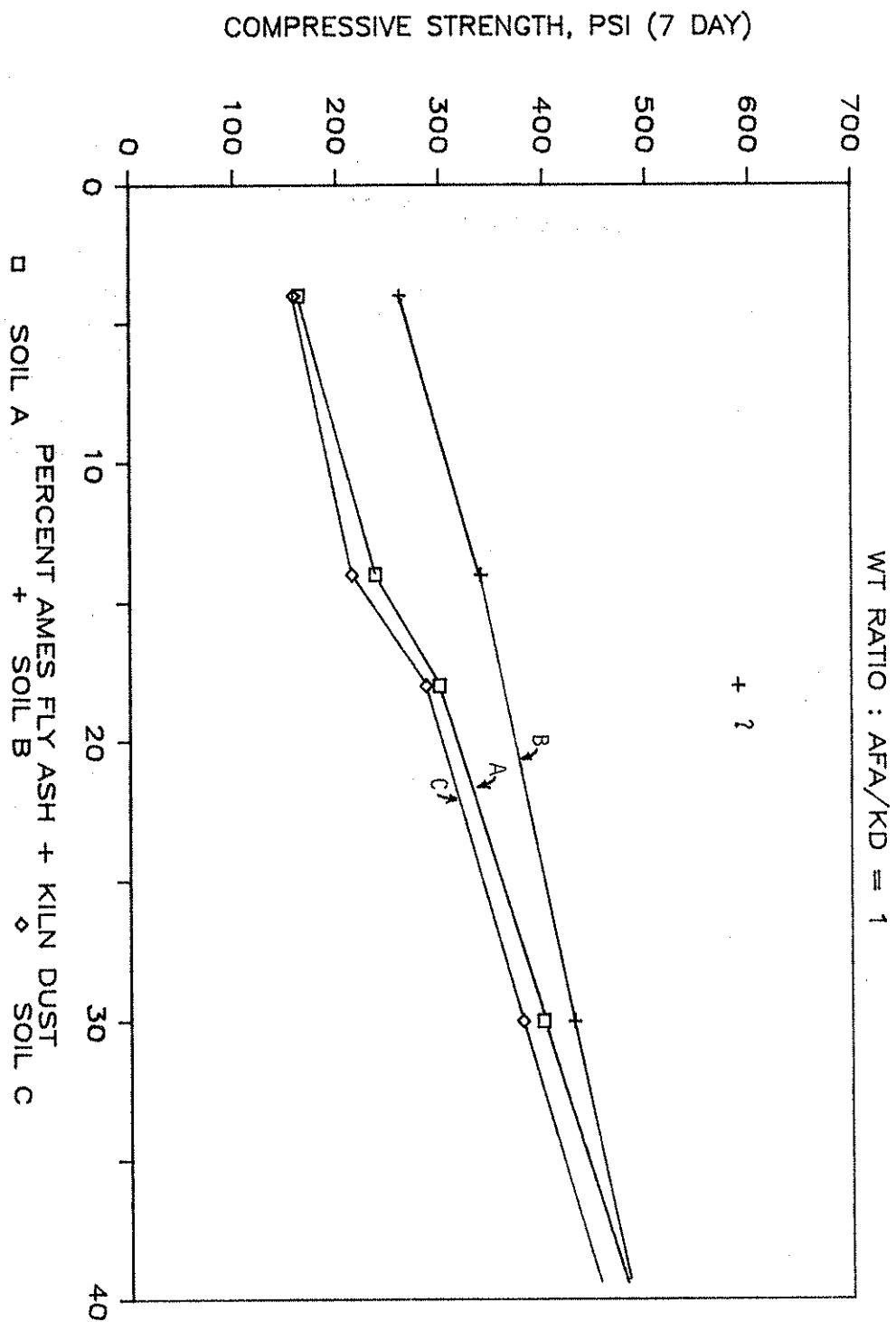




Table 25. Equivalent Stabilizer Concentrations  
Percentage of Stabilizer (Cost)

| <u>Soil</u> | <u>Fly Ash (\$)</u> | <u>PC (\$)</u> | <u>PC + FA (\$)</u> | <u>KD + FA (\$)</u> |
|-------------|---------------------|----------------|---------------------|---------------------|
| A           | 20 (2.60)           | 10 (6.20)      | 22 (5.39)           | 40 (4.80)           |
| B           | 20 (2.60)           | 9 (5.58)       | 20 (4.90)           | >40 (>4.80)         |
| C           | 20 (2.60)           | <2 (1.24)      | 8 (1.96)            | 15 (1.80)           |
| A           | 30 (3.90)           | 6 (3.72)       | 16 (3.92)           | 26 (3.72)           |
| B           | 30 (3.90)           | >12 (7.44)     | 30 (7.35)           | >40 (4.80)          |
| C           | 30 (3.90)           | 11 (6.82)      | 30 (7.35)           | 40 (4.80)           |

and much of Eastern Iowa should have this advantage. With the exception of limited production at Ames, conventional alternatives could be better for the central part of the state. Chemically treated fly ash is at a disadvantage in that it represents a new and possibly more complex construction technology.

- Suitable Fly Ash Sources
- △ Portland Cement Sources

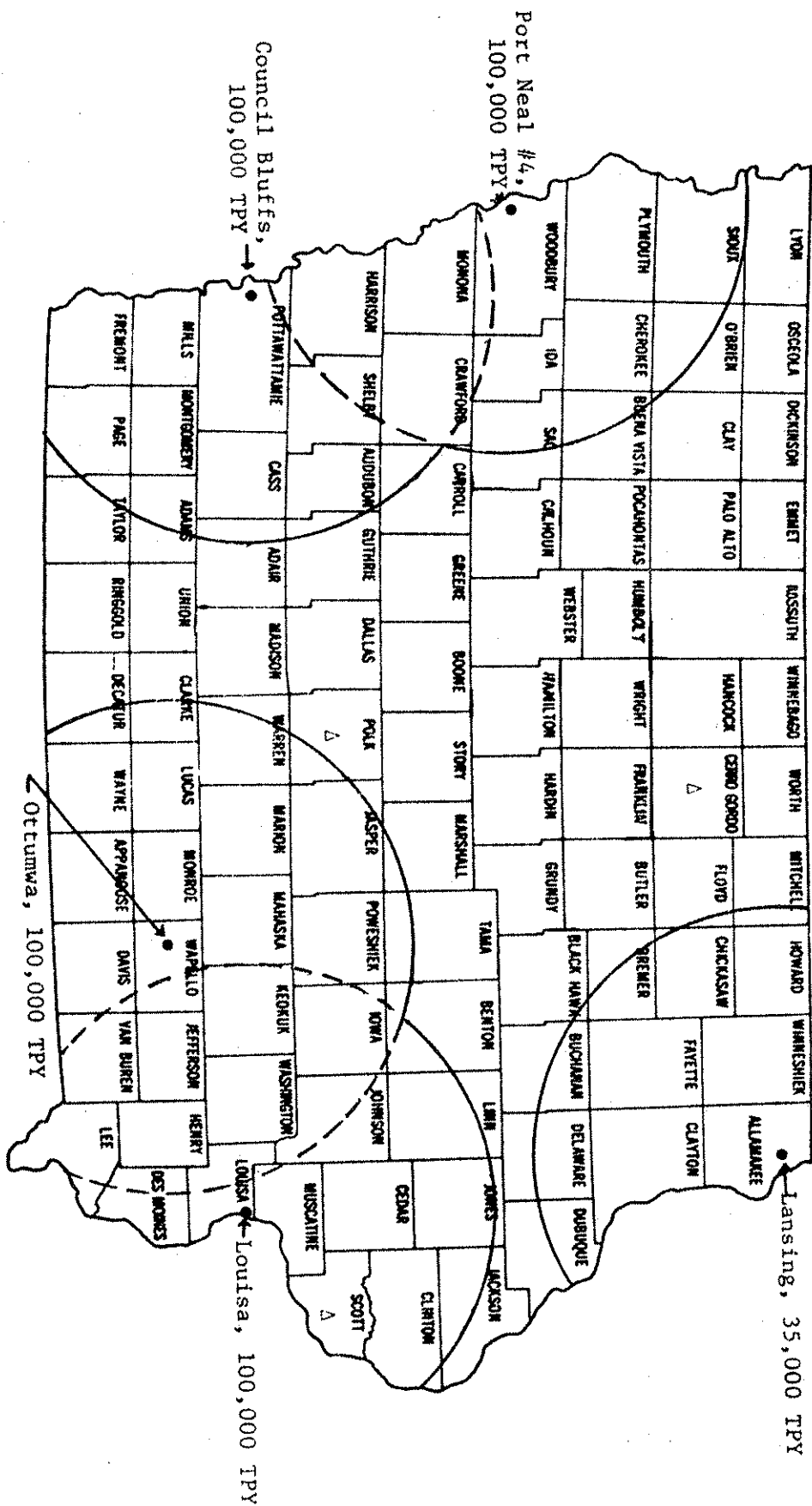


FIGURE 40: SUITABLE FLY ASH SOURCES IN IOWA (CIRCLE RADIUS = 65 MILES)

## DESIGN

One goal of this research was to develop a systematic design methodology based on fly ash behavior. One first must recognize that conventional ASTM C 618 classification is irrelevant to chemically treated fly ash design. Therefore, experimentation on hydraulic behavior and reaction with the appropriate chemicals becomes the first step in design. This can proceed as follows:

1. Chemical selection - If the fly ash is Category II, ammonium nitrate is the additive of choice. If Category III fly ash is available, dibasic ammonium phosphate is appropriate.
2. Chemical quantity - Conceptual plots representing the effect of chemical additive concentration in strength and set time are shown in Figures 41 and 42. Based on work done in this project, it is anticipated that evaluation of chemical effects can be done with seven day strengths on five test specimens at a 0.24 water/cement ratio.
3. Water/cement ratio - After selecting an additive concentration, test specimens at different water/cement ratios should be produced and the strength of the paste defined. A generalized relationship determined from this test will be used as input for design, Figure 43. This evaluation also requires approximately five tests. Again, seven day strengths can be used as the design parameter.

The relationship of paste compressive strength to water/cement ratio at a given additive concentration should be the only input parameter needed for design. Given this information about the fly ash source, the second phase in the design process involves

# COMPRESSIVE STRENGTH

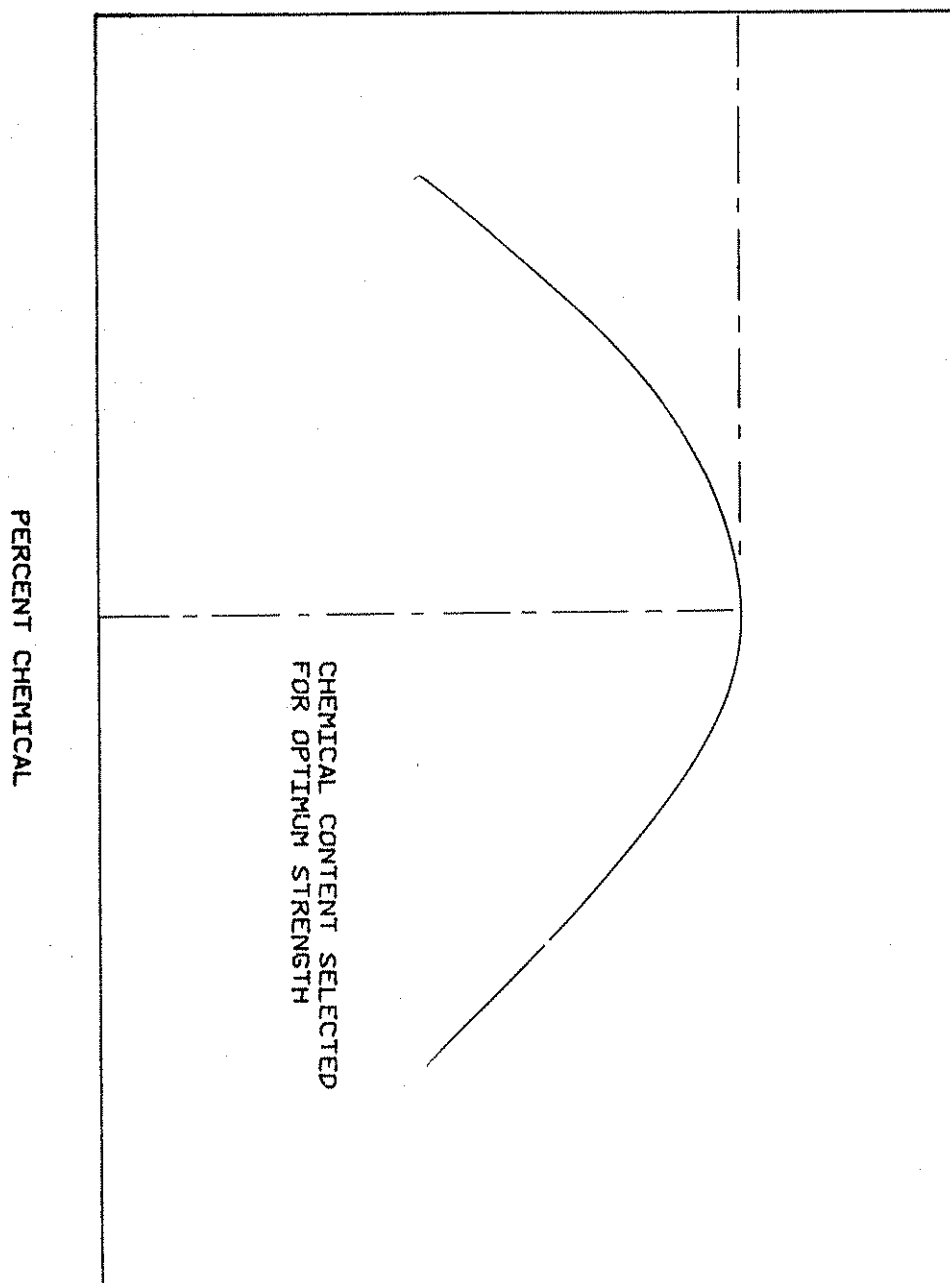


FIGURE 41 : CONCEPTUAL PLOT OF COMPRESSIVE STRENGTH VERSUS PERCENT ADDITIVE

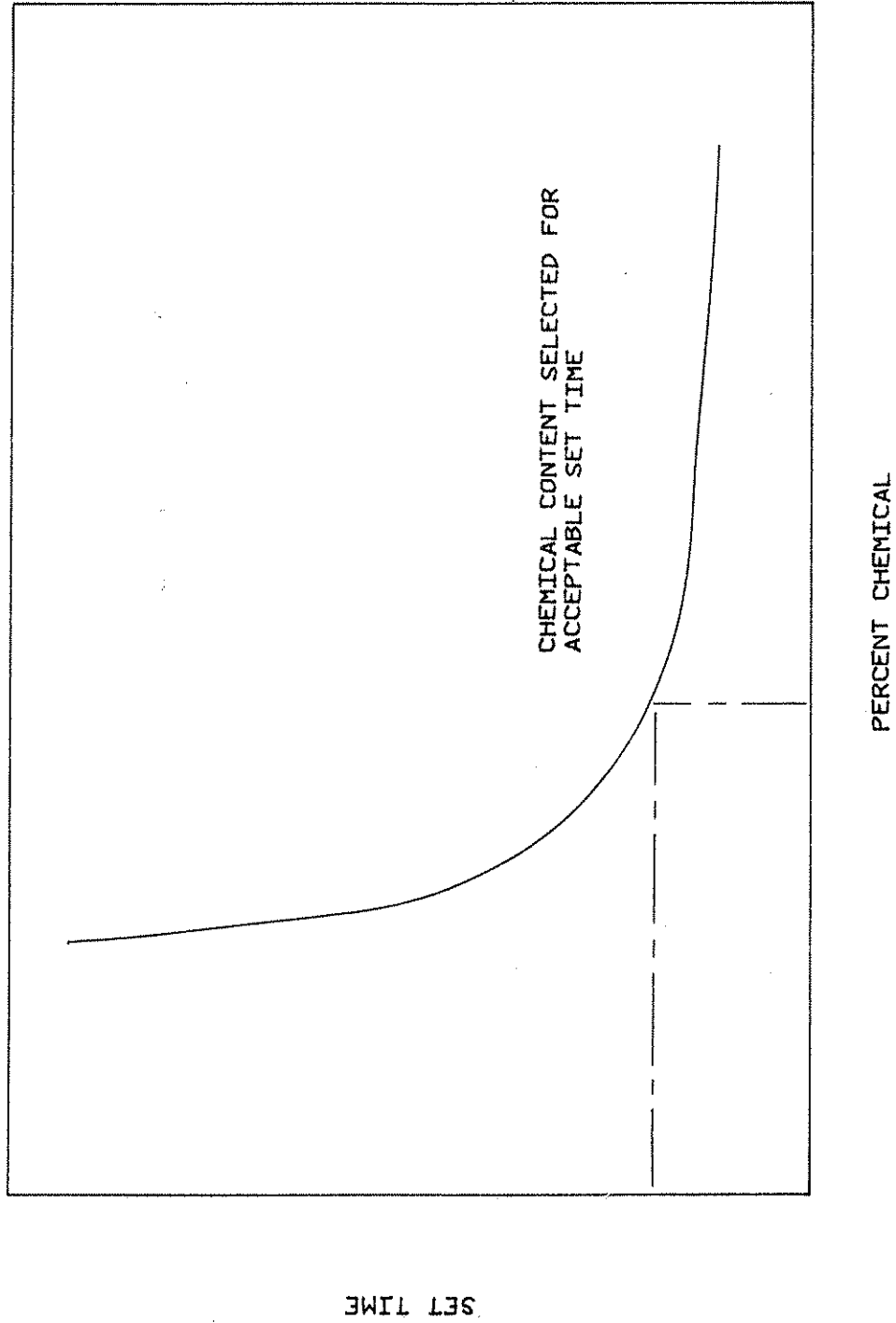


FIGURE 42: CONCEPTUAL PLOT OF SET TIME VERSUS PERCENT ADDITIVE

COMPRESSIVE STRENGTH

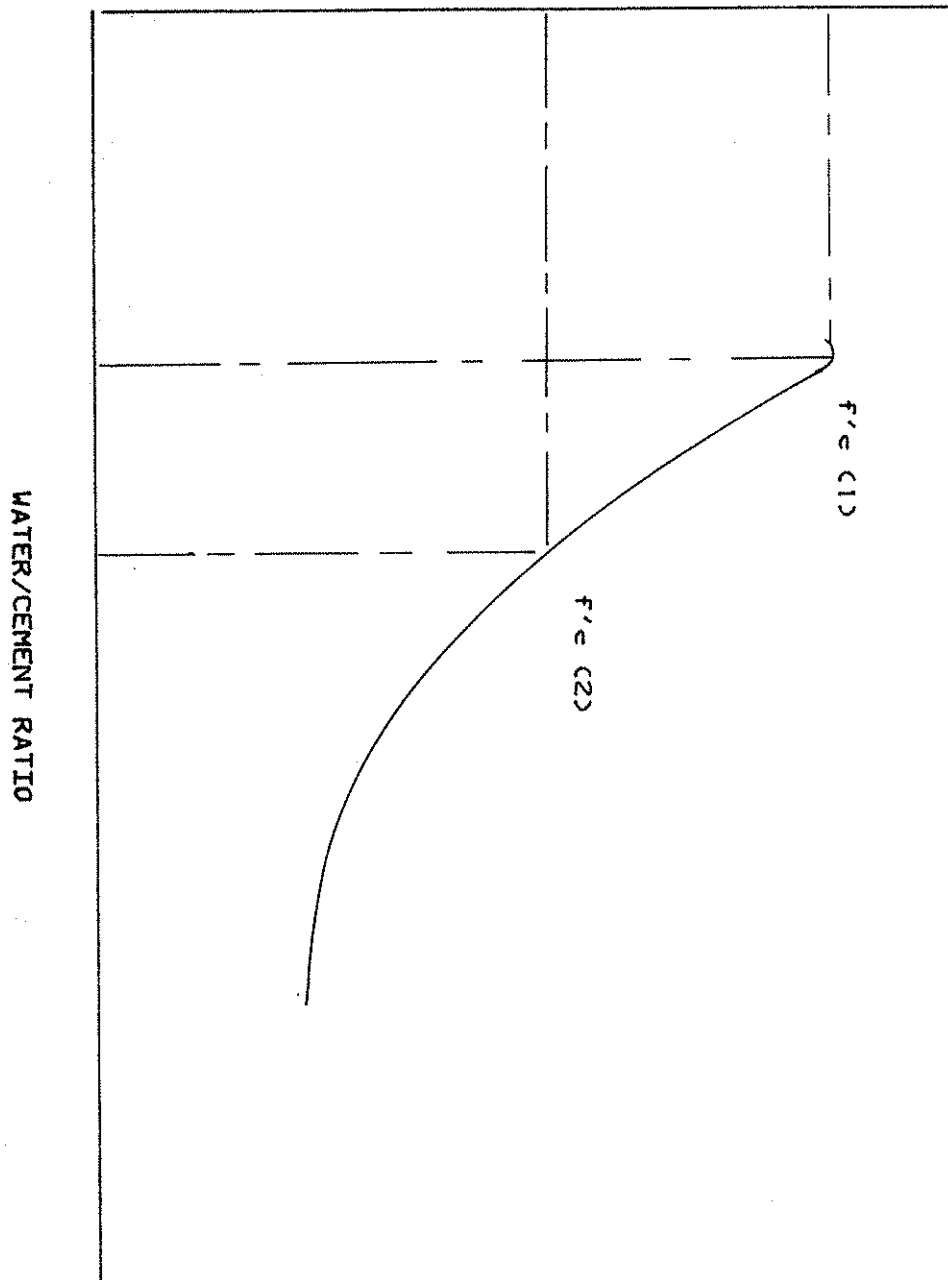


FIGURE 43: CONCEPTUAL PLOT OF COMPRESSIVE STRENGTH VERSUS WATER/CEMENT RATIO

evaluation of the aggregate. The only parameters required for design are a sieve analysis, specific gravity, and dry rodded unit weight. The amount of fly ash required to fill the voids with hydrated paste can be determined from the dry rodded unit weight and specific gravity. In practice, the quantity can be reduced by one third and still produce suitable bases.

Knowledge of relative surface area is a final element to design. The intent of this project was to provide a definitive relationship between strength of a cement paste and relative surface area. This did not work because of the scatter in data. Thus relative surface area can only be used in general terms. In other words, aggregates having relative surface area greater than 80 should not be considered; in general, one might observe that smaller values of relative surface area indicate better aggregate.

## SUMMARY AND CONCLUSIONS

### Secondary Additives

It has been shown that desirable reactions with high calcium fly ash could be initiated with trace additives, thus making calcium available for formation of useful calcium-aluminum-silicate hydrates (stratlingite), rather than the expected (and hypothesized) calcium-silicate hydrates. The variability in physical properties of fly ash (e.g., compressive strength development, setting time, etc.) from a given generating station has been shown to depend on the amount of calcium bearing crystalline compounds present and not on conventional elemental composition. In practice, ammonium nitrate appears to perform best with Category II fly ashes and dibasic ammonium phosphate is suited to Category III fly ashes. It is fortunate that fertilizer grade compounds are suited for secondary additives. Additionally, it was demonstrated that conventional additives (such as portland cement, lime and dolomitic lime) have little or no effect on fly ashes, supporting the view that additional calcium should have no effect on amorphous material already rich in calcium.

### Crusher Fines Stabilization

1. Cement strength and aggregate surface area exert a significant influence on strength of crusher fines stabilized with chemically modified fly ash.



2. Cement strength is a complex function of fly ash composition which appears to vary with time for specific generating stations. Thus, design must depend on experimental work for each specific batch of fly ash.
3. Relative surface area is a convenient parameter useful to gauge suitability of an aggregate for base stabilization. Strength generally decreases as relative surface area increases. Fly ash cements having strength on the order of 1000 psi are capable of producing 500 psi base courses with relative surface areas as high as 80. The natural crusher fines used in this research had a relative surface area of 30.
4. Compressive strengths of the base (from a high strength cement) are very sensitive to water/cement ratio. The best construction results should be achieved on the wet side of an optimum water/cement ratio. Although quality control is less critical for weaker cements, bases obtained are not particularly strong.
5. Sensitivity between strength and density increases with cement strength. Stronger base courses are possible with stronger cements but the compaction process must be more closely supervised.
6. Category III fly ash stabilized materials surpassed frost action standards established for portland cement stabilized bases, while Category II fly ash did not. Fortunately, Category III is Iowa's most abundant fly ash.
7. Shrinkage of both Category II and Category III fly ashes is about one-half that of portland cement.

### Soil Stabilization

1. A wide range of soils can be stabilized with ammonium phosphate treated fly ash. Significant strength increases were observed when fly ash content exceeded 10 percent.
2. Clayey/silty sands showed more gain in strength than gravelly and clayey soils, indicating that a combination of silty or clayey particles and sands is more suitable for stabilization than sandy or clayey soils alone.
3. Fly ash can be used as a soil modifier. The addition of fly ash significantly reduces plasticity.
4. An optimum concentration of soil particles (50 to 60 percent passing through the #200 sieve), produces maximum strength.
5. Soil compressive strength decreases with increasing plasticity index.
6. Stabilized fine grained soils may not be sufficiently resistant to wet-dry and freeze-thaw conditions.

### Equivalent Strength

Chemically treated fly ash is consistently cost effective for granular soils, but is not always economical for plastic clays.

## RECOMMENDATIONS FOR FURTHER STUDY

Work thus far is based on data from a laboratory environment. The next logical step is field application, which will probably indicate we did not learn all the essentials of the process. Attempts at field construction will allow evaluation of laboratory assessments of workability and set time. Additionally, potential problems associated with introducing chemicals, mixing, placement, and compaction will surface and need to be solved.

A nagging problem that still remains unresolved is the potential for variability in fly ash, even from a single source. Physical experimentation seems the only reliable method of assessment.

Lastly, all likely trace additives have not been fully explored. Additional work should be done to look for less costly additives or additive combinations. A good direction might be less costly superphosphates in combination with lignin retarders.

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## APPENDIX A

### SOURCE MONITORING



Table A1. Source Monitoring - Neal #2

| Chemical Composition (%) | Sample Data |        |        |        |
|--------------------------|-------------|--------|--------|--------|
|                          | 032383      | 060183 | 070883 | 091383 |
| Silicon Oxide            | 51.70       | 54.06  | 48.49  | 52.49  |
| Aluminum Oxide           | 19.96       | 19.28  | 16.81  | 18.08  |
| Iron Oxide               | 6.43        | 7.96   | 6.21   | 6.22   |
| Total                    | 78.09       | 81.30  | 71.51  | 76.79  |
| Calcium Oxide            | 16.57       | 14.29  | 14.76  | 17.34  |
| Magnesium Oxide          | 3.91        | 3.44   | 1.80   | 3.20   |
| Sulphur Trioxide         | 1.51        | 2.76   | 1.77   | 1.61   |
| Phosphorous Pentoxide    | 0.72        | 0.60   | 0.33   | 0.53   |
| Potassium Oxide          | 1.63        | 1.77   | 1.57   | 1.72   |
| Sodium Oxide             | 0.32        | 0.28   | 0.21   | 0.33   |
| Titanium Oxide           | 0.71        | 0.68   | 0.53   | 0.62   |
| Physical Test Results    |             |        |        |        |
| Alkalies as Sodium Oxide | 0.27        | 0.39   | 0.31   | 0.49   |
| Moisture (%)             | 0.17        | 0.13   | 0.10   | 0.23   |
| Loss on Ignition         | 0.26        | 0.58   | 0.34   | 0.19   |
| Fineness ( + #325 )      | 19.0        | 23.8   | 21.1   | 17.1   |
| Specific Gravity         | 2.35        | 2.38   | 2.38   | 2.37   |
| Lime-pozzolan (psi)      | 937         | 1246   | 1219   | 2030   |
| Cement-Pozzolan (%)      | 101         | 100    | 99     | 95     |
| Autoclave Expansion (%)  | 0.115       | 0.058  | 0.110  | 3.7    |
| Water Requirements (%)   | 86          | 86     | 90     | 96     |



Table A2. Source Monitoring - Neal #3

|                          | Sample Data |        |
|--------------------------|-------------|--------|
| Chemical Composition (%) | 000000      | 102783 |
| Silicon Oxide            | 55.29       | 48.80  |
| Aluminum Oxide           | 17.31       | 18.62  |
| Iron Oxide               | 5.72        | 7.02   |
| Total                    | 78.32       | 74.49  |
| Calcium Oxide            | 15.51       | 17.59  |
| Magnesium Oxide          | 3.30        | 3.93   |
| Sulphur Trioxide         | 0.94        | 1.31   |
| Phosphorous Pentoxide    | 0.76        | 0.81   |
| Potassium Oxide          | 1.47        | 1.43   |
| Sodium Oxide             | 0.56        | 0.35   |
| Titanium Oxide           | 0.65        | 0.75   |

Table A3. Source Monitoring - Neal #4

| Chemical Composition (%) | Sample Data |        |        |        |
|--------------------------|-------------|--------|--------|--------|
|                          | 020883      | 060183 | 070883 | 090183 |
| Silicon Oxide            | 34.10       | 36.29  | 33.00  | 35.65  |
| Aluminum Oxide           | 16.05       | 15.63  | 15.97  | 15.58  |
| Iron Oxide               | 6.30        | 6.11   | 4.70   | 6.07   |
| Total                    | 56.45       | 58.03  | 53.67  | 57.30  |
| Calcium Oxide            | 26.49       | 25.59  | 22.35  | 25.72  |
| Magnesium Oxide          | 6.32        | 5.94   | 3.57   | 6.10   |
| Sulphur Trioxide         | 2.95        | 3.53   | 1.78   | 3.23   |
| Phosphorous Pentoxide    | -           | 0.74   | 0.68   | 0.81   |
| Potassium Oxide          | 0.24        | 0.29   | 0.37   | 0.36   |
| Sodium Oxide             | 1.96        | 2.08   | 1.18   | 2.25   |
| Titanium Oxide           | -           | 1.03   | 0.86   | 0.99   |
| Physical Test Results    |             |        |        |        |
| Alkalies as Sodium Oxide | 1.39        | 1.45   | 1.05   | 1.43   |
| Moisture (%)             | 0.04        | 0.012  | 0.03   | 0.02   |
| Loss on Ignition         | 0.16        | 0.19   | 0.19   | 0.17   |
| Fineness ( +#325 )       | 10.2        | 7.0    | 10.6   | 12.55  |
| Specific Gravity         | 2.69        | 2.69   | 2.70   | 2.71   |
| Lime-pozzolan (psi)      | 1420        | 1800   | 1657   | 1222   |
| Cement-Pozzolan (%)      | 100         | 113    | 100    | 94     |
| Autoclave Expansion (%)  | 0.089       | 0.086  | 0.080  | 0.074  |
| Water Requirements (%)   | 88          | 88     | 90     | 89     |

Table A4. Source Monitoring - Council Bluffs

| Chemical Composition (%) | Sample Data |        |        |        |
|--------------------------|-------------|--------|--------|--------|
|                          | 031183      | 062483 | 082683 | 042883 |
| Silicon Oxide            | 30.64       | 32.15  | 31.32  | 31.79  |
| Aluminum Oxide           | 17.27       | 16.87  | 16.72  | 16.74  |
| Iron Oxide               | 4.93        | 5.26   | 5.14   | 5.26   |
| Total                    | 52.84       | 54.28  | 53.18  | 53.79  |
| Calcium Oxide            | 27.80       | 27.22  | 28.58  | 28.02  |
| Magnesium Oxide          | 6.47        | 6.58   | 6.74   | 6.82   |
| Sulphur Trioxide         | 2.65        | 3.03   | 3.29   | 3.29   |
| Phosphorous Pentoxide    | -           | 0.72   | 0.88   | 1.00   |
| Potassium Oxide          | 0.31        | 0.29   | 0.37   | 0.34   |
| Sodium Oxide             | 1.70        | 1.89   | 1.78   | 1.76   |
| Titanium Oxide           | -           | 1.40   | 1.36   | 1.32   |
| Physical Test Results    |             |        |        |        |
| Alkalies as Sodium Oxide | 1.18        | 1.40   | 1.35   | 1.31   |
| Moisture (%)             | 0.06        | 0.05   | 0.08   | 0.08   |
| Loss on Ignition         | 0.19        | 0.36   | 0.265  | 0.48   |
| Fineness ( + #325 )      | 18.0        | 10.0   | 10.2   | 12.3   |
| Specific Gravity         | 2.69        | 2.72   | 2.69   | 2.75   |
| Lime-pozzolan (psi)      | 417         | 1257   | 1684   | 1828   |
| Cement-Pozzolan (%)      | 94          | 103    | 97     | 101    |
| Autoclave Expansion (%)  | 0.121       | 0.129  | 0.148  | 0.142  |
| Water Requirements (%)   | 86          | 96     | 96     | 88     |

Table A5. Source Monitoring - Nebraska City

| Chemical Composition (%) | Sample Data |
|--------------------------|-------------|
|                          | 000000      |
| Silicon Oxide            | 42.46       |
| Aluminum Oxide           | 21.46       |
| Iron Oxide               | 4.35        |
| Total                    | 68.27       |
| Calcium Oxide            | 18.06       |
| Magnesium Oxide          | 4.40        |
| Sulphur Trioxide         | 0.81        |
| Phosphorous Pentoxide    | 2.00        |
| Potassium Oxide          | 0.68        |
| Sodium Oxide             | 0.72        |
| Titanium Oxide           | 1.26        |

Table A6. Source Monitoring - Ottumwa

| Chemical Composition (%) | Sample Data |       |        |       |
|--------------------------|-------------|-------|--------|-------|
|                          | COMP3       | COMP5 | COMP7  | COMP4 |
| Silicon Oxide            | 34.26       | 37.04 | 35.52  | 34.10 |
| Aluminum Oxide           | 20.19       | 20.34 | 19.26  | 20.46 |
| Iron Oxide               | 5.32        | 5.10  | 5.18   | 5.26  |
| Total                    | 78.09       | 81.30 | 71.51  | 76.79 |
| Calcium Oxide            | 25.12       | 24.18 | 24.25  | 25.40 |
| Magnesium Oxide          | 4.99        | 4.86  | 4.71   | 5.26  |
| Sulphur Trioxide         | 1.76        | 1.57  | 2.02   | 1.88  |
| Phosphorous Pentoxide    | 1.86        | 1.32  | 0.94   | 1.34  |
| Potassium Oxide          | 0.39        | 0.45  | 0.41   | 0.40  |
| Sodium Oxide             | 1.91        | 1.92  | 2.36   | 2.00  |
| Titanium Oxide           | 1.48        | 1.48  | 1.50   | 1.54  |
| Physical Test Results    |             |       |        |       |
| Alkalies as Sodium Oxide | 1.36        | 1.36  | 1.74   | 1.45  |
| Moisture (%)             | 0.03        | 0.038 | 0.02   | 0.03  |
| Loss on Ignition         | 0.28        | 0.28  | 0.27   | 0.25  |
| Fineness ( +#325 )       | 11.5        | 12.57 | 12.00  | 12.6  |
| Specific Gravity         | 2.64        | 2.58  | 2.59   | 2.62  |
| Lime-pozzolan (psi)      | 1091        | 1253  | 854    | 1301  |
| Cement-Pozzolan (%)      | 82          | 112   | 111    | 92    |
| Autoclave Expansion (%)  | 0.058       | 0.058 | 0.0055 | 0.060 |
| Water Requirements (%)   | 86          | 89    | 92     | 87    |

Table A7. Source Monitoring - Lansing

| Chemical Composition (%) | Sample Data |        |        |        |
|--------------------------|-------------|--------|--------|--------|
|                          | 021583      | 062083 | 070183 | 091383 |
| Silicon Oxide            | 34.48       | 41.20  | 33.69  | 33.50  |
| Aluminum Oxide           | 16.74       | 16.79  | 17.47  | 15.87  |
| Iron Oxide               | 5.58        | 5.58   | 5.24   | 5.76   |
| Total                    | 56.80       | 63.57  | 56.40  | 55.13  |
| Calcium Oxide            | 26.81       | 25.74  | 27.17  | 27.18  |
| Magnesium Oxide          | 6.14        | 6.74   | 7.30   | 6.34   |
| Sulphur Trioxide         | 3.51        | 4.47   | 2.84   | 3.82   |
| Phosphorous Pentoxide    | -           | 0.90   | 1.33   | 0.76   |
| Potassium Oxide          | 0.40        | 0.38   | 0.36   | 0.36   |
| Sodium Oxide             | 2.33        | 1.88   | 2.04   | 1.94   |
| Titanium Oxide           | -           | 1.32   | 1.26   | 1.28   |
| Physical Test Results    |             |        |        |        |
| Alkalies as Sodium Oxide | 1.58        | 1.33   | 1.41   | 1.37   |
| Moisture (%)             | 0.01        | 0.05   | 0.02   | 0.075  |
| Loss on Ignition         | 0.22        | 0.20   | 0.76   | 0.56   |
| Fineness ( + #325 )      | 13.5        | 15.0   | 19.0   | 12.9   |
| Specific Gravity         | 2.72        | 2.81   | 2.75   | 2.80   |
| Lime-pozzolan (psi)      | 1553        | 1266   | 1771   | 1521   |
| Cement-Pozzolan (%)      | 86          | 89     | 75     | 93     |
| Autoclave Expansion (%)  | 0.110       | 0.091  | -      | 0.126  |
| Water Requirements (%)   | 88          | 95     | 100    | 89     |

Table A8. Source Monitoring - Ames

| Chemical Composition (%) | Sample Data |        |        |
|--------------------------|-------------|--------|--------|
|                          | 072583      | 042183 | 090883 |
| Silicon Oxide            | 36.07       | 37.81  | 39.98  |
| Aluminum Oxide           | 17.51       | 19.54  | 18.42  |
| Iron Oxide               | 5.58        | 5.62   | 5.20   |
| Total                    | 59.16       | 62.97  | 63.60  |
| Calcium Oxide            | 23.49       | 22.31  | 22.20  |
| Magnesium Oxide          | 5.50        | 5.21   | 5.02   |
| Sulphur Trioxide         | 3.35        | 4.06   | 3.43   |
| Phosphorous Pentoxide    | 1.22        | 0.94   | 0.93   |
| Potassium oxide          | 0.72        | 0.69   | 0.62   |
| Sodium Oxide             | 2.22        | 2.72   | 2.33   |
| Titanium Oxide           | 1.19        | 1.45   | 1.56   |
| Physical Test Results    |             |        |        |
| Alkalies as Sodium Oxide | -           | 2.03   | 1.52   |
| Moisture (%)             | -           | 0.09   | 0.09   |
| Loss on Ignition         | -           | 0.43   | 0.28   |
| Fineness ( + #325 )      | -           | 19.6   | 15.6   |
| Specific Gravity         | -           | 2.51   | 2.51   |
| Lime-pozzolan (psi)      | -           | 950    | 1205   |
| Cement-Pozzolan (%)      | -           | 107    | 92     |
| Autoclave Expansion (%)  | -           | 0.102  | 0.09   |
| Water Requirements (%)   | -           | 99     | 103    |

Table A9. Source Monitoring - Louisa

| Chemical Composition (%) | Sample Data |        |        |        |
|--------------------------|-------------|--------|--------|--------|
|                          | 080383      | 100383 | 111983 | 010984 |
| Silicon Oxide            | 39.58       | 42.57  | 37.35  | 37.77  |
| Aluminum Oxide           | 19.82       | 19.33  | 19.50  | 19.51  |
| Iron Oxide               | 5.46        | 5.27   | 5.18   | 5.12   |
| Total                    | 64.86       | 67.17  | 62.03  | 62.40  |
| Calcium Oxide            | 23.55       | 22.20  | 23.52  | 23.39  |
| Magnesium Oxide          | 4.71        | 4.16   | 4.68   | 4.49   |
| Sulphur Trioxide         | 1.60        | 1.31   | 1.47   | 1.34   |
| Phosphorous Pentoxide    | 1.34        | 0.64   | 1.36   | 1.39   |
| Potassium oxide          | 0.48        | 0.44   | 0.43   | 0.42   |
| Sodium Oxide             | 1.93        | 1.86   | 1.80   | 1.77   |
| Titanium Oxide           | 1.42        | 1.44   | 1.45   | 1.44   |
| Physical Test Results    |             |        |        |        |
| Alkalies as Sodium Oxide | 1.42        | -      | -      | -      |
| Moisture (%)             | 0.042       | 0.068  | 0.049  | -      |
| Loss on Ignition         | 0.30        | 0.15   | 0.21   | -      |
| Fineness ( + #325 )      | 17.9        | 8.5    | 7.2    | -      |
| Specific Gravity         | 2.69        | 2.42   | 2.59   | -      |
| Lime-pozzolan (psi)      | 1173        | 1182   | 1169   | -      |
| Cement-Pozzolan (%)      | 93          | -      | -      | -      |
| Autoclave Expansion (%)  | 0.056       | 0.008  | 0.002  | -      |
| Water Requirements (%)   | 82          | -      | -      | -      |

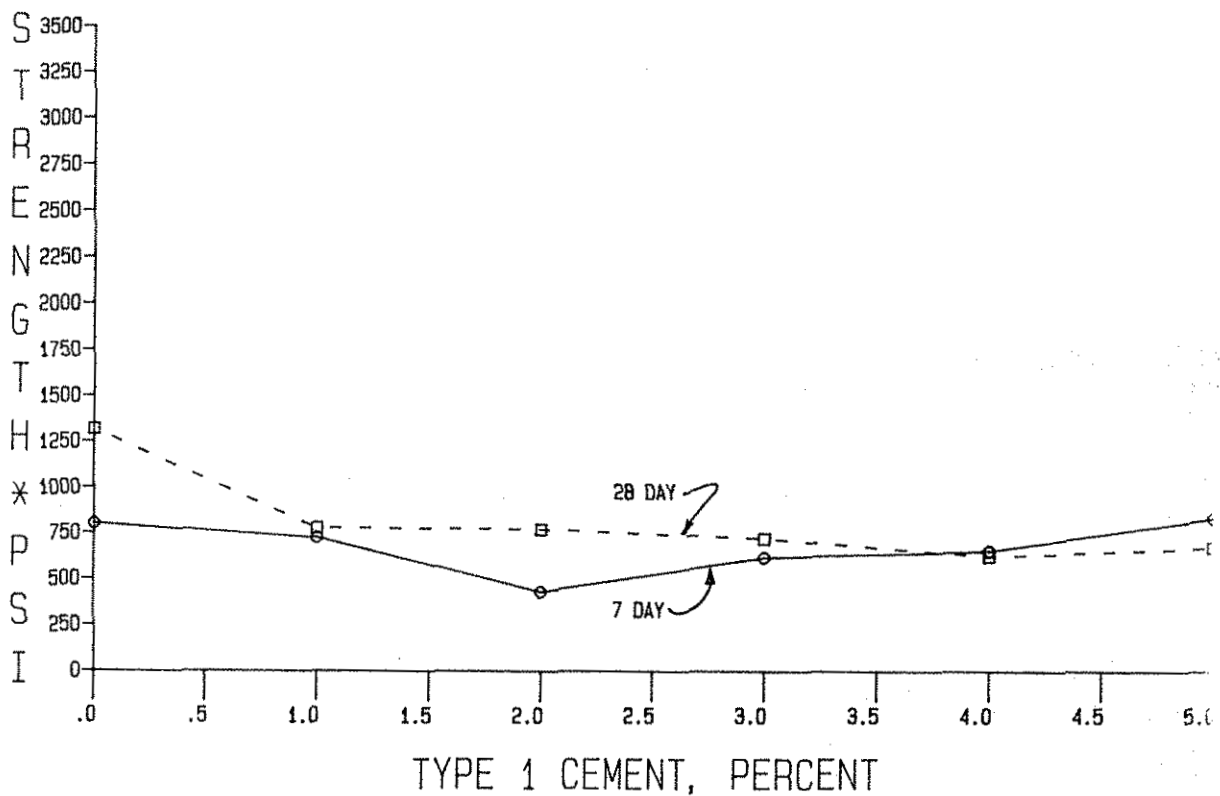
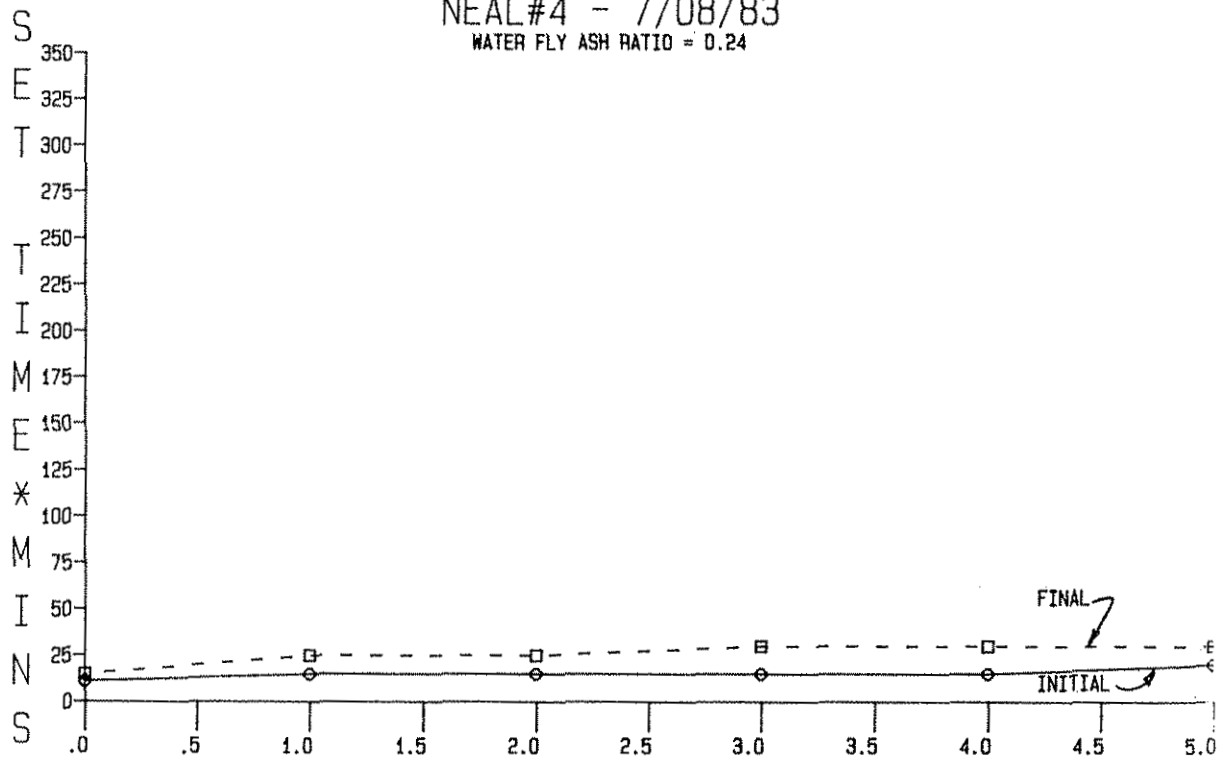


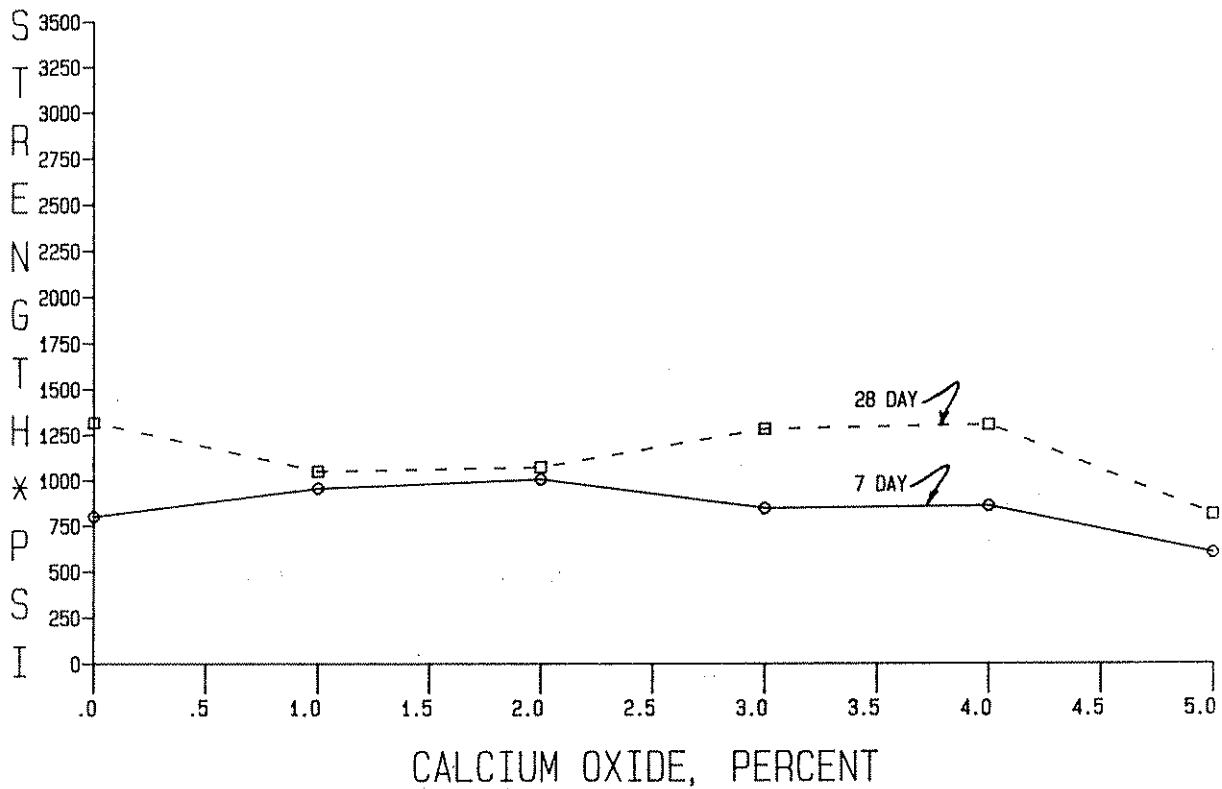
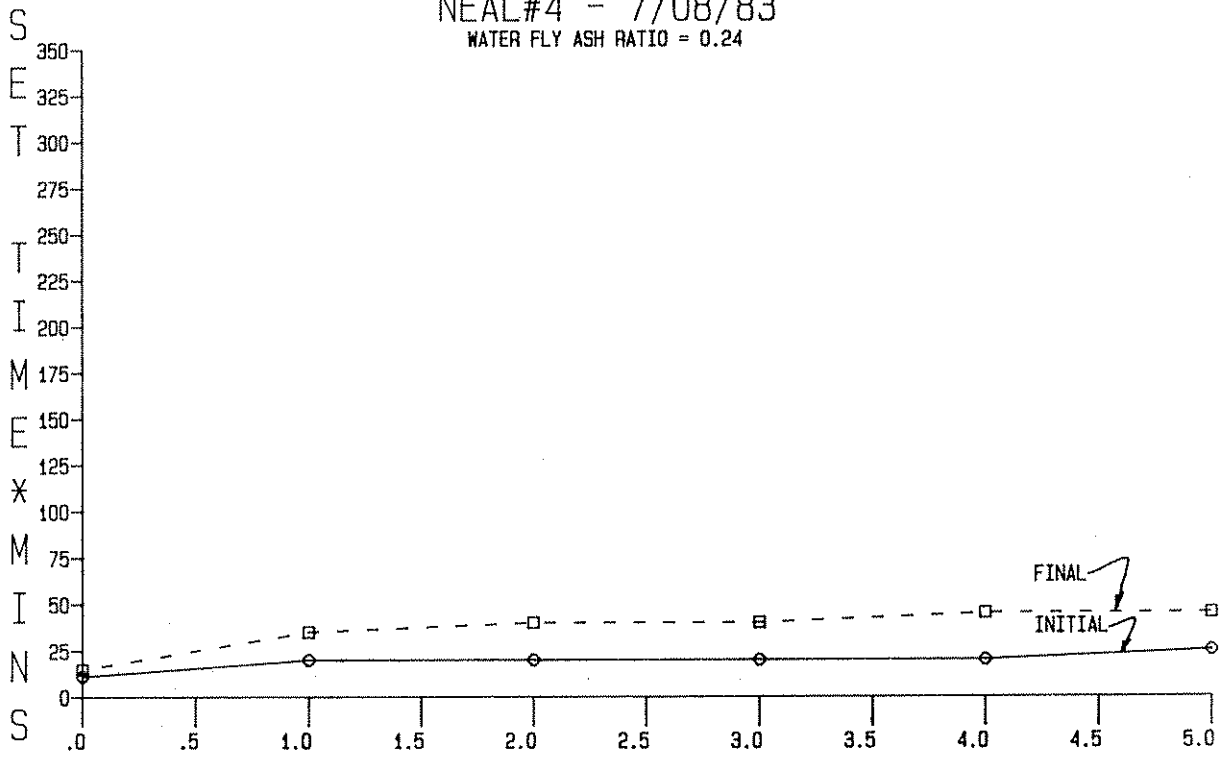
## APPENDIX B

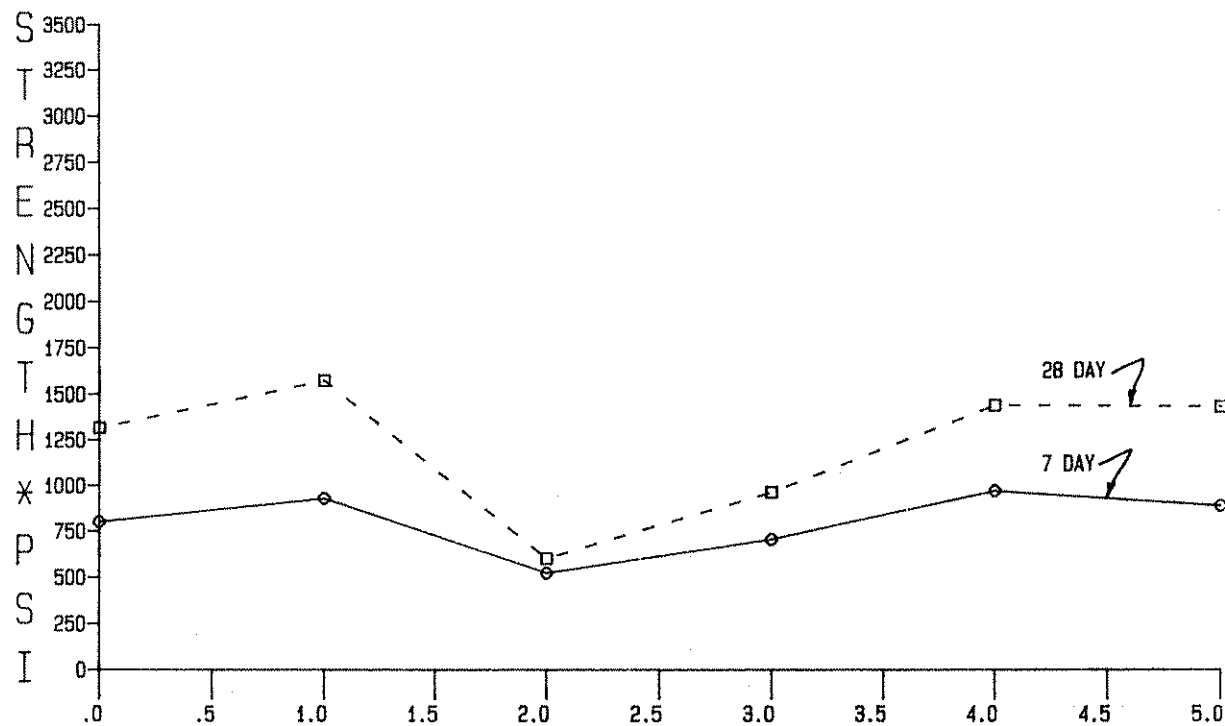
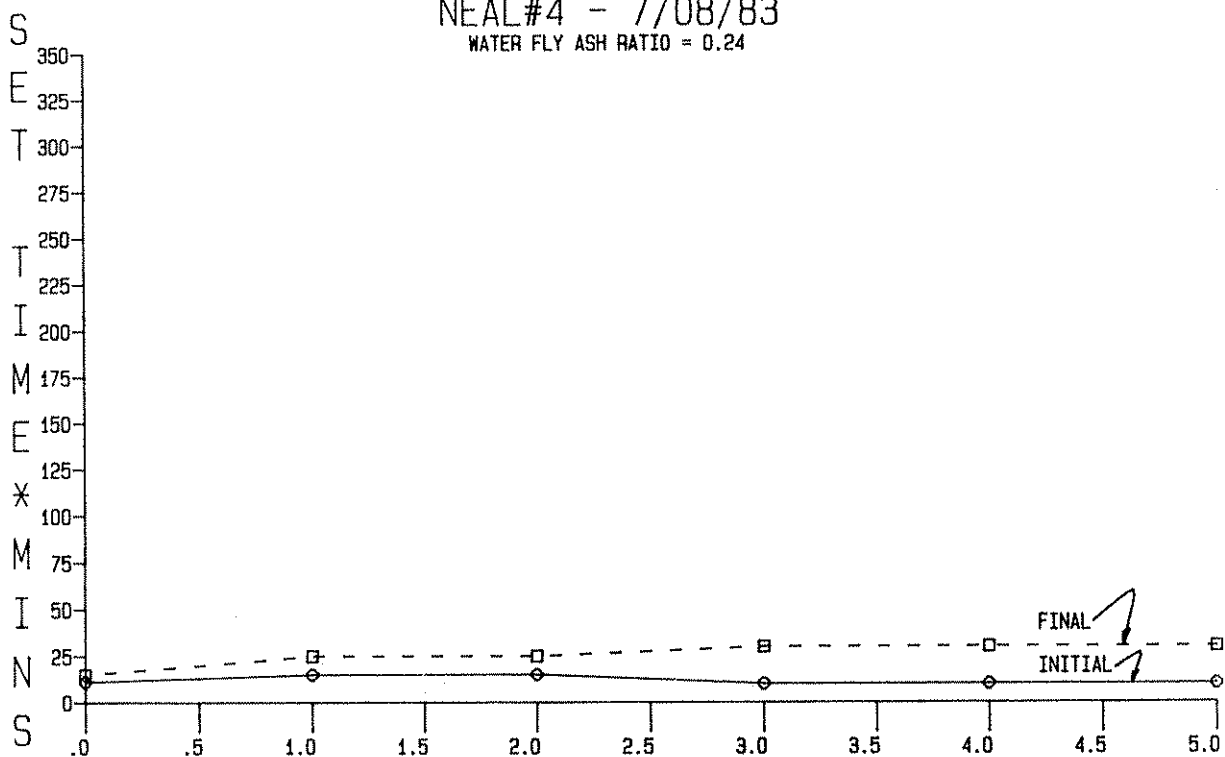
### ADDITIVE OPTIMIZATION

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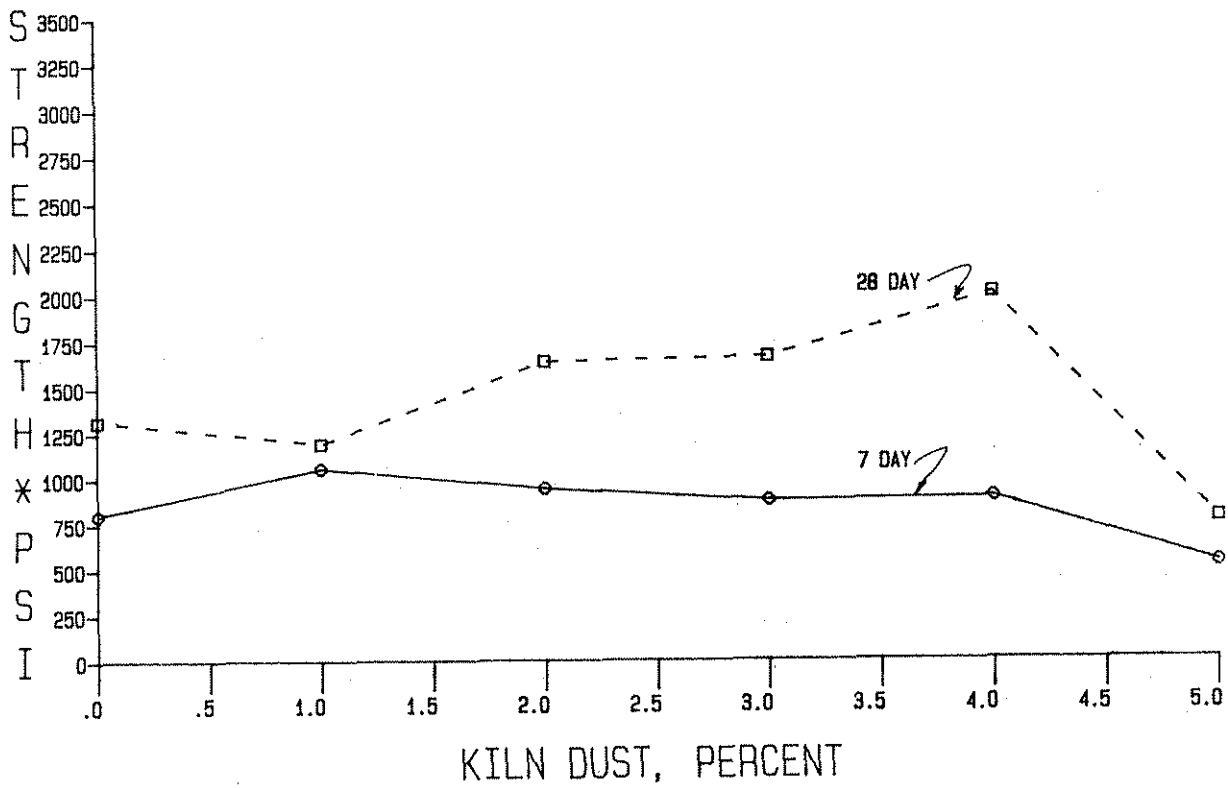
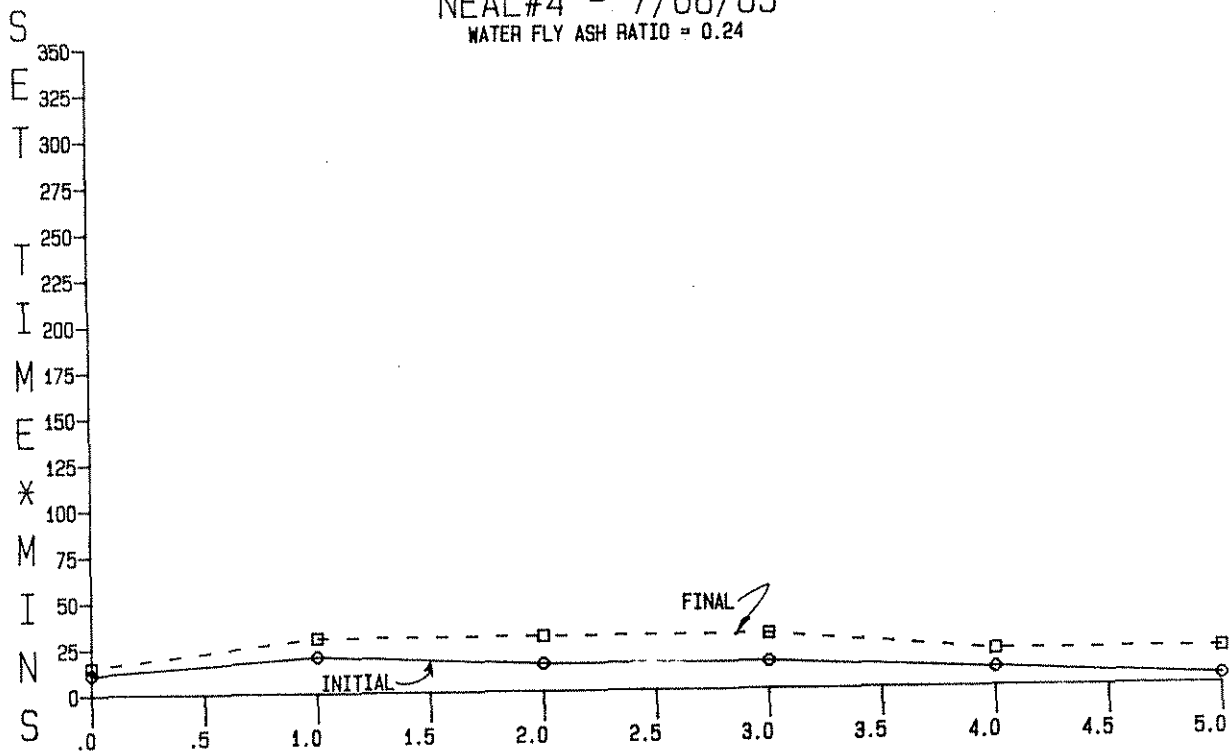
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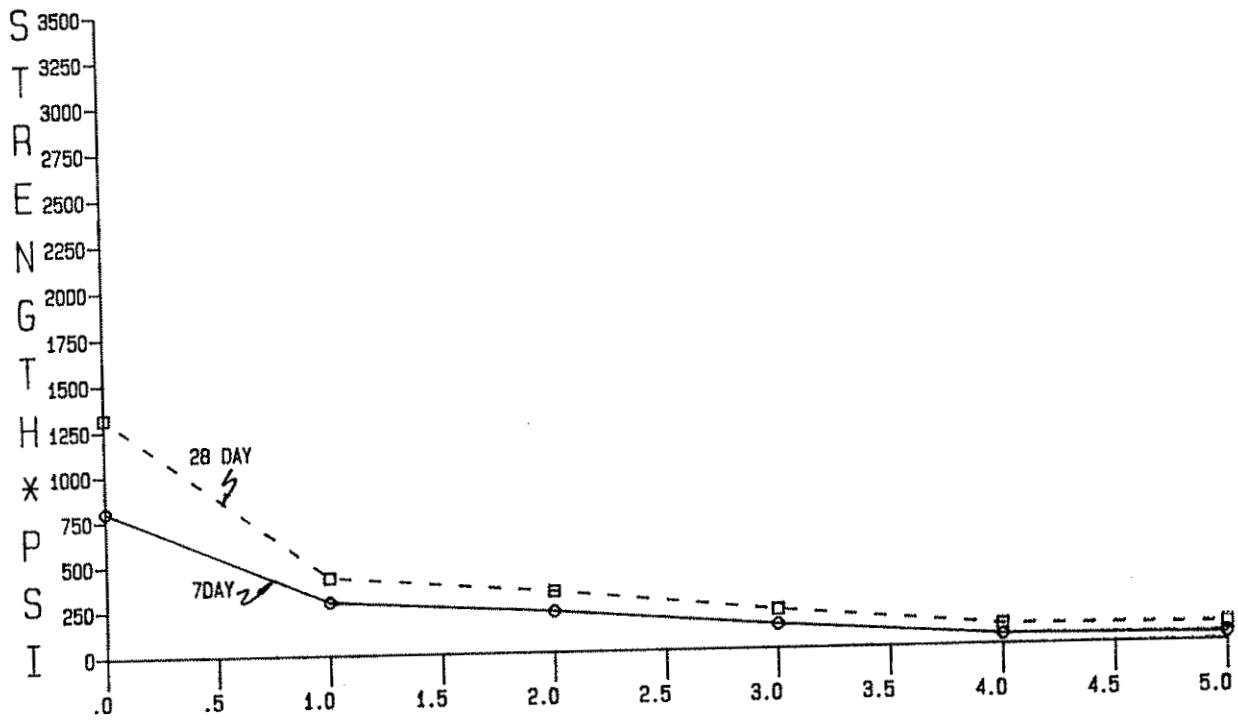
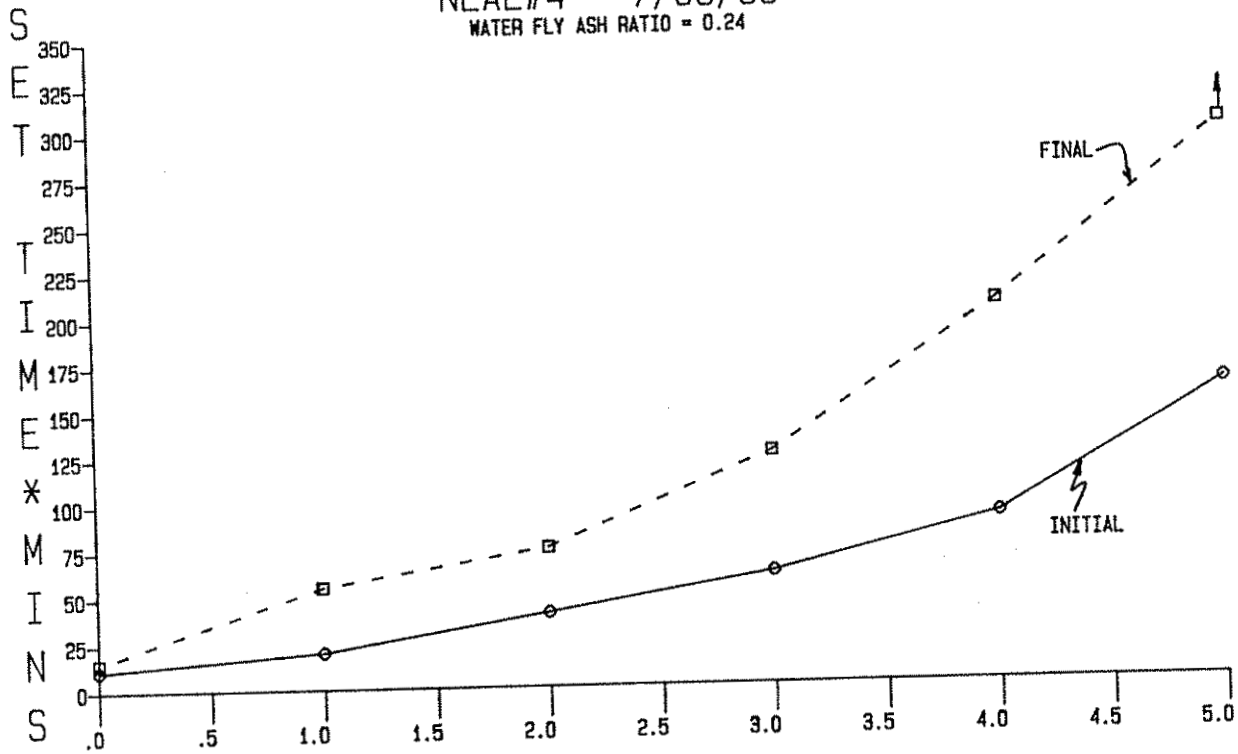
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MAGNESIUM + CALCIUM OXIDE (MGO=0.57 CAO), PERCENT

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B-5

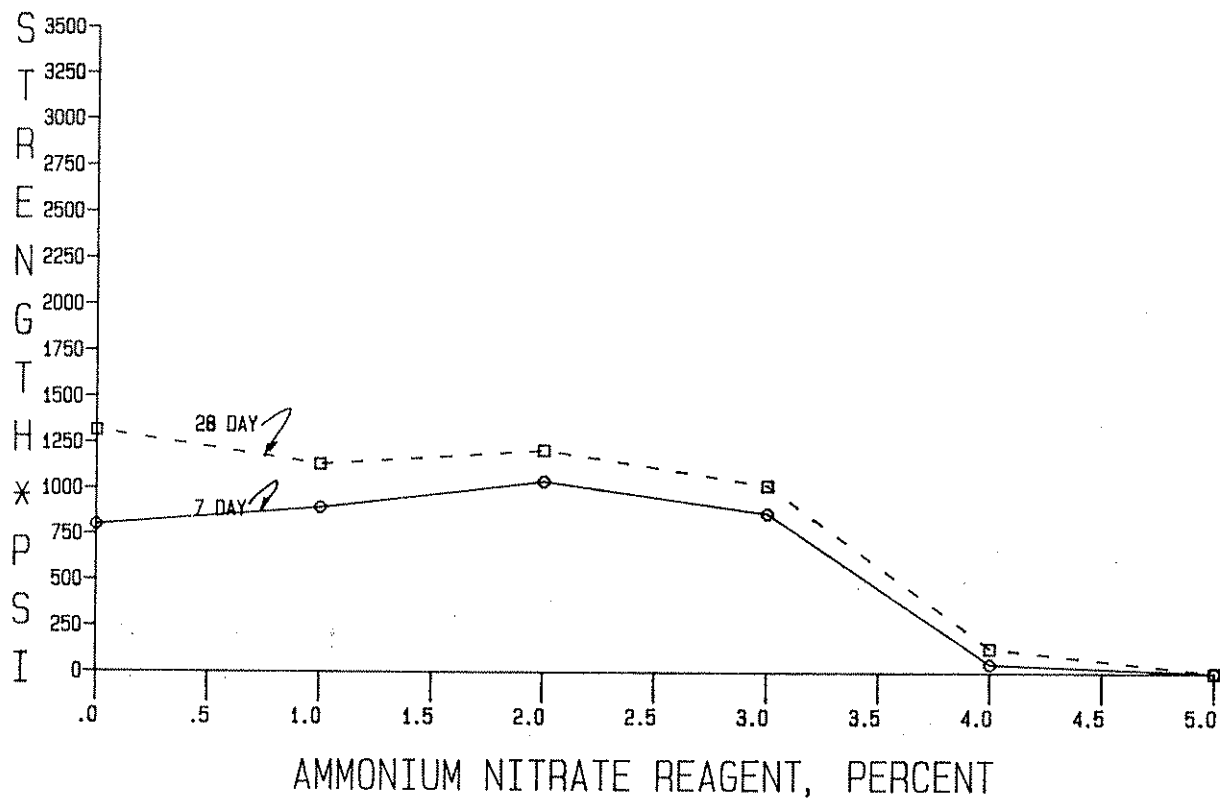
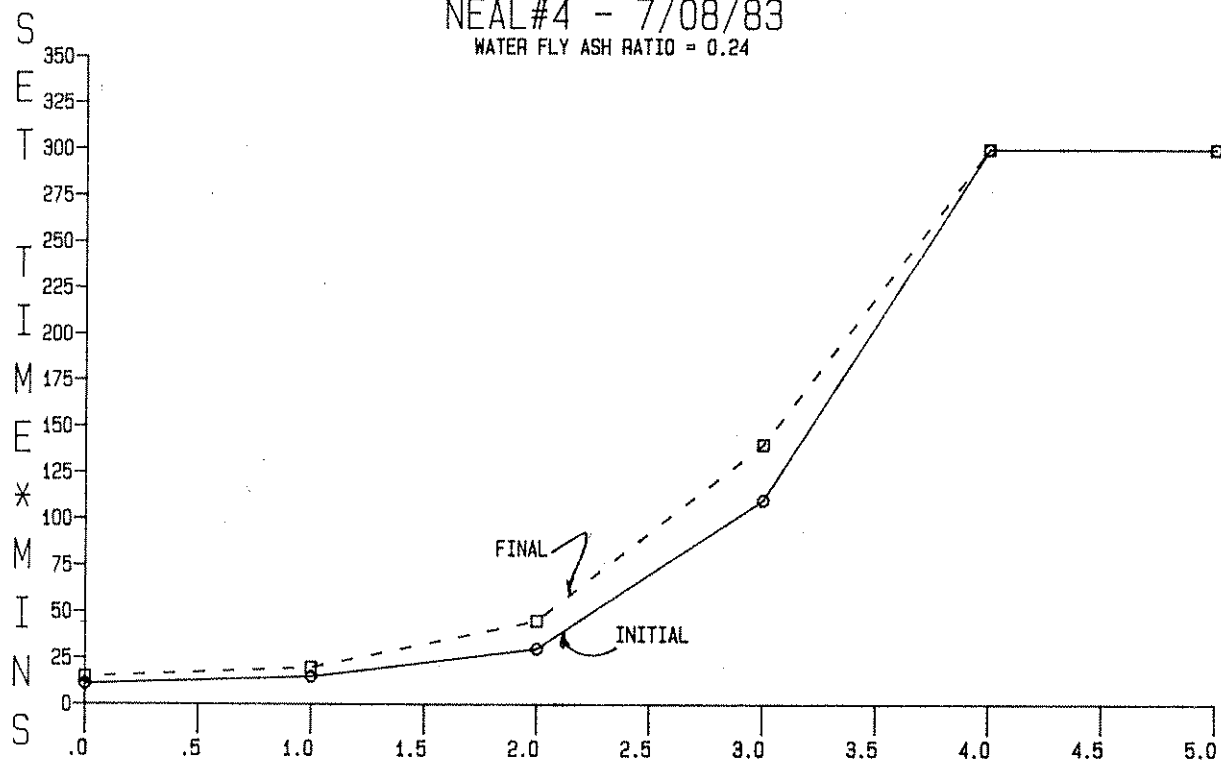
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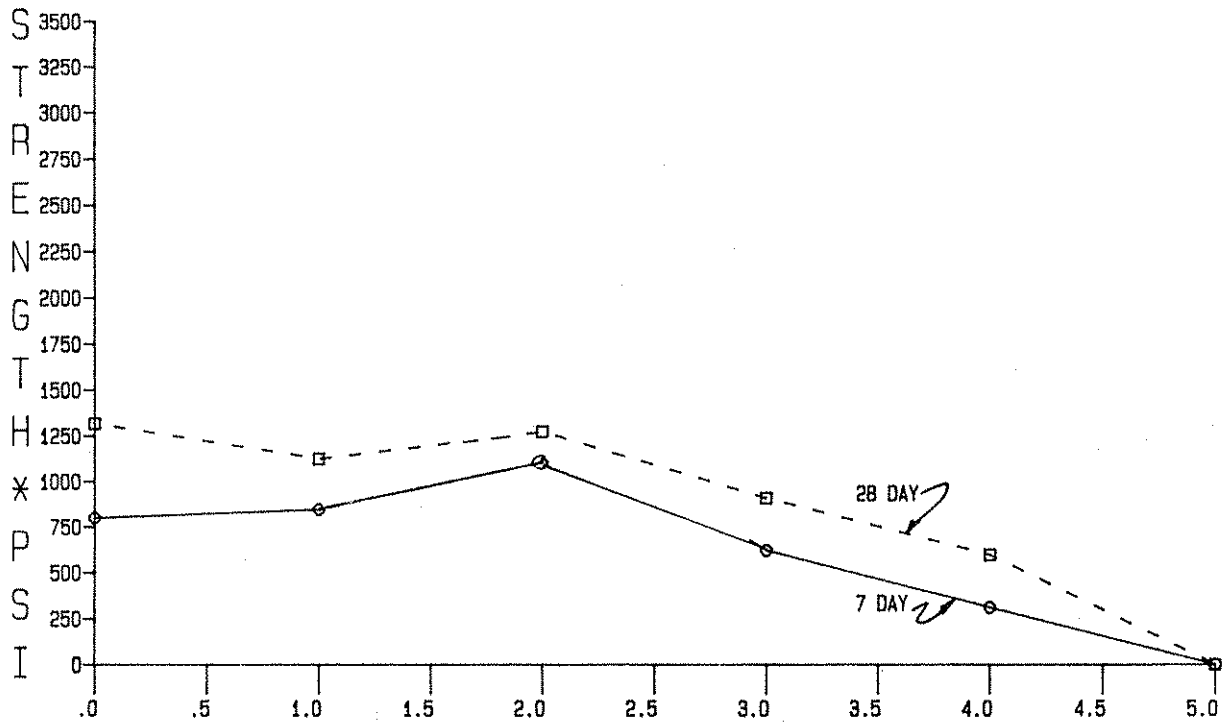
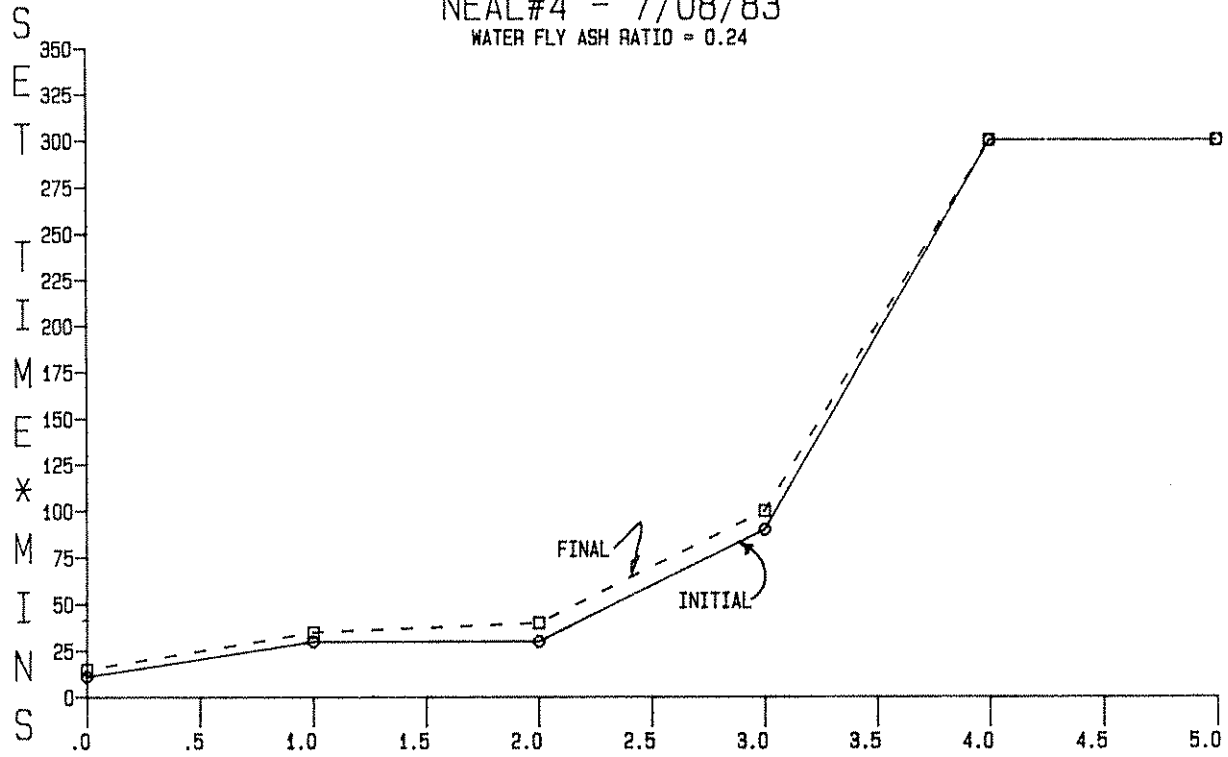
SODIUM PHOSPHATE, PERCENT

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WATER FLY ASH RATIO = 0.24

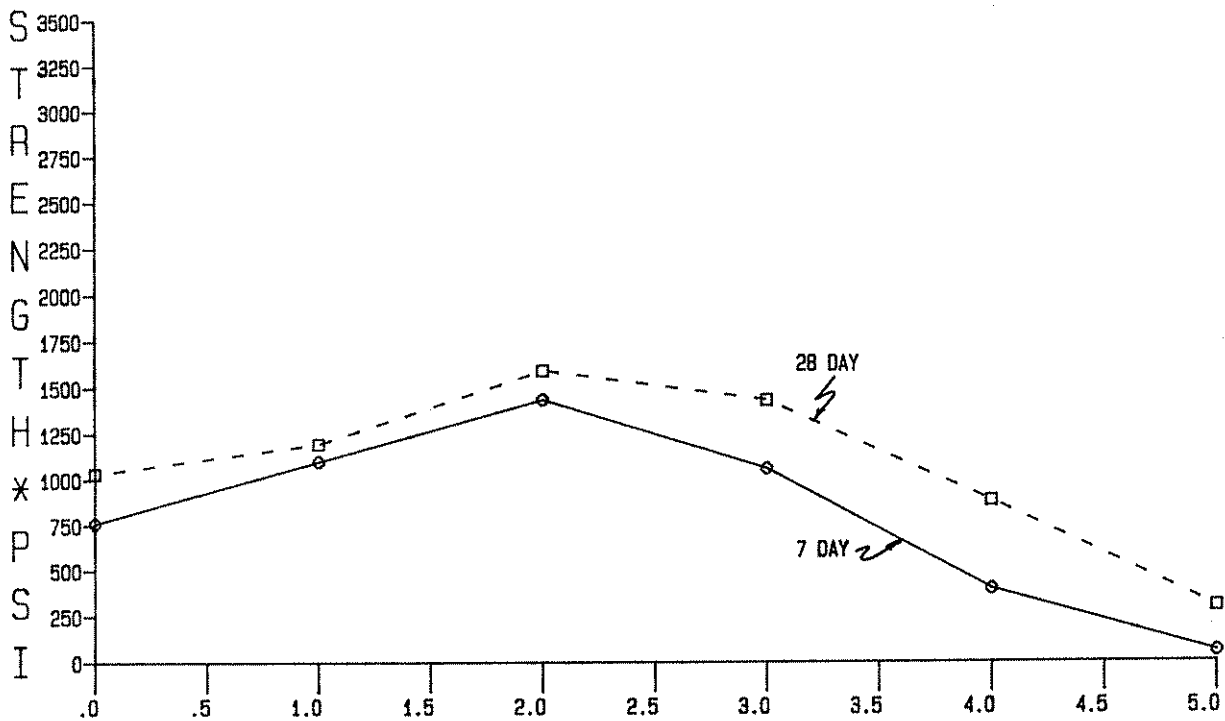
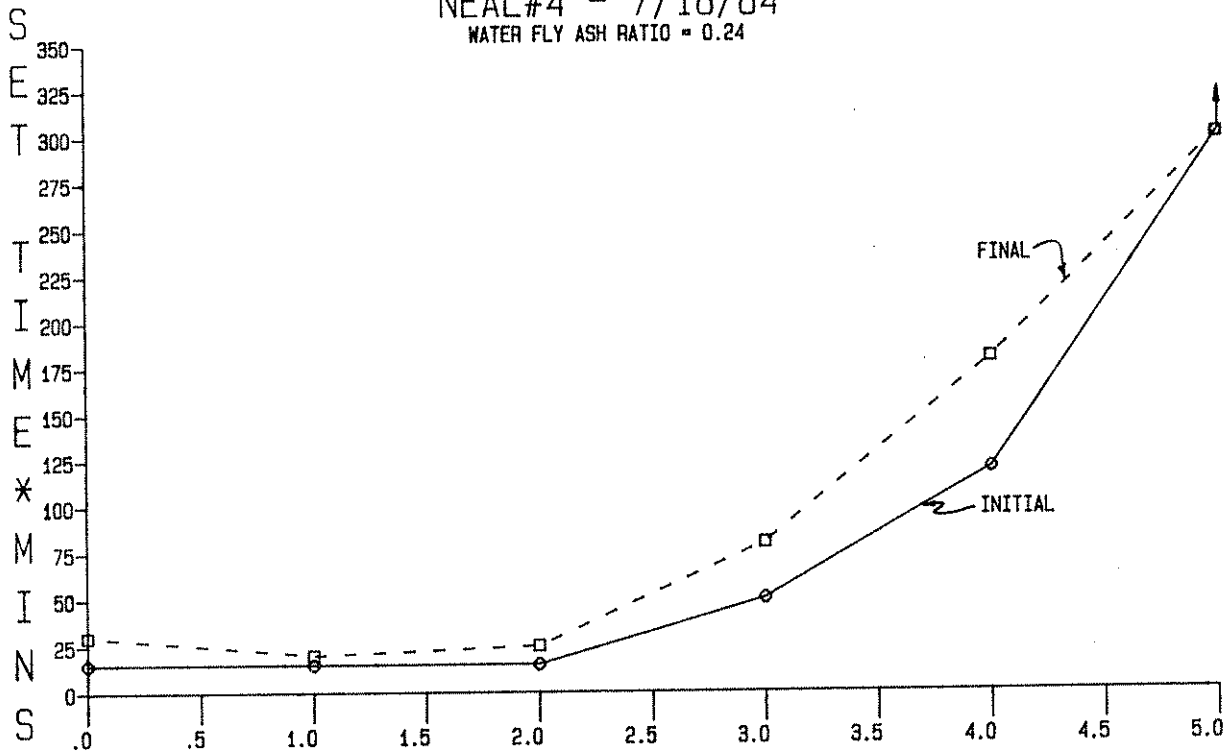


AMMONIUM NITRATE FERTILIZER, PERCENT



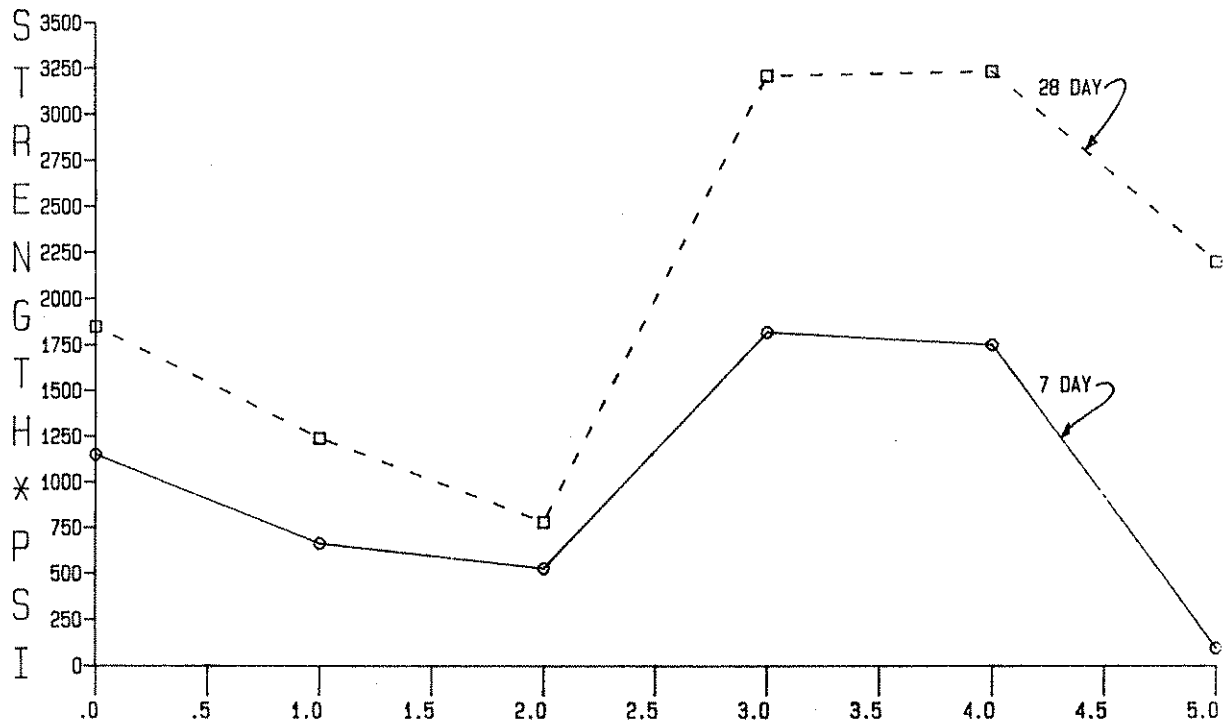
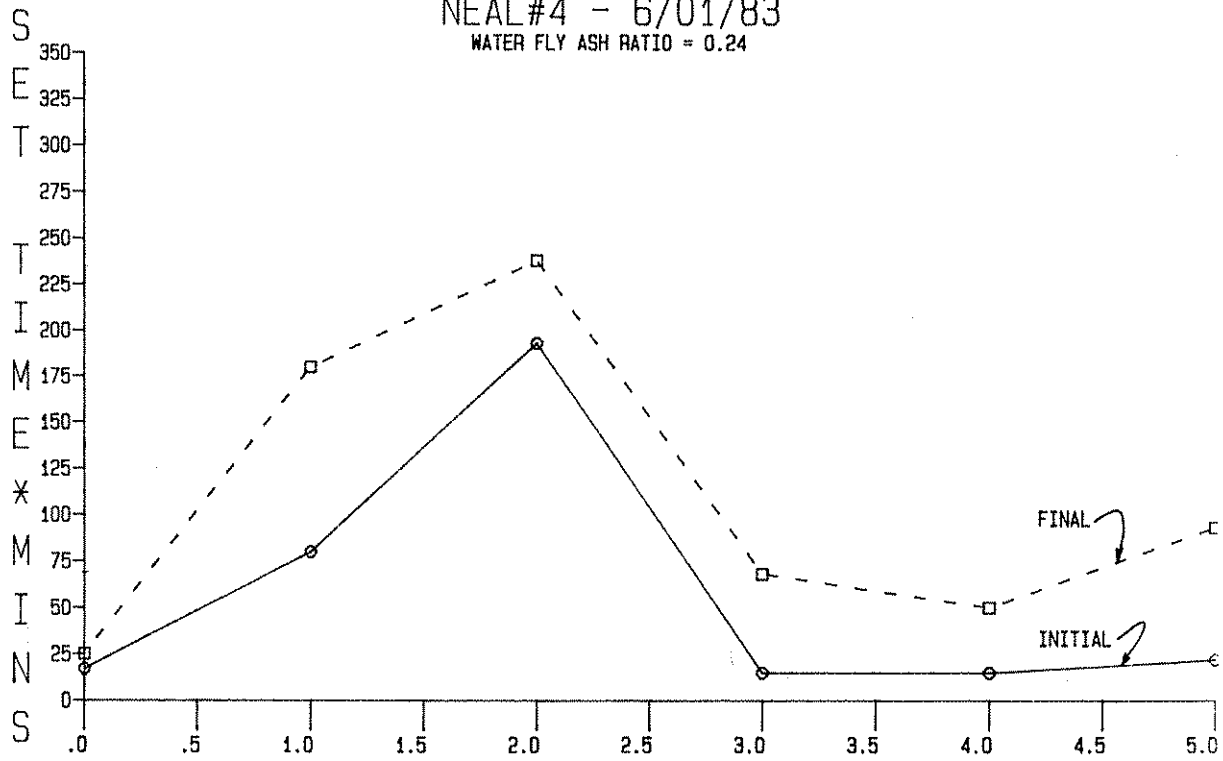
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AMMONIUM NITRATE FERTILIZER, PERCENT

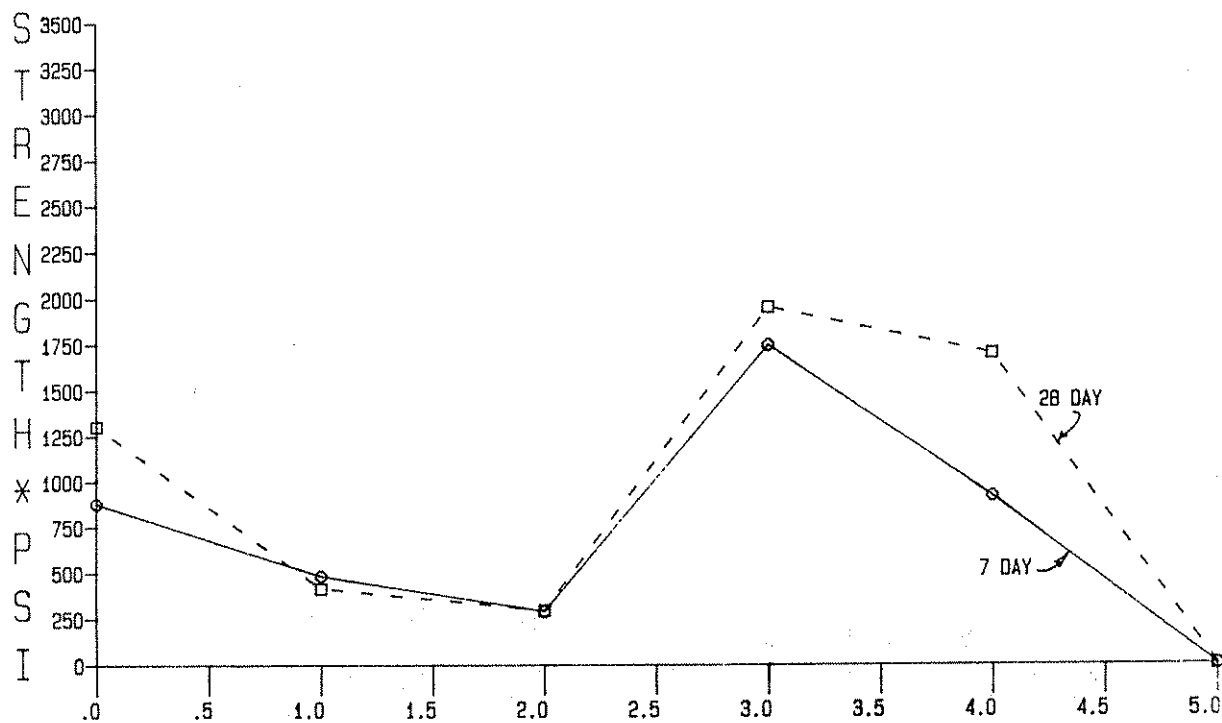
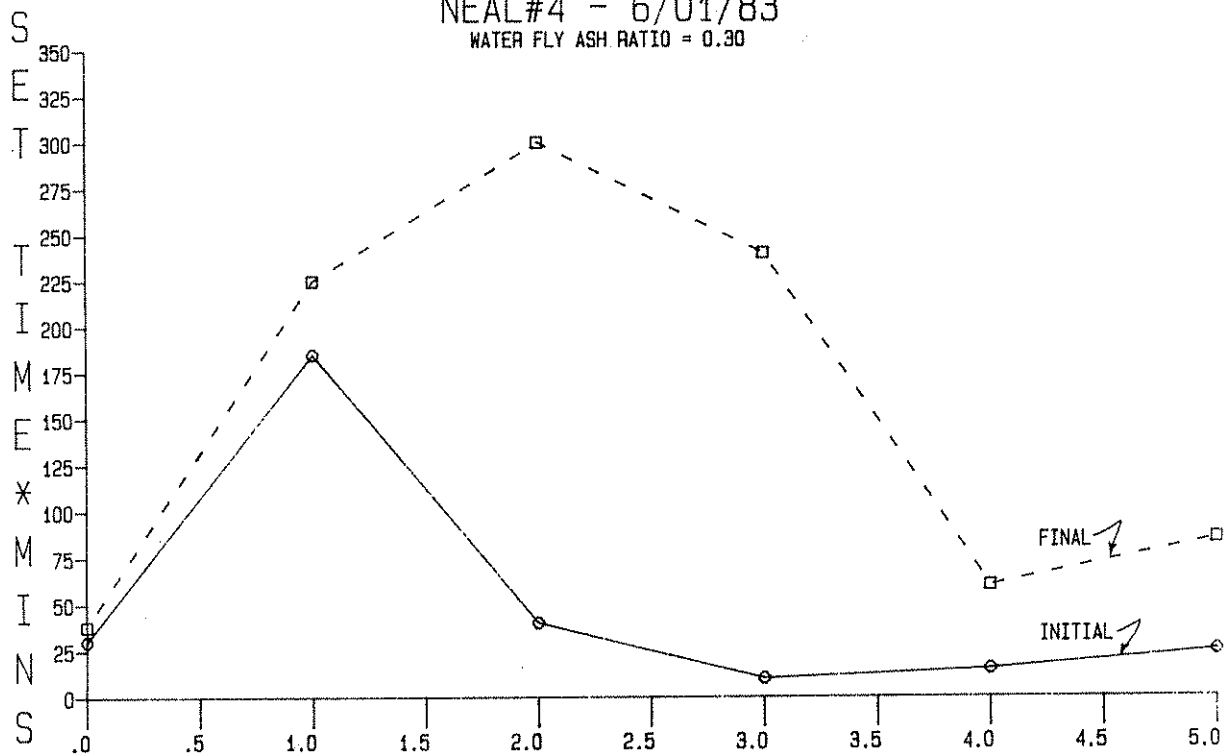
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DIBASIC AMMONIUM PHOSPHATE REAGENT, PERCENT

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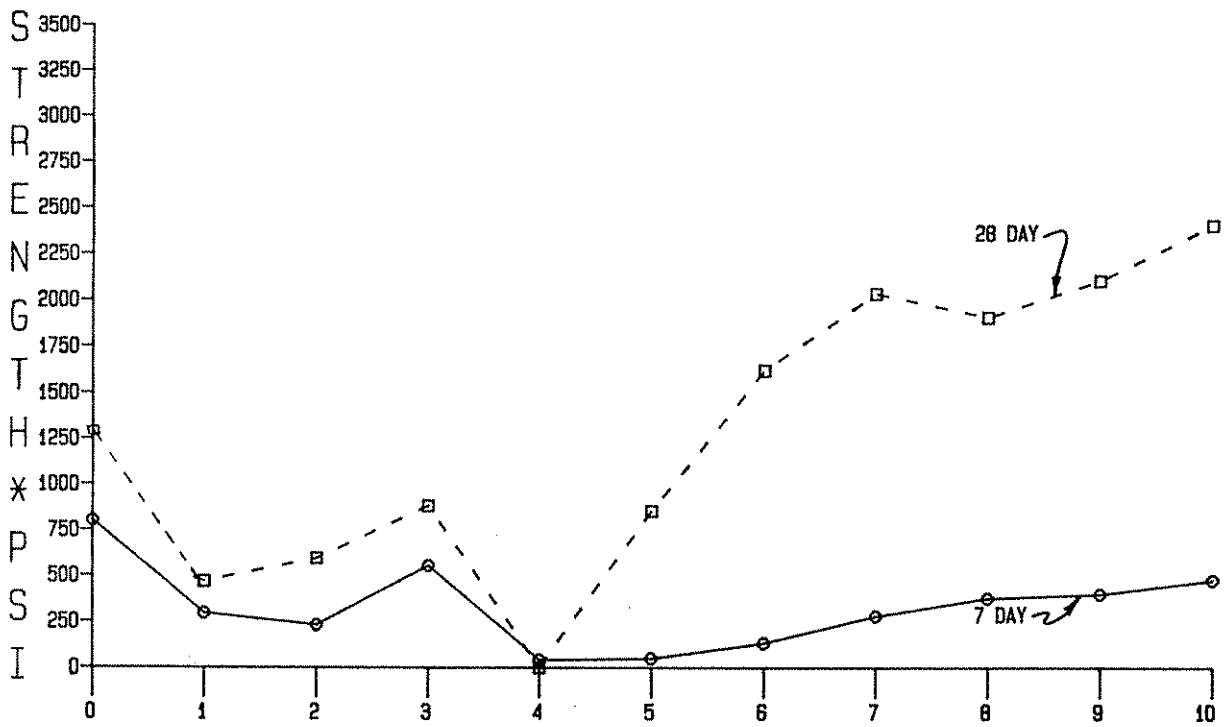
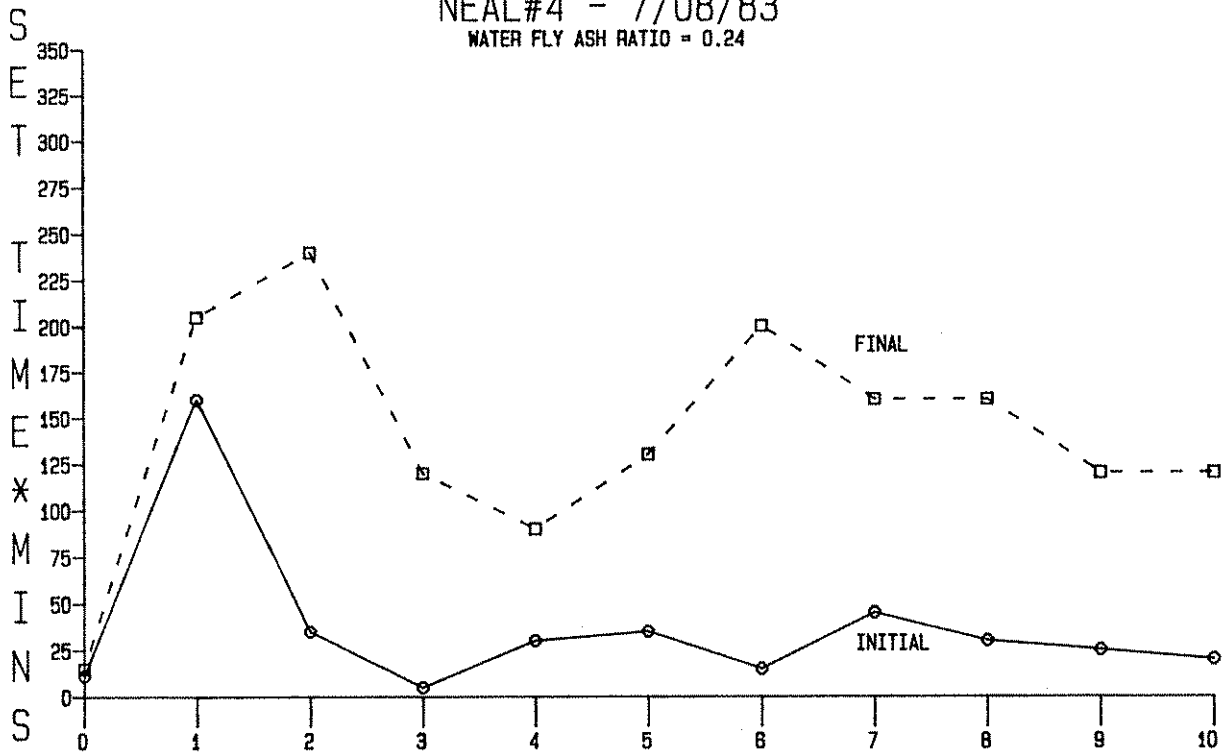


DIBASIC AMMONIUM PHOSPHATE REAGENT, PERCENT

B-11

NEAL#4 - 7/08/83

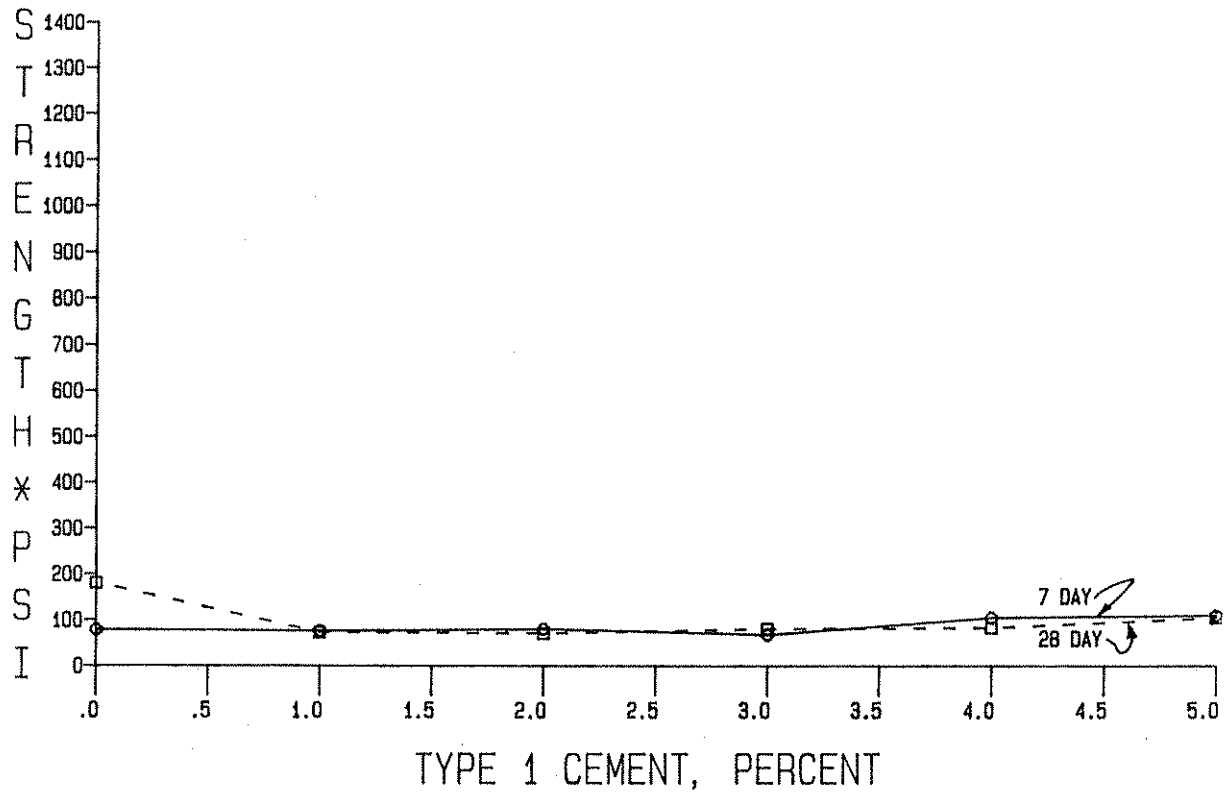
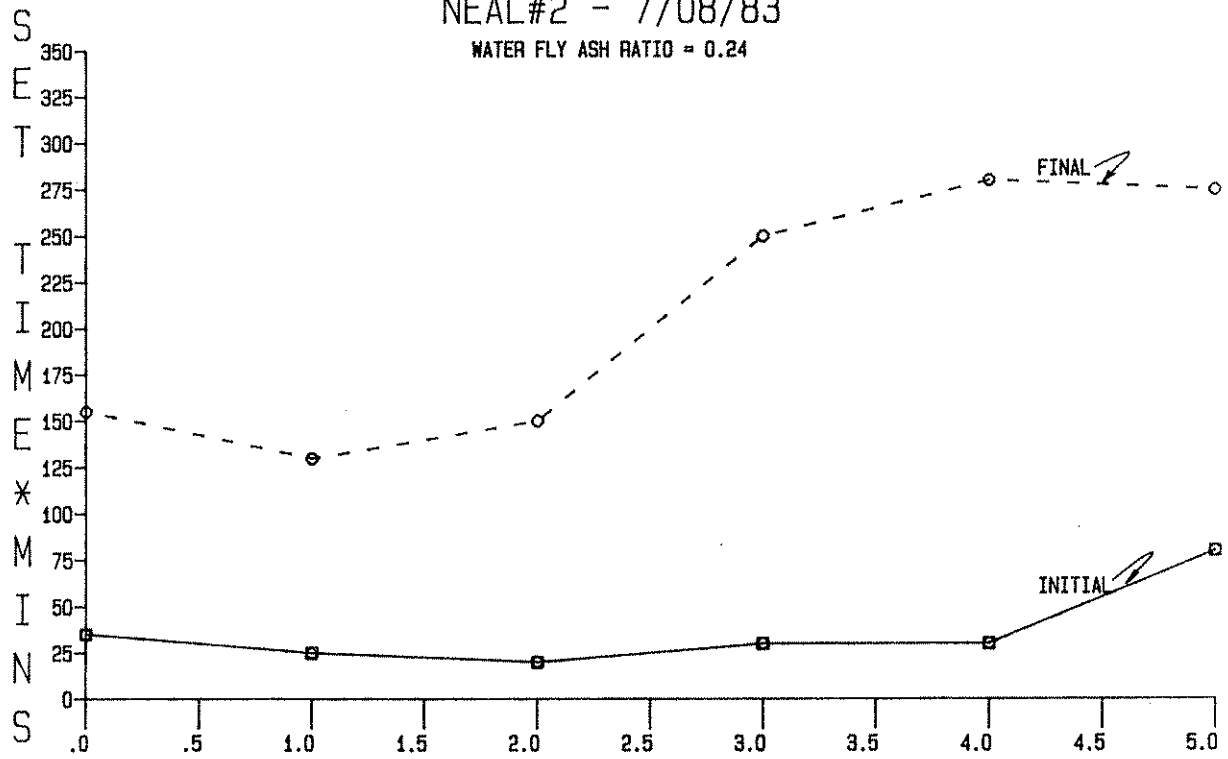
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DIBASIC AMMONIUM PHOSPHATE FERTILIZER, PERCENT

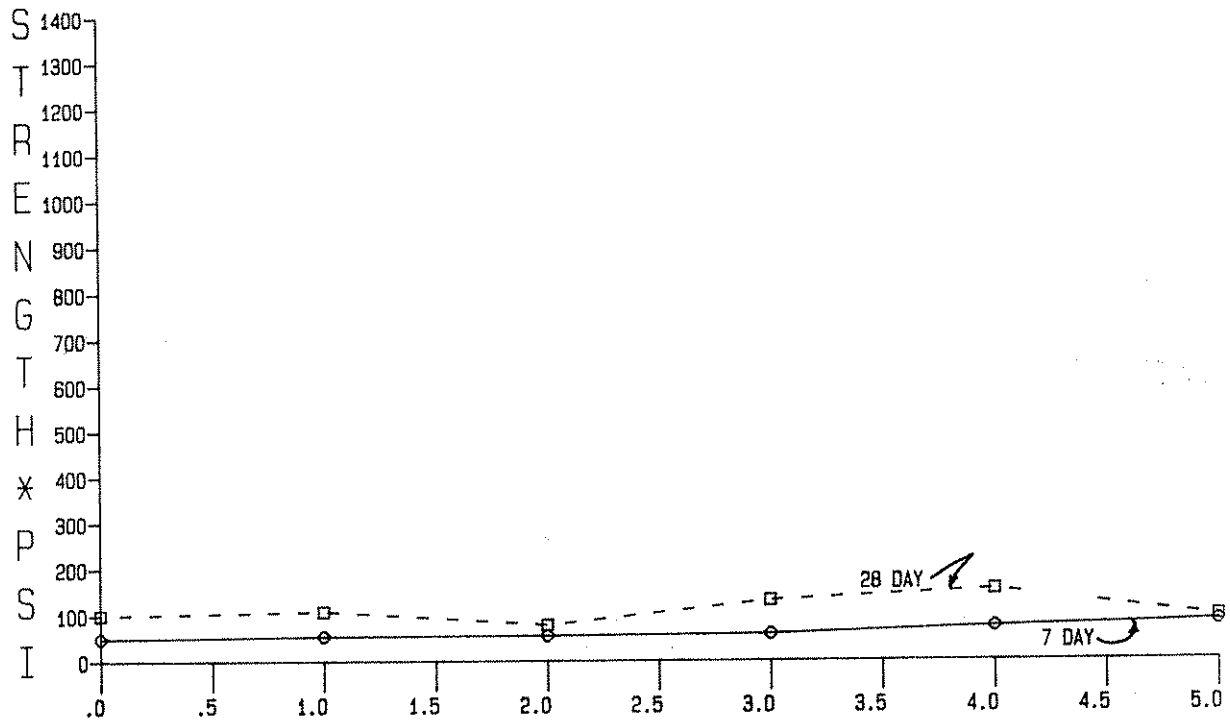
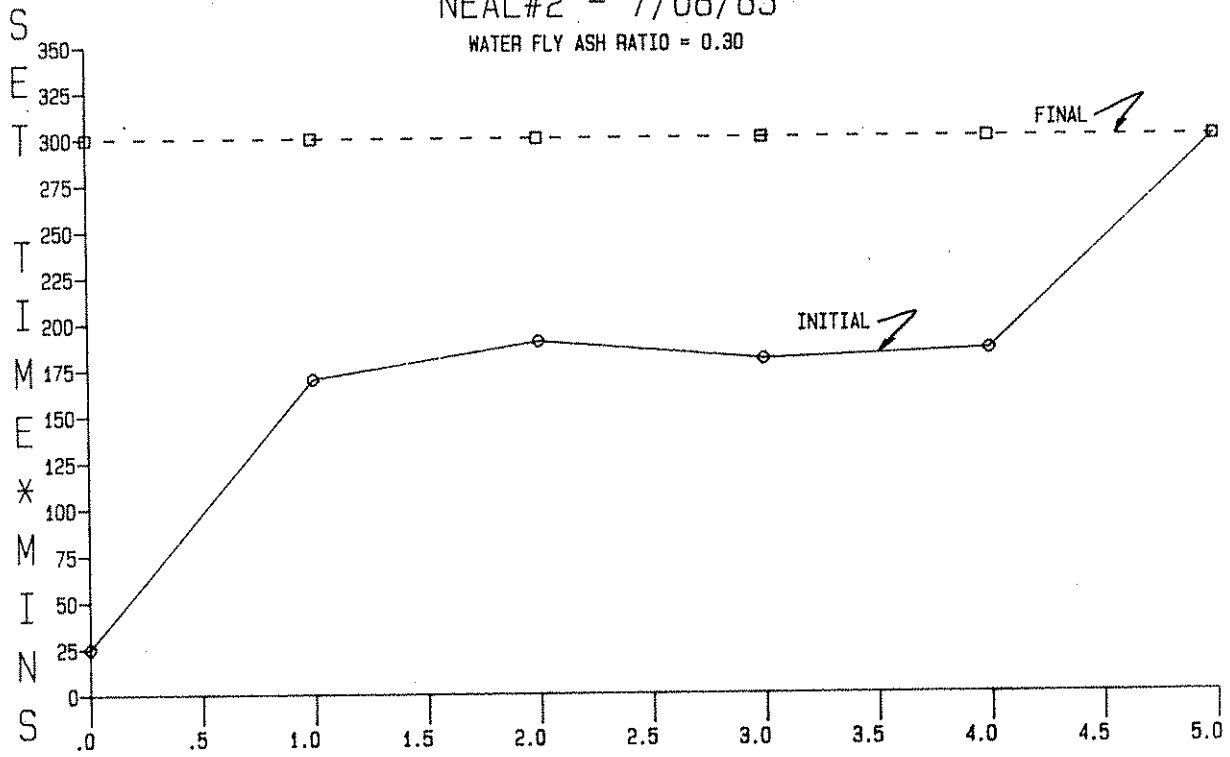
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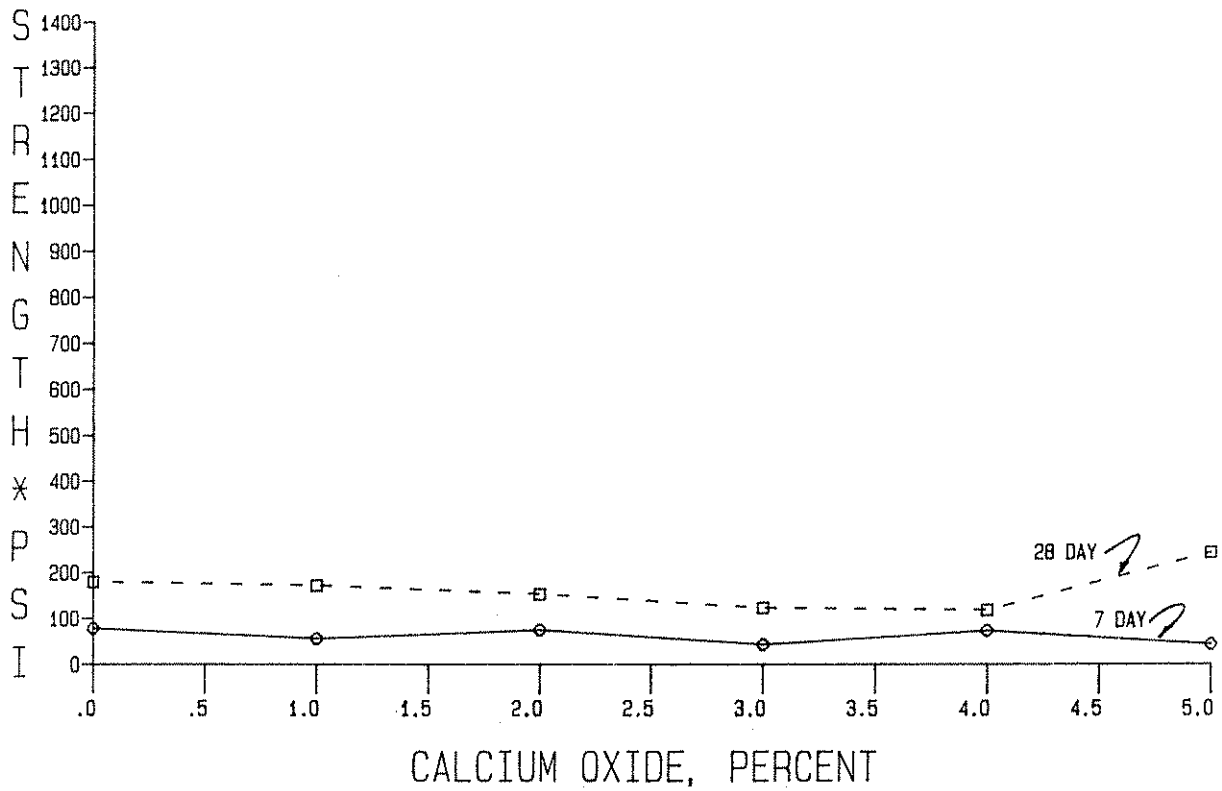
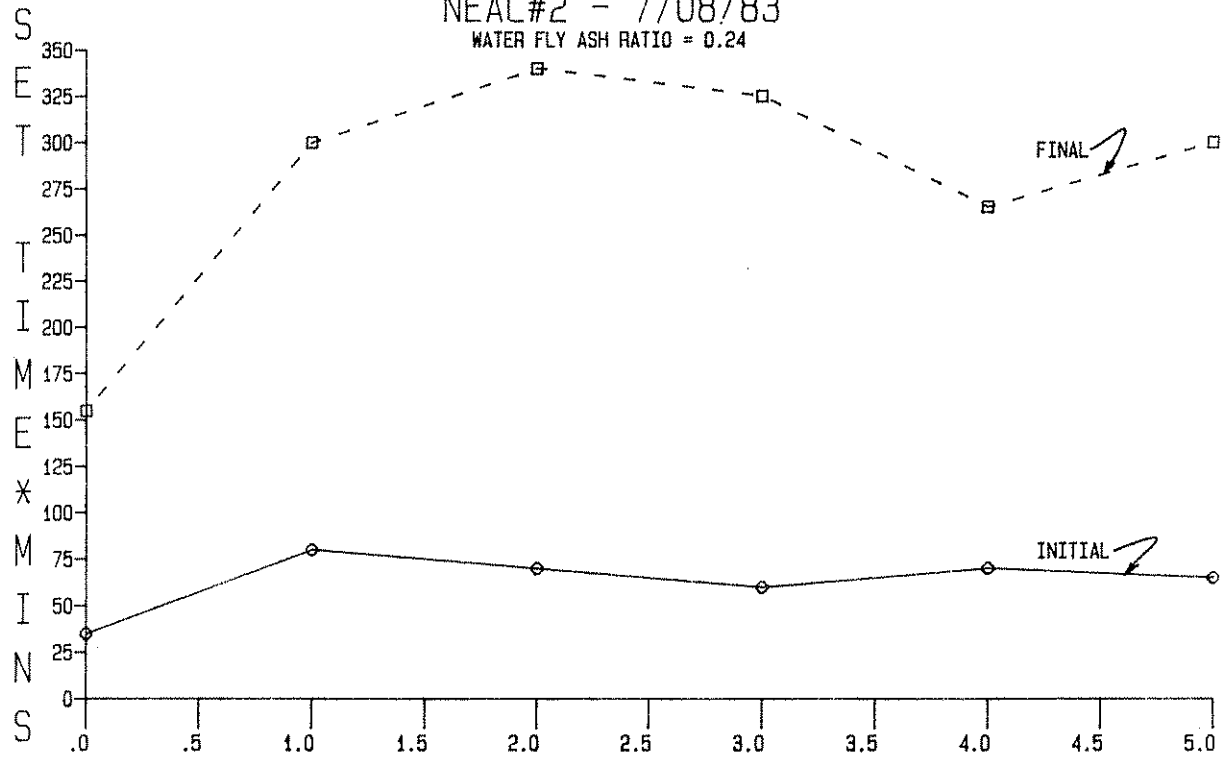
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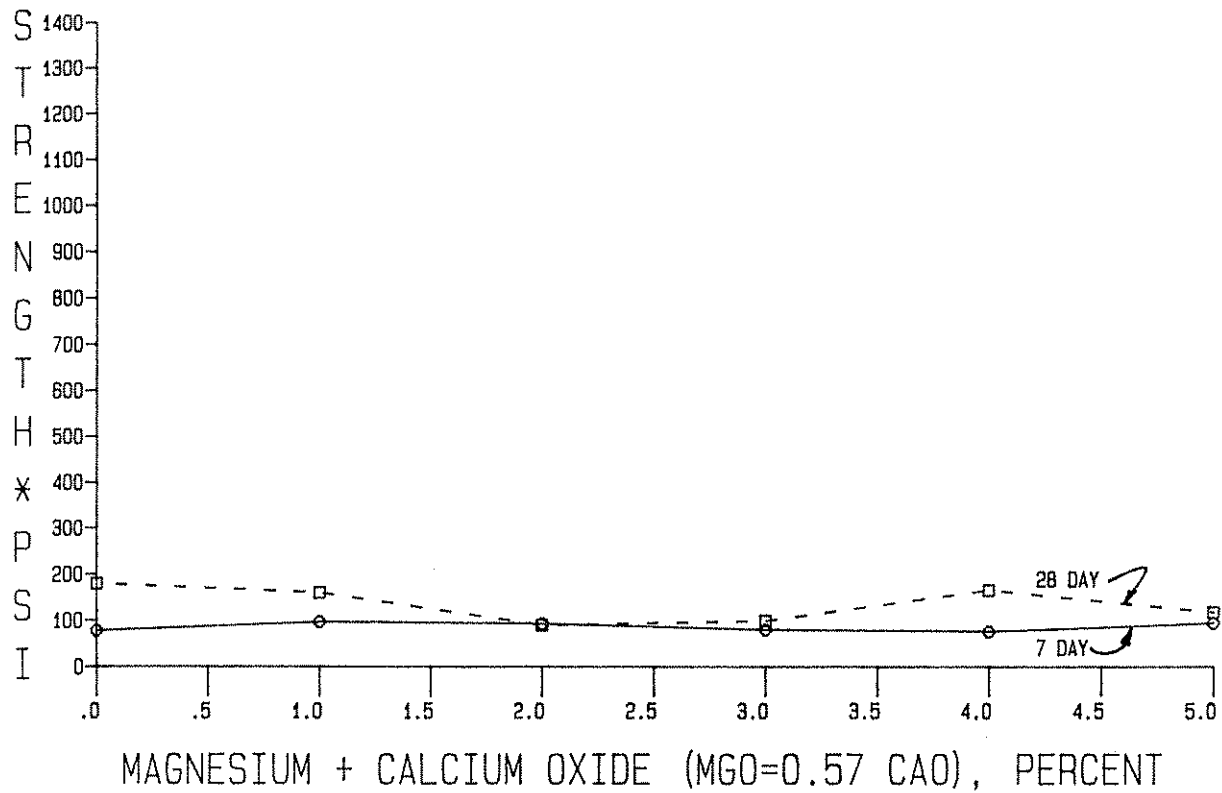
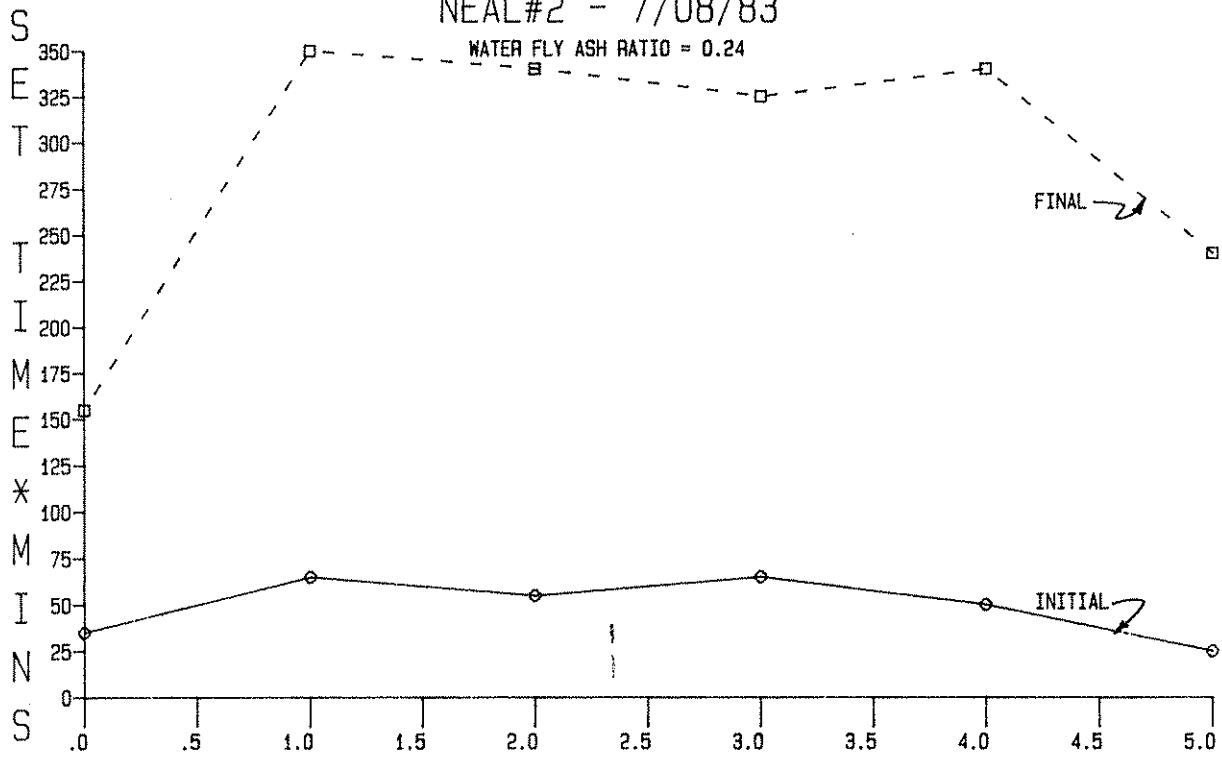
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NEAL#2 - 7/08/83

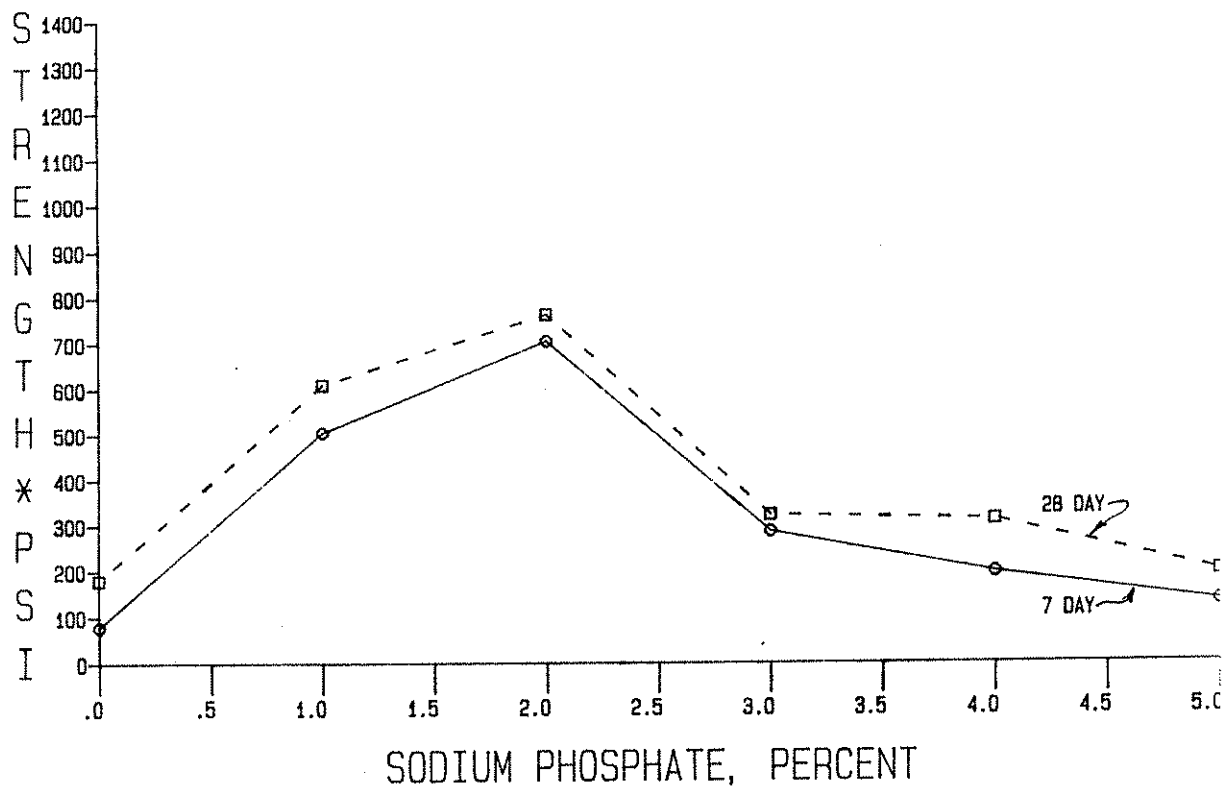
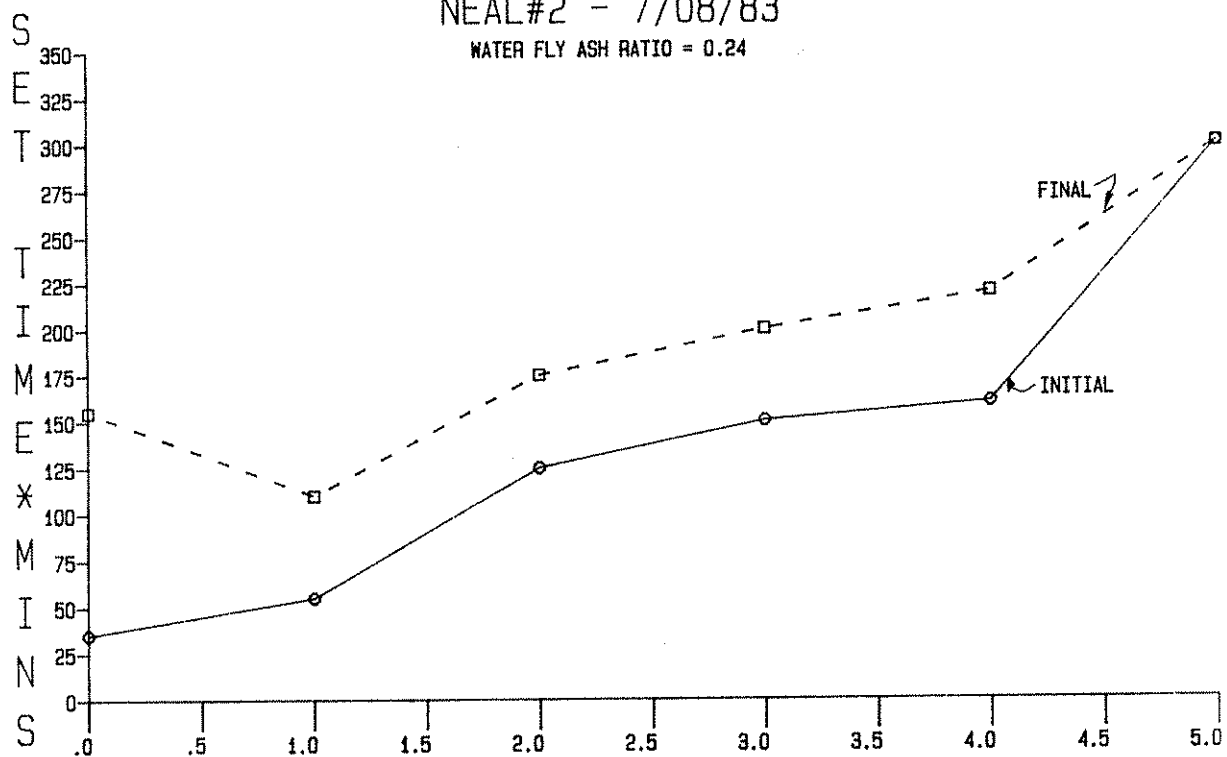
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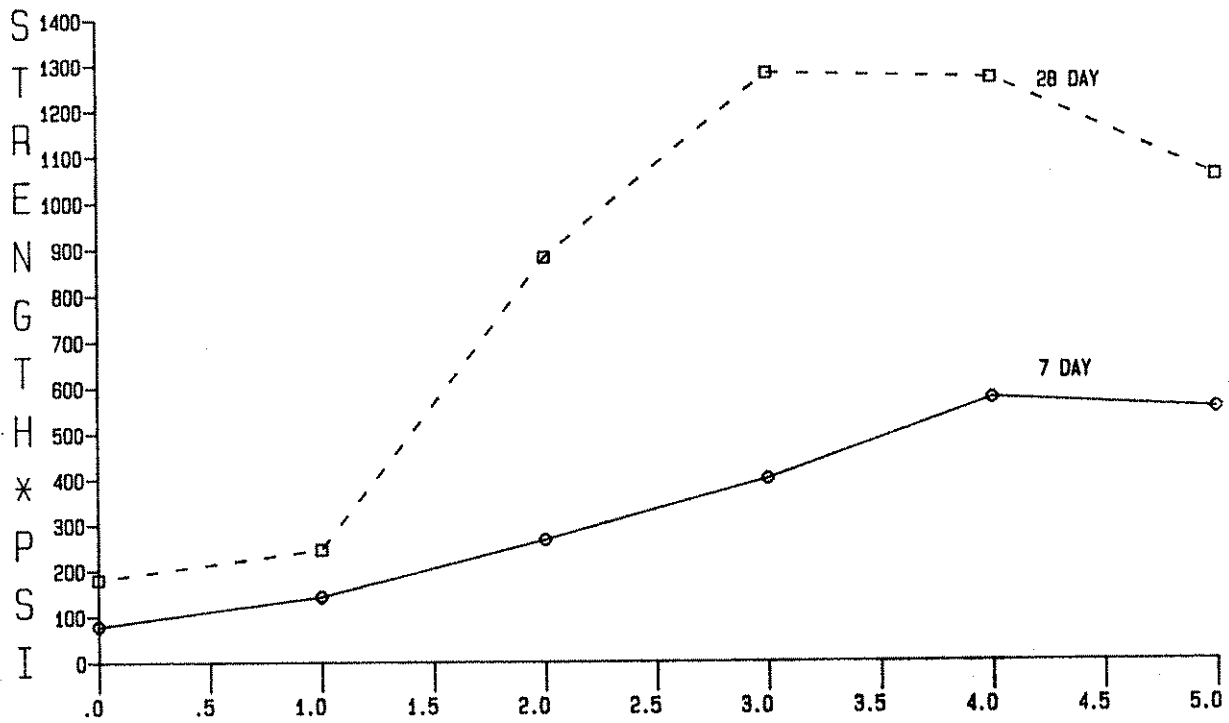
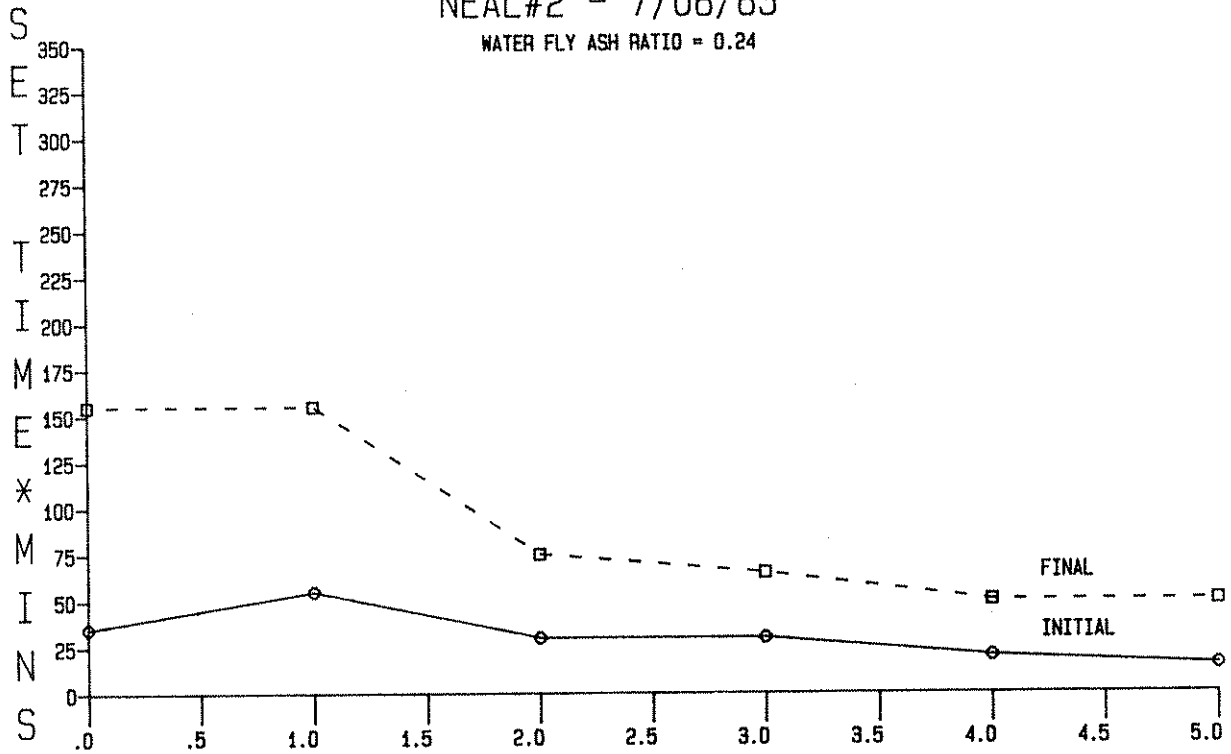
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WATER FLY ASH RATIO = 0.24



NEAL#2 - 7/08/83

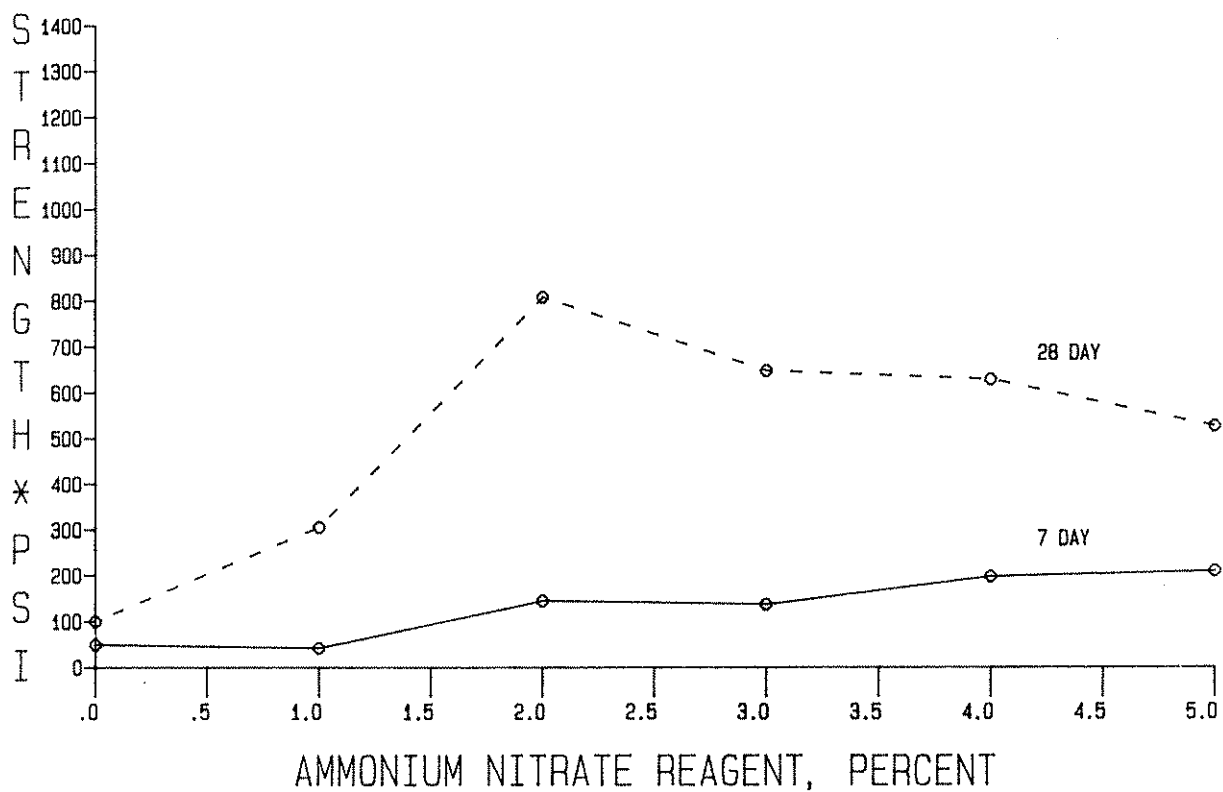
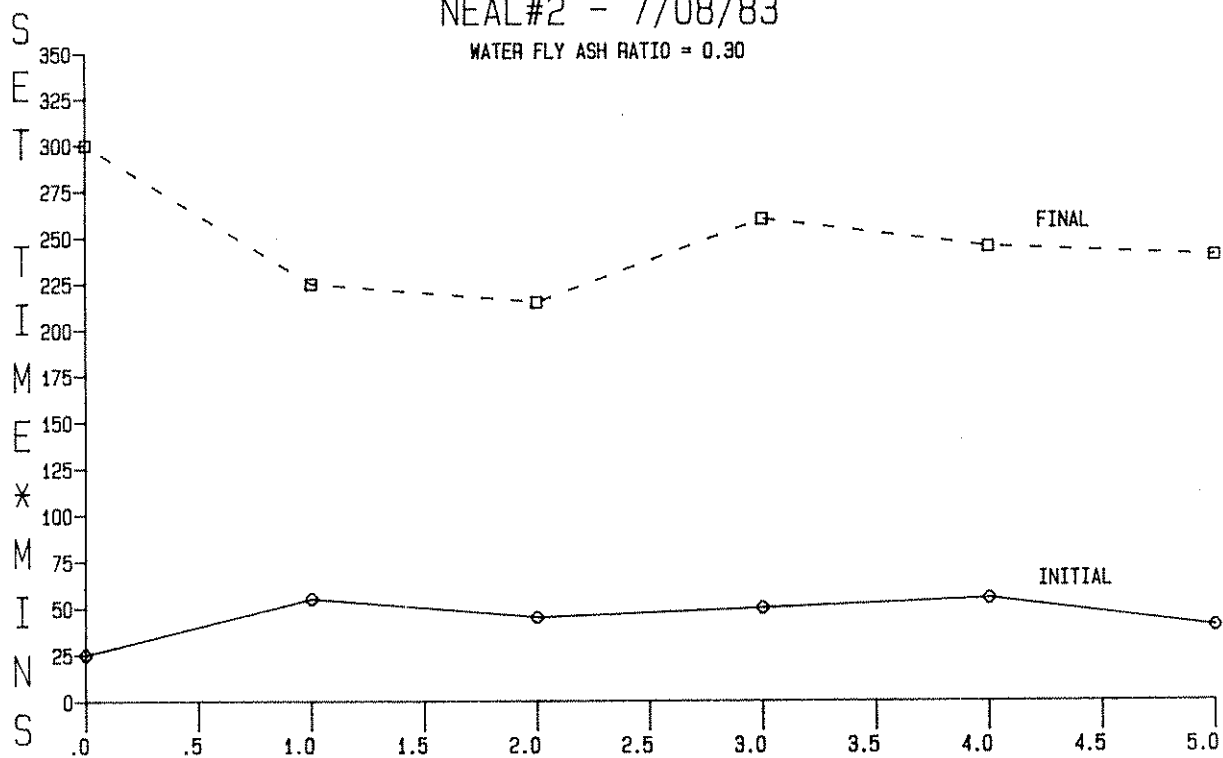
WATER FLY ASH RATIO = 0.24



AMMONIUM NITRATE REAGENT, PERCENT

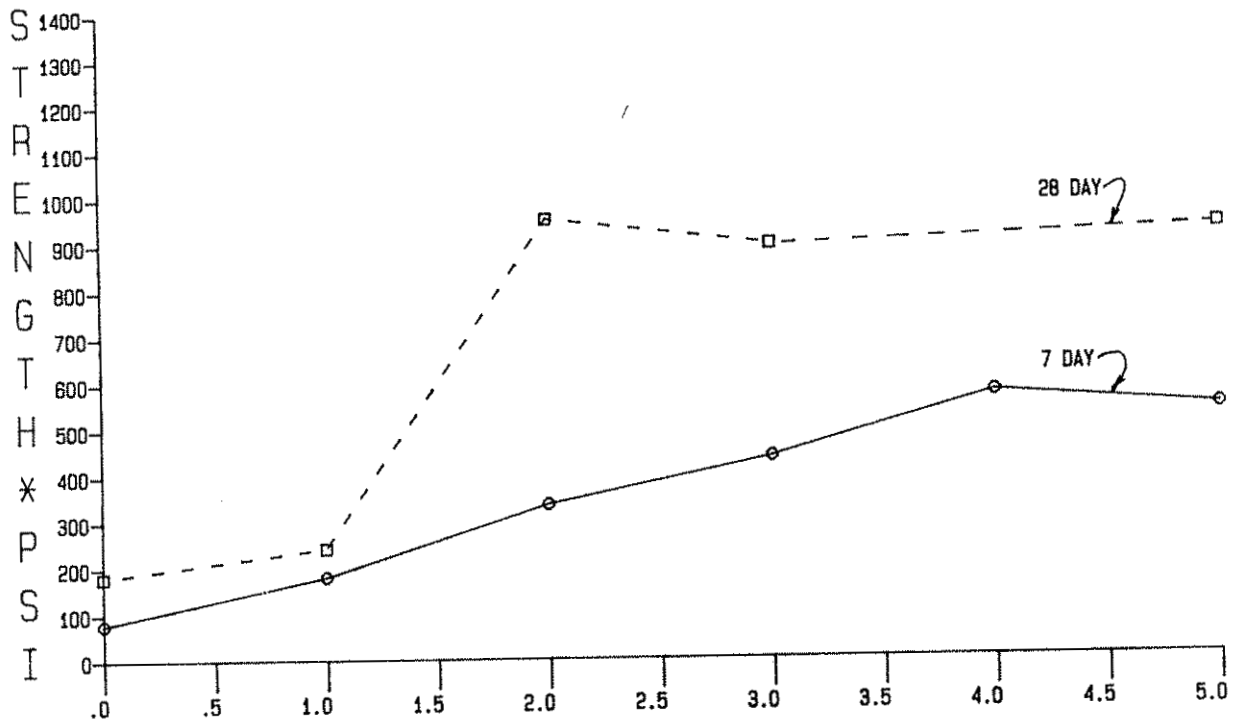
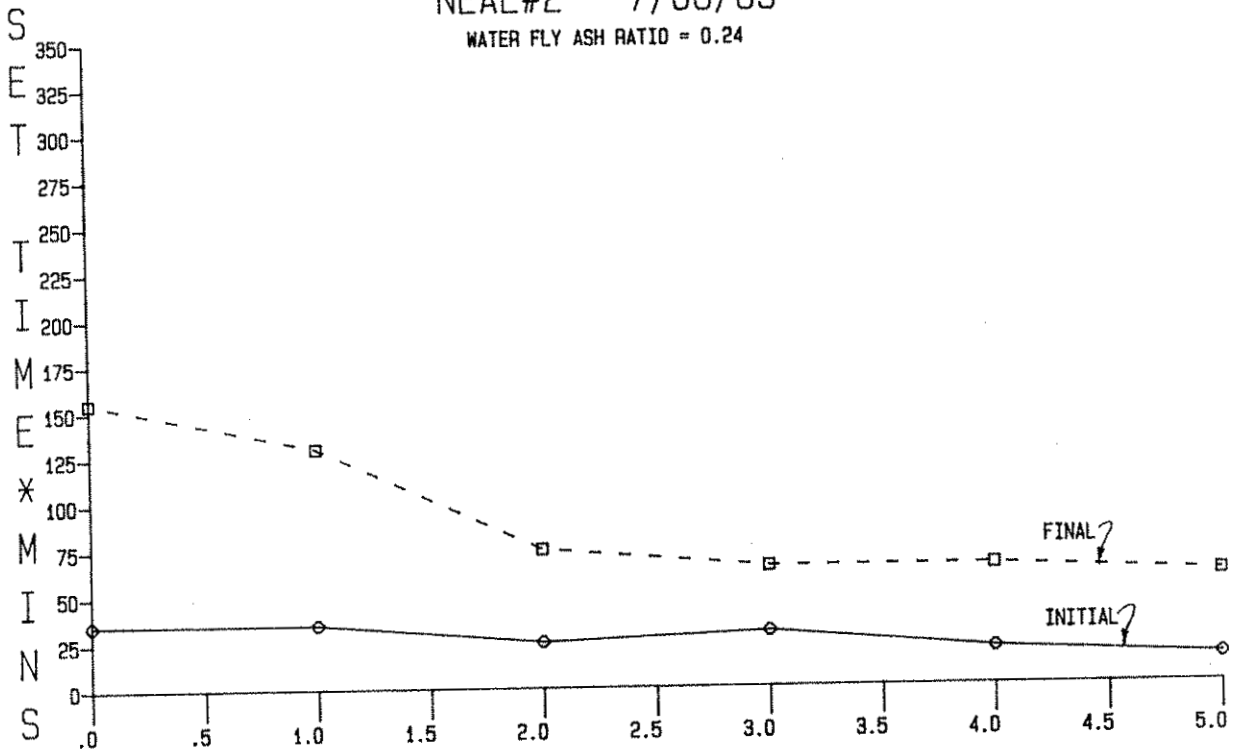
NEAL#2 - 7/08/83

WATER FLY ASH RATIO = 0.30



NEAL#2 - 7/08/83

WATER FLY ASH RATIO = 0.24

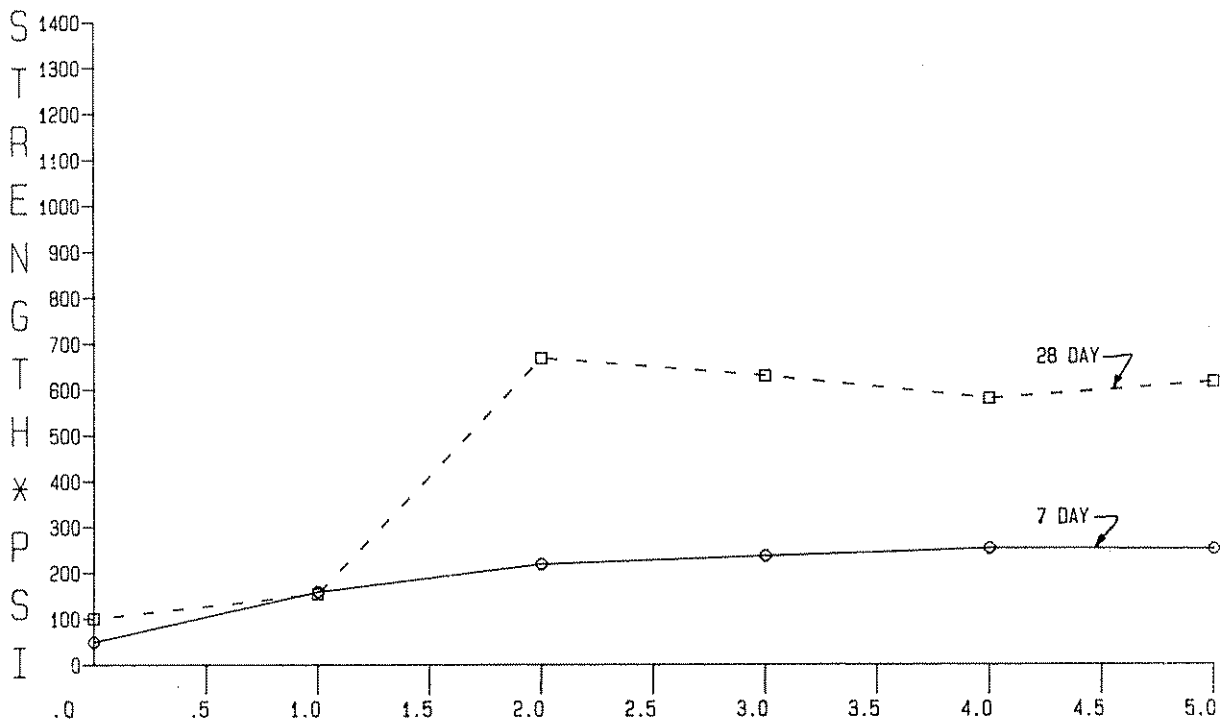
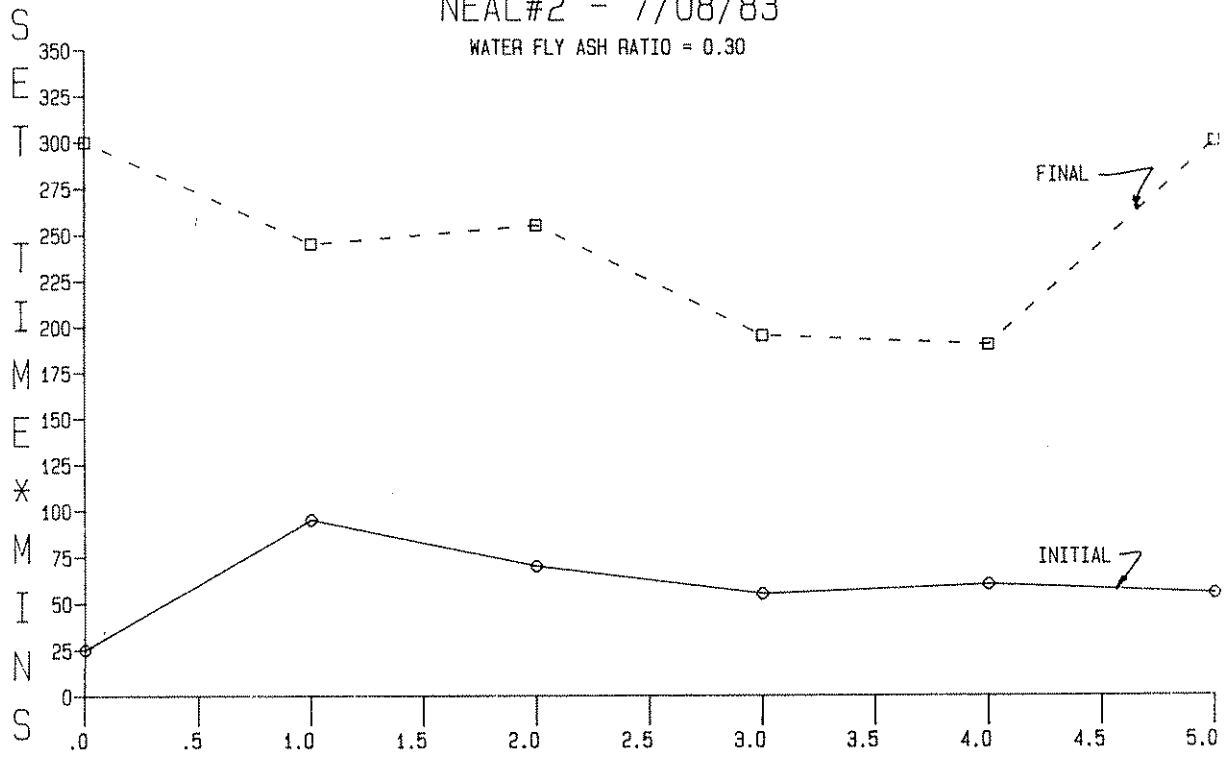


AMMONIUM NITRATE FERTILIZER, PERCENT

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NEAL#2 - 7/08/83

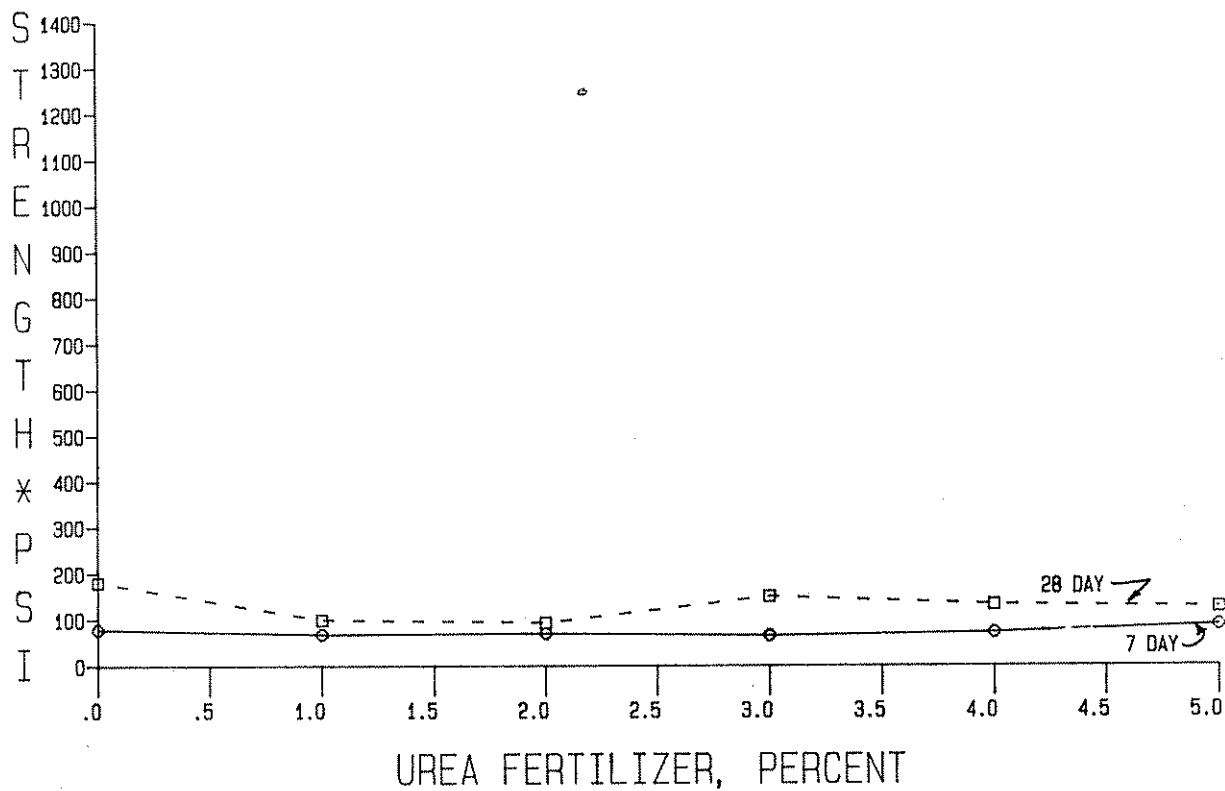
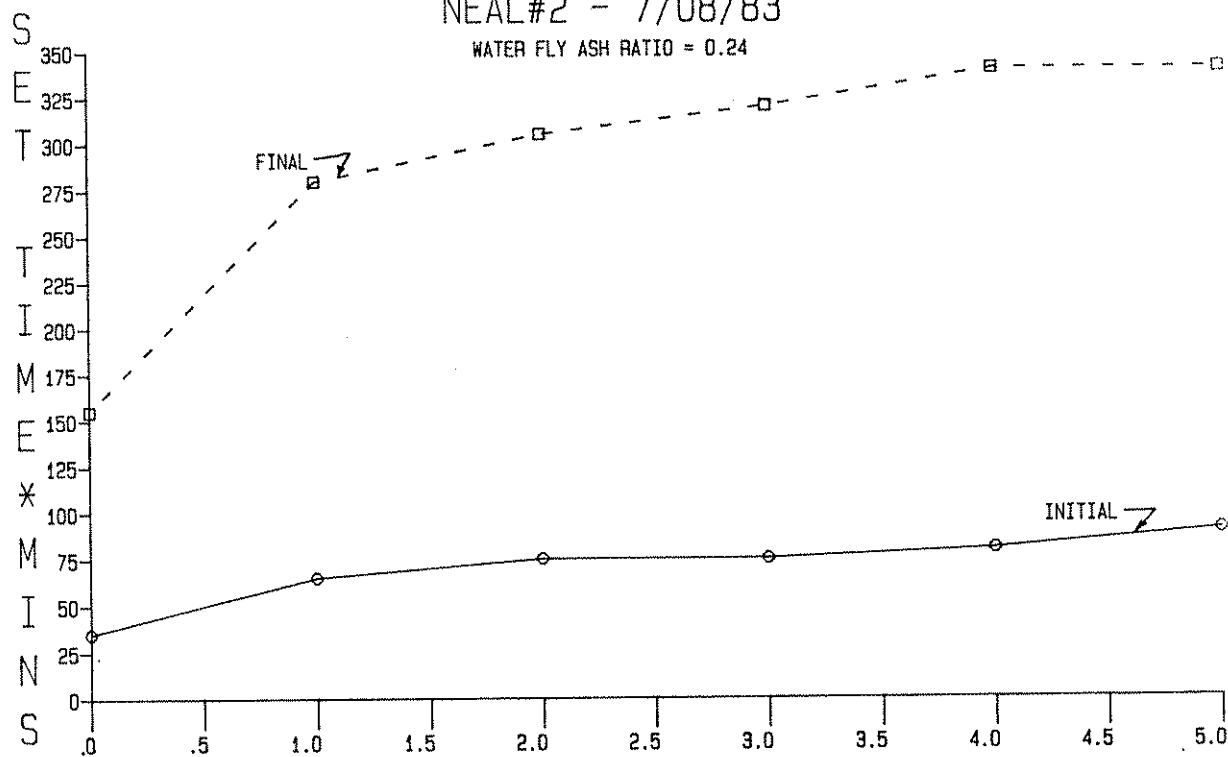
WATER FLY ASH RATIO = 0.30

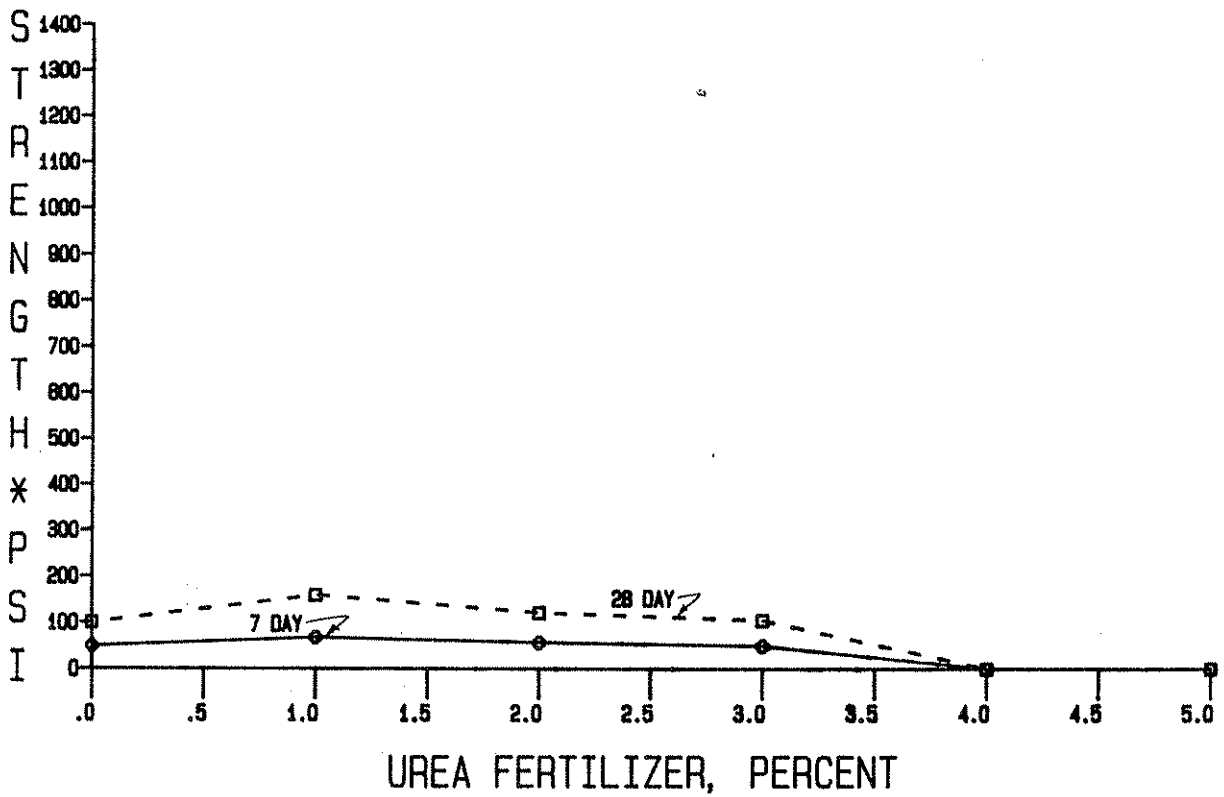
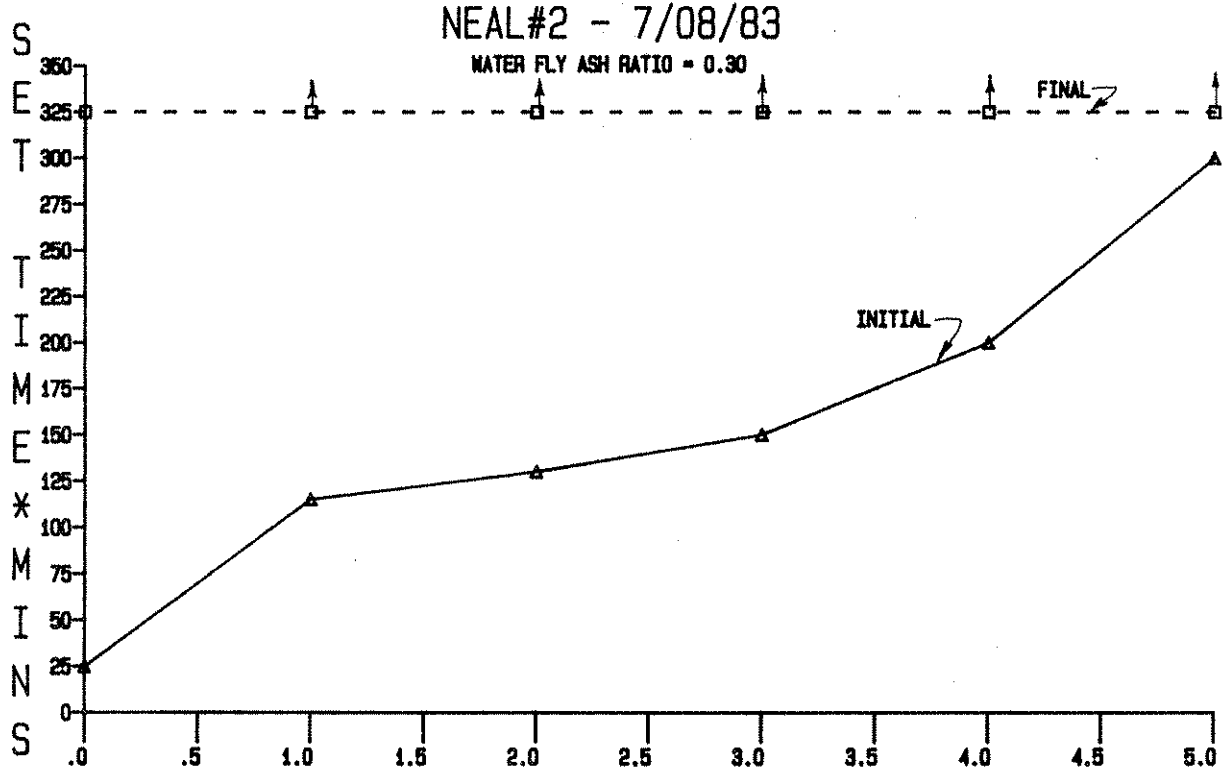


AMMONIUM NITRATE FERTILIZER, PERCENT

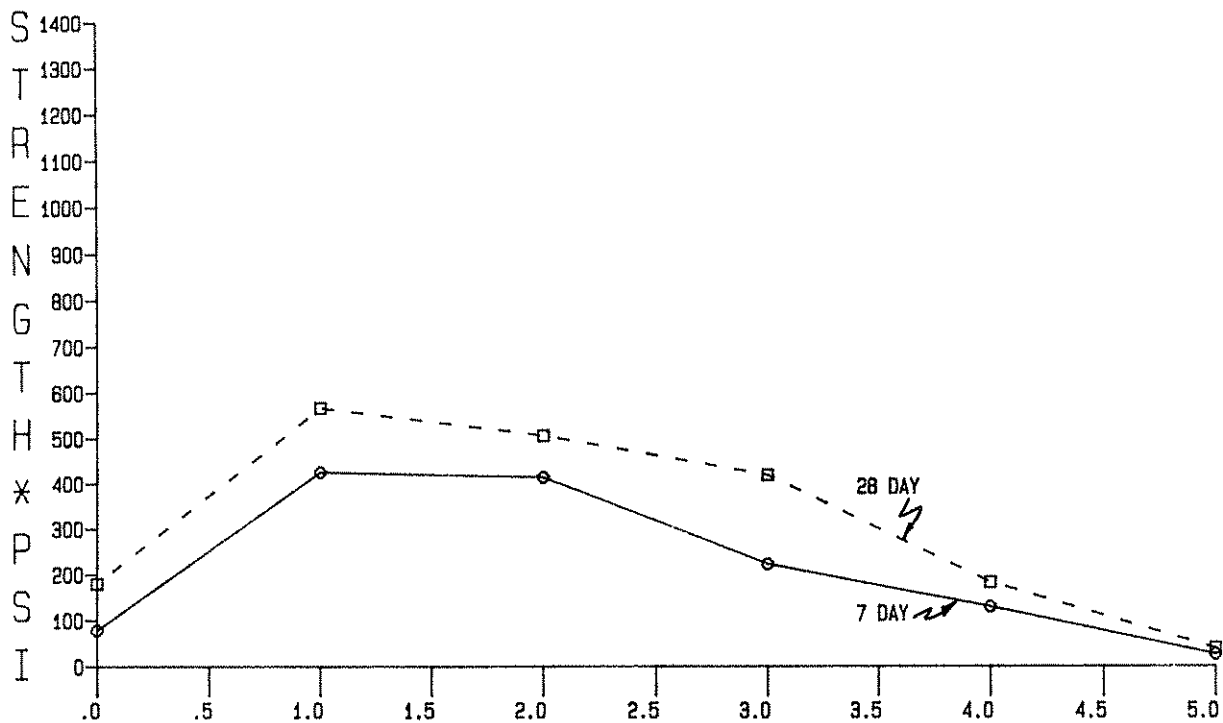
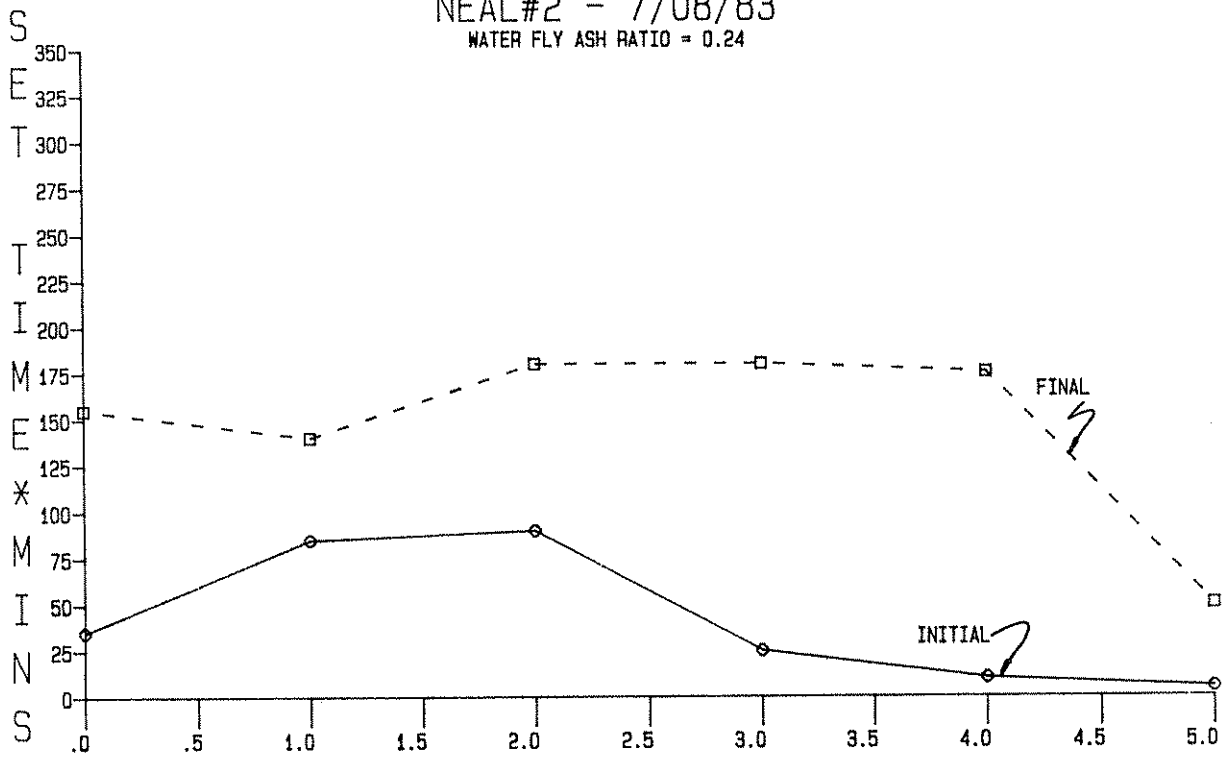
NEAL#2 - 7/08/83

WATER FLY ASH RATIO = 0.24



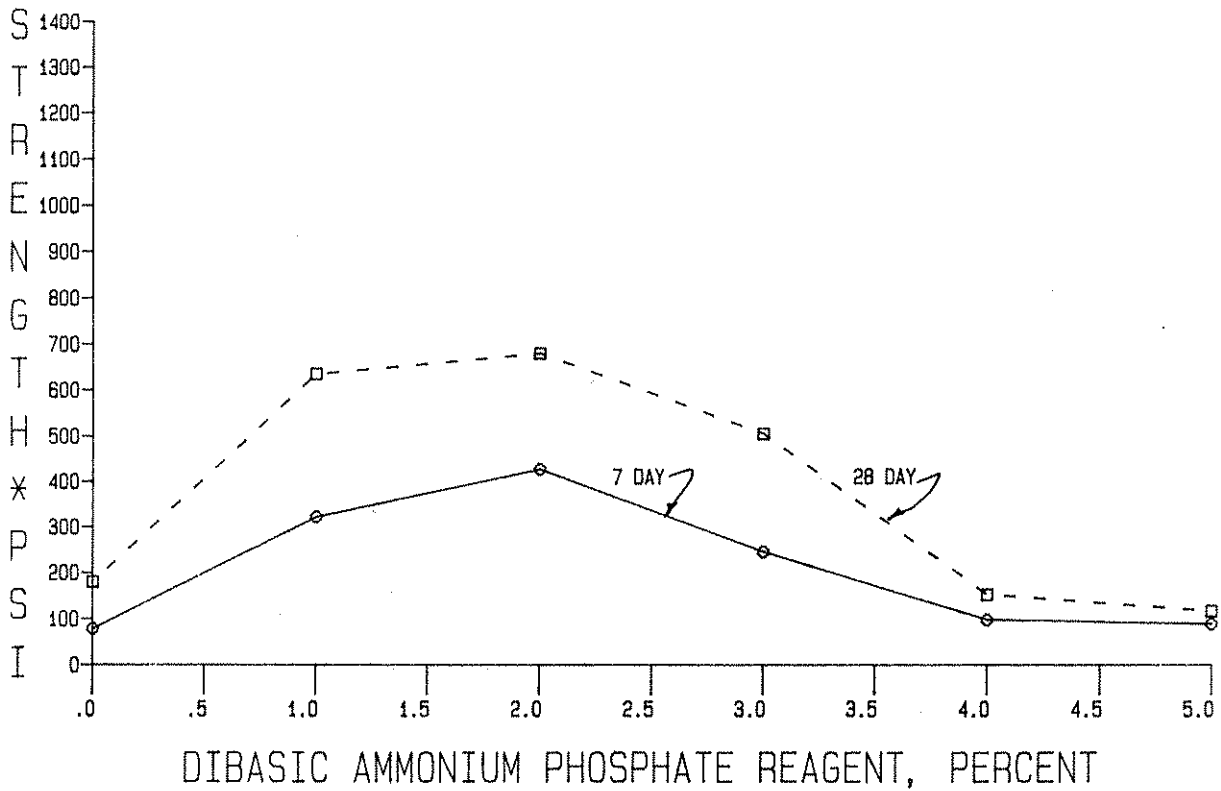
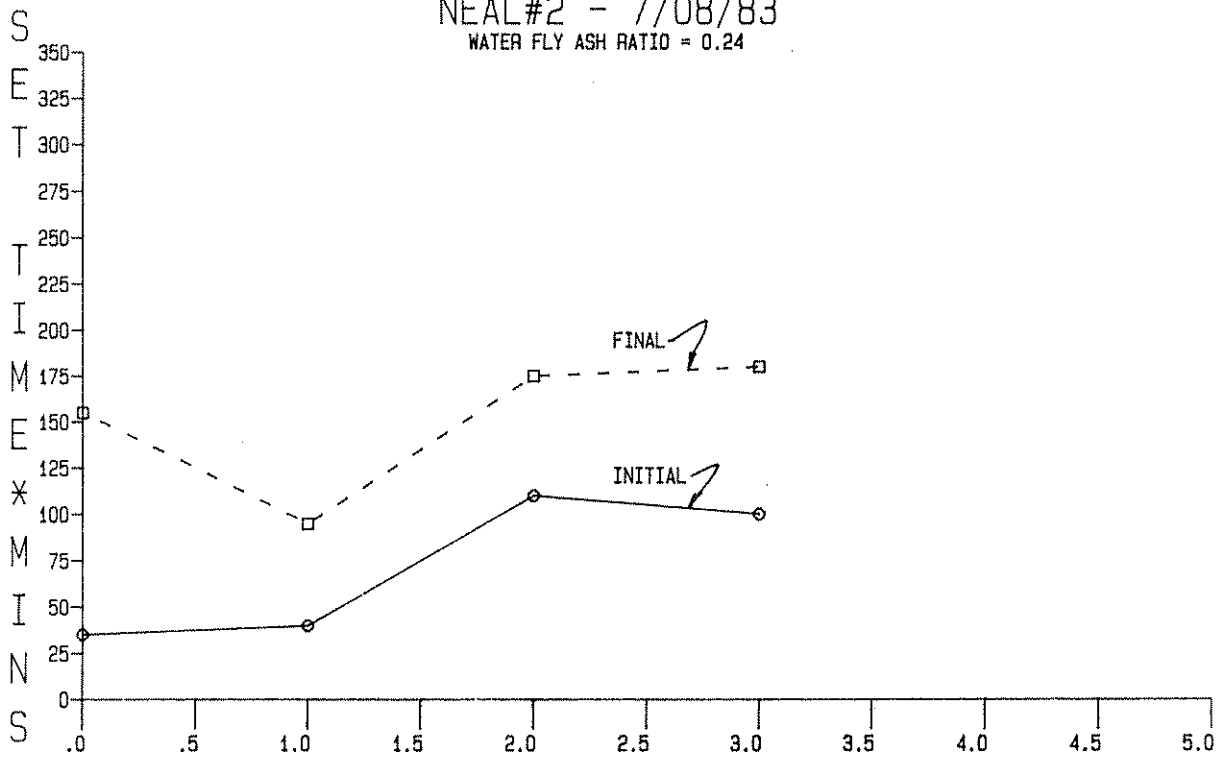


NEAL#2 - 7/08/83  
WATER FLY ASH RATIO = 0.24



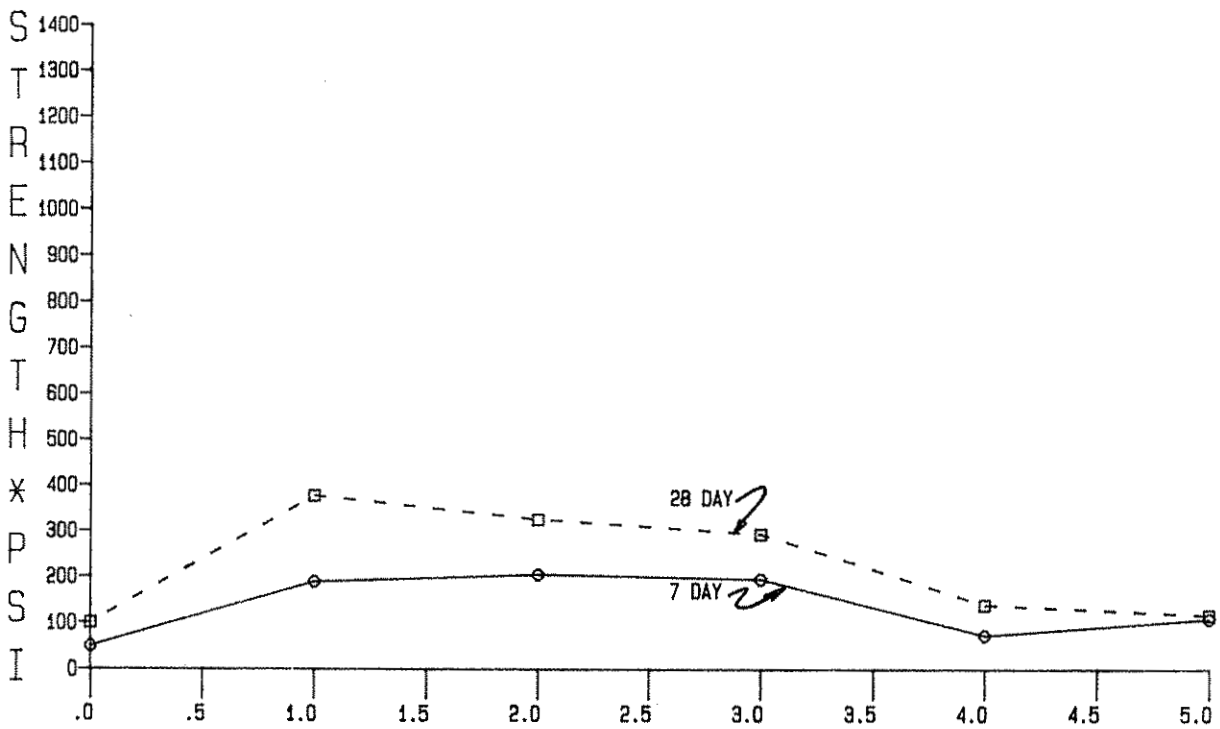
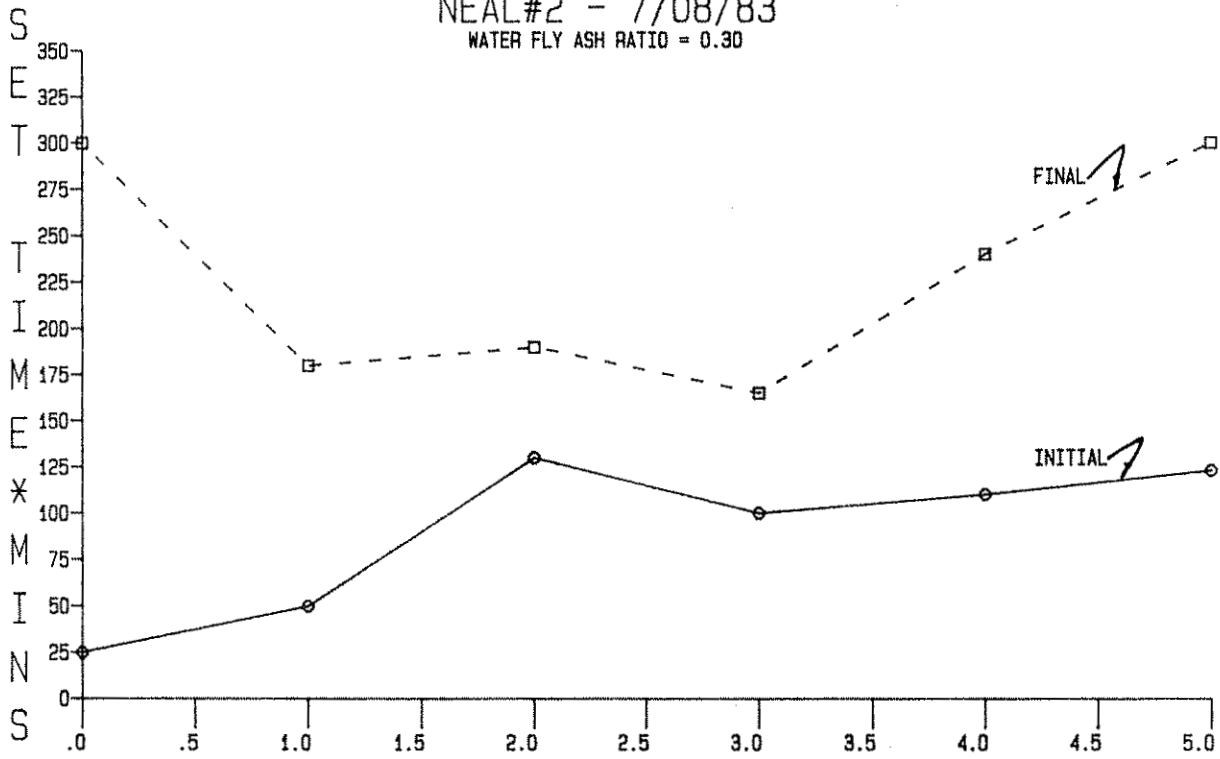
MONOBASIC AMMONIUM PHOSPHATE REAGENT, PERCENT



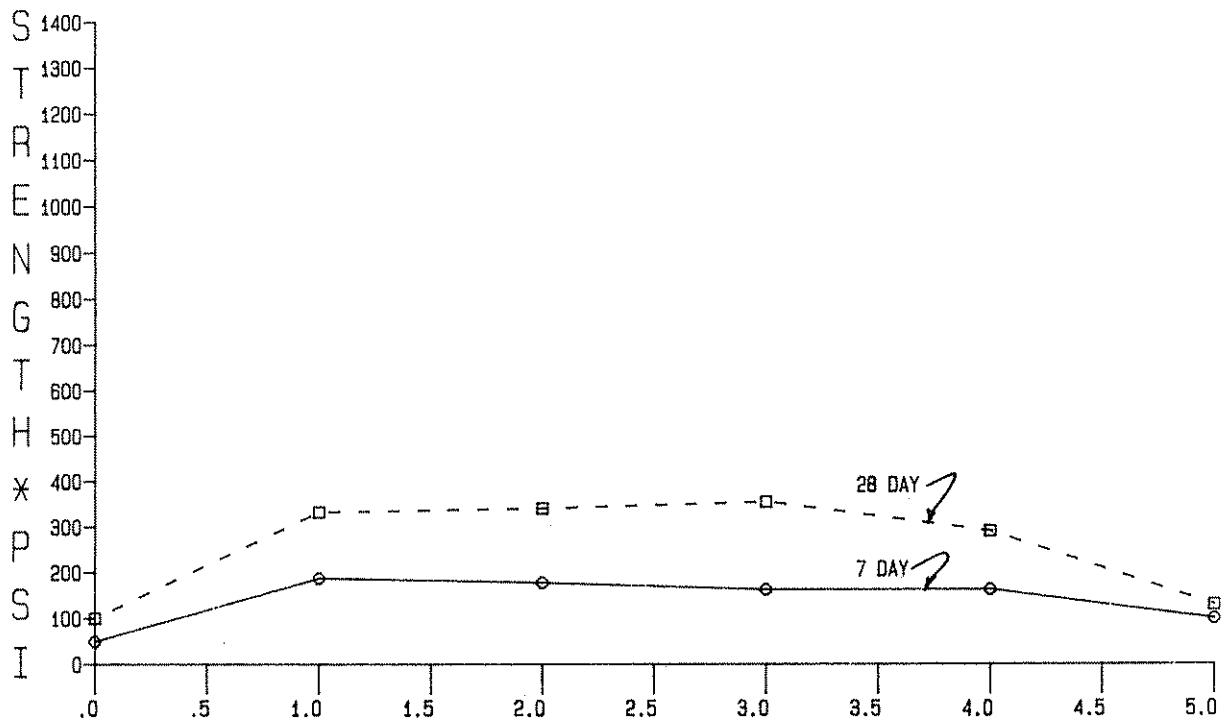
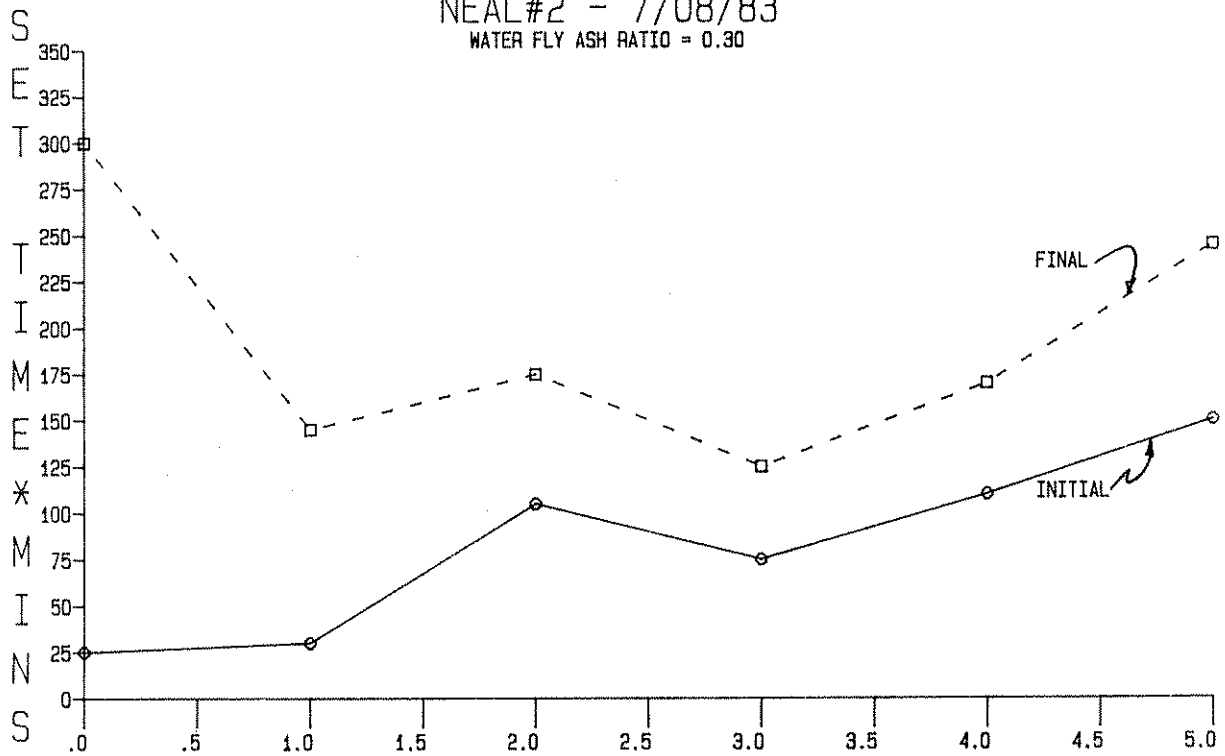
NEAL#2 - 7/08/83  
WATER FLY ASH RATIO = 0.24

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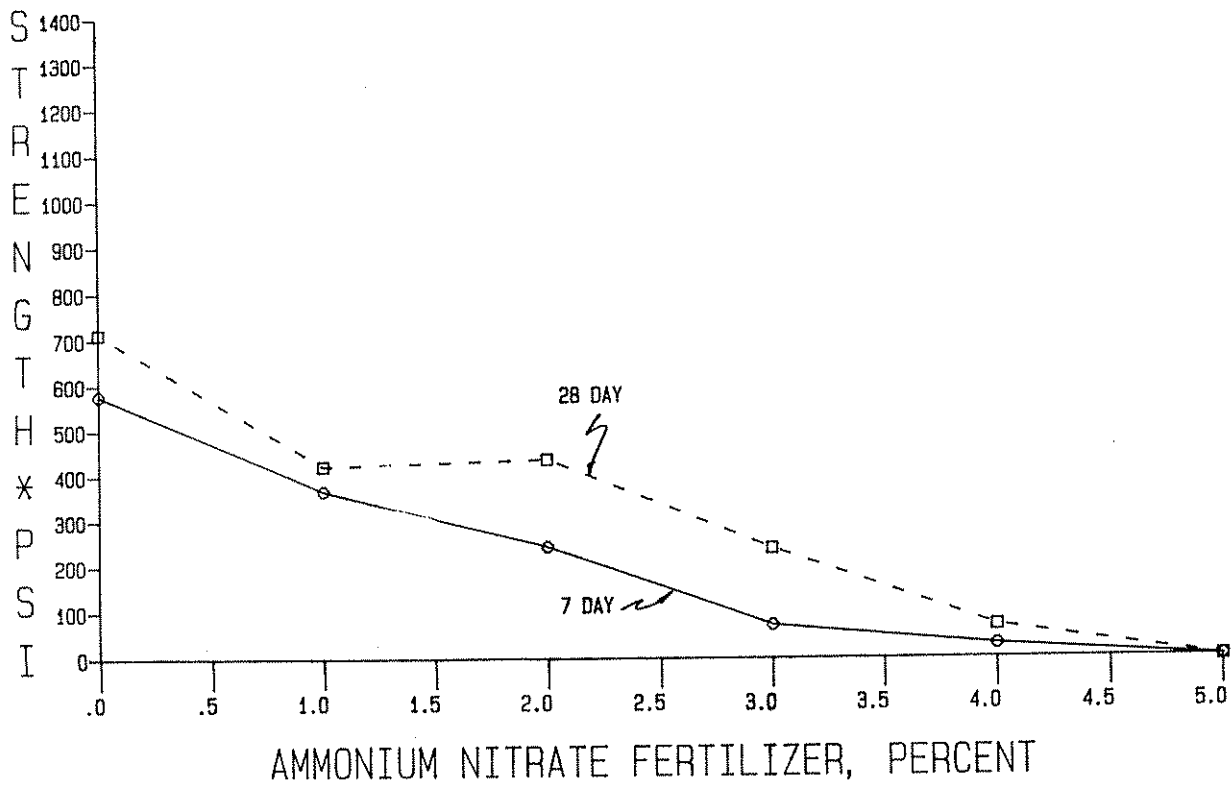
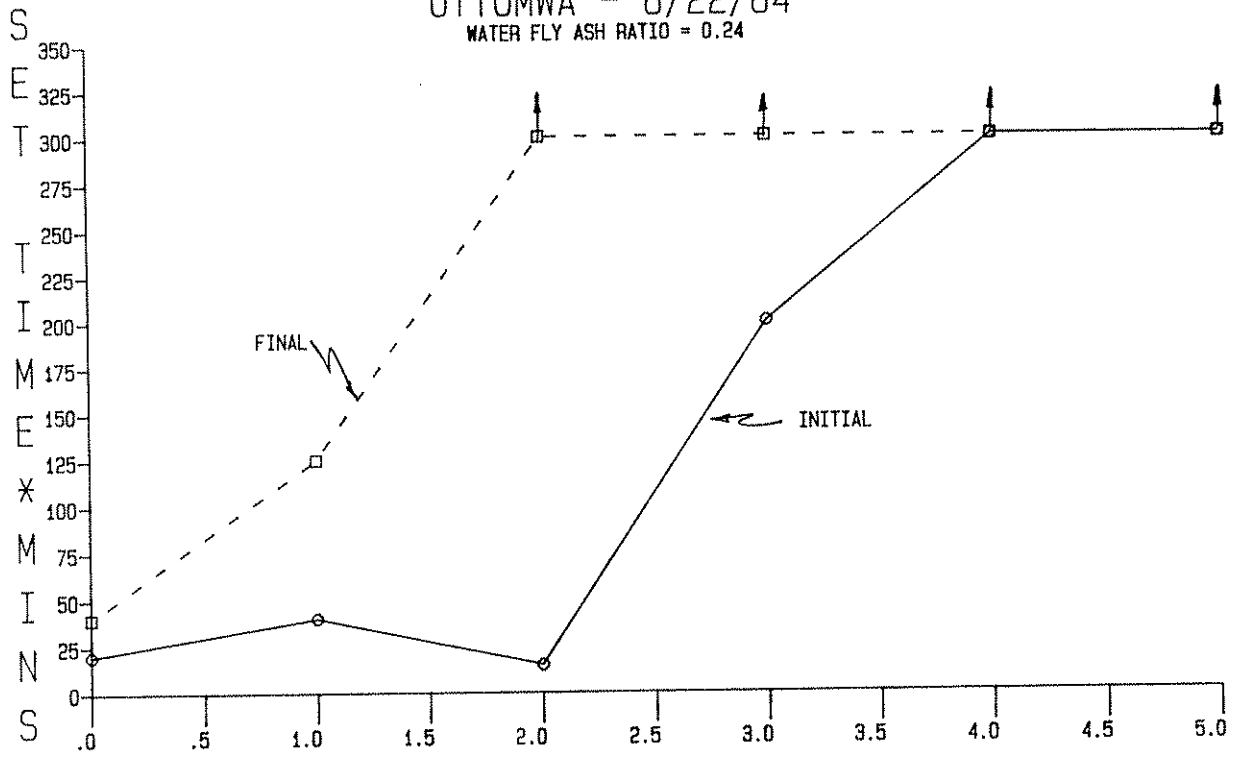
NEAL#2 - 7/08/83  
WATER FLY ASH RATIO = 0.30

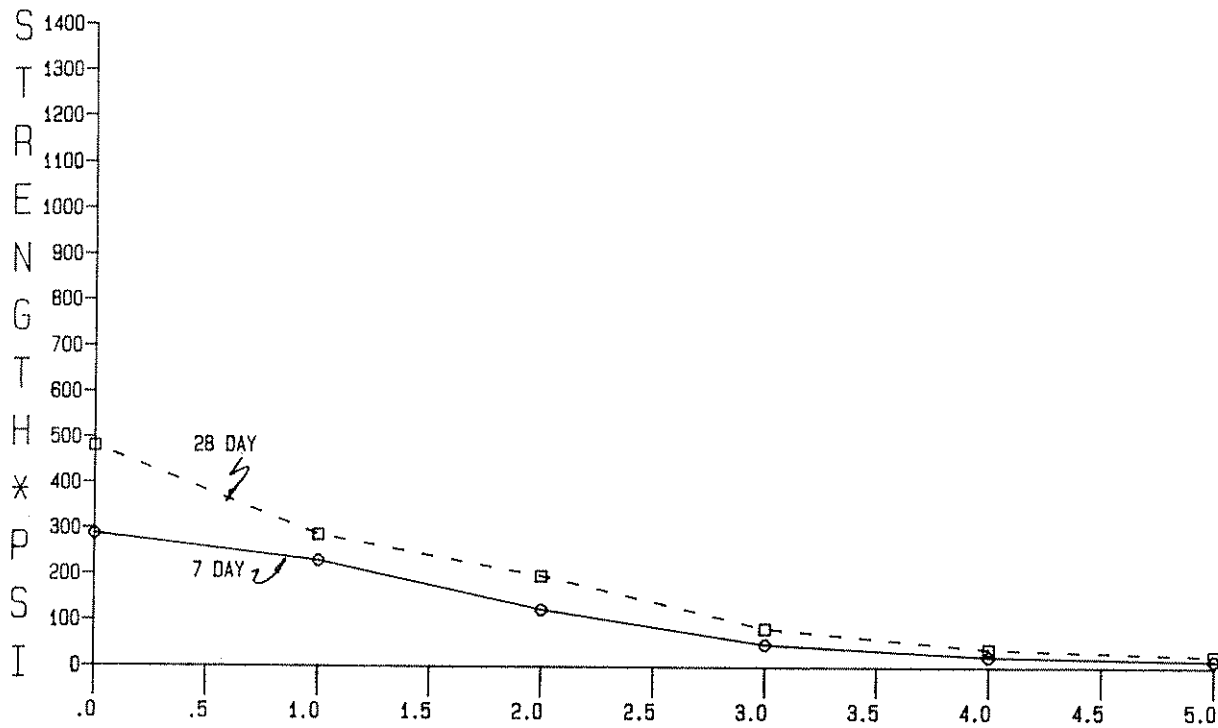
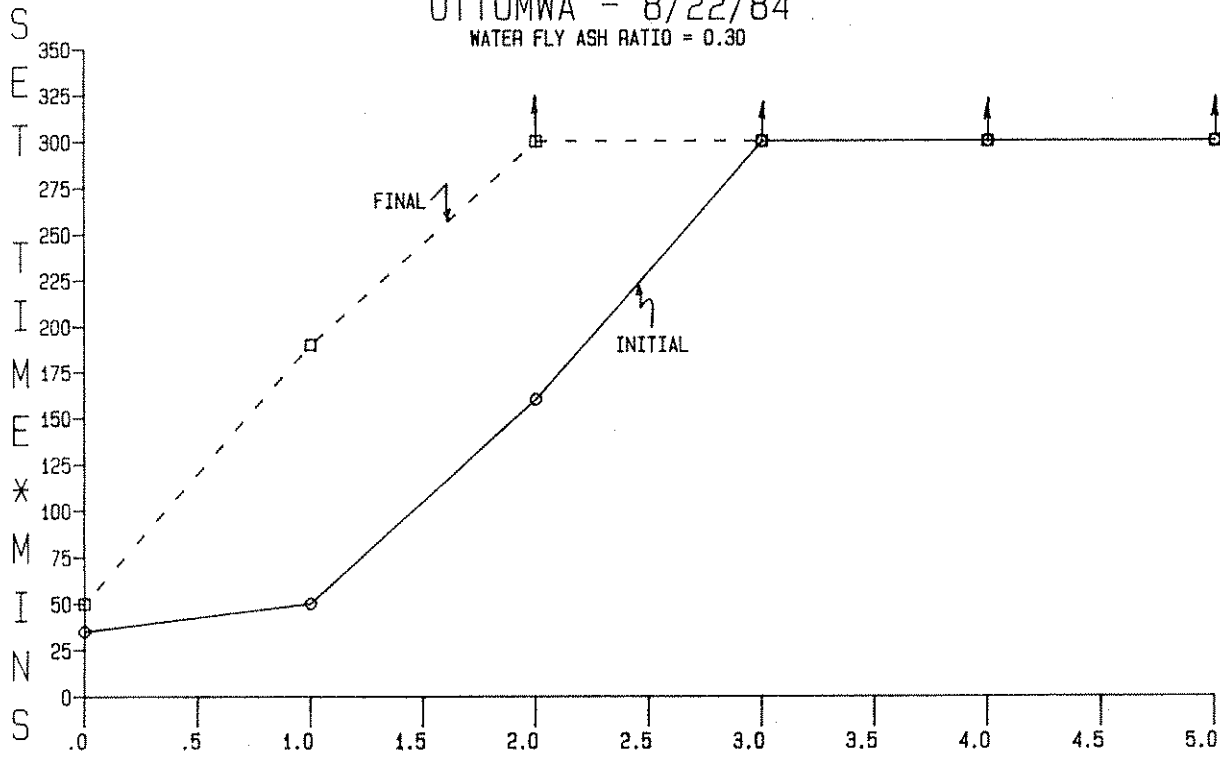


DIBASIC AMMONIUM PHOSPHATE REAGENT, PERCENT

NEAL#2 - 7/08/83  
WATER FLY ASH RATIO = 0.30

DIBASIC AMMONIUM PHOSPHATE FERTILIZER, PERCENT

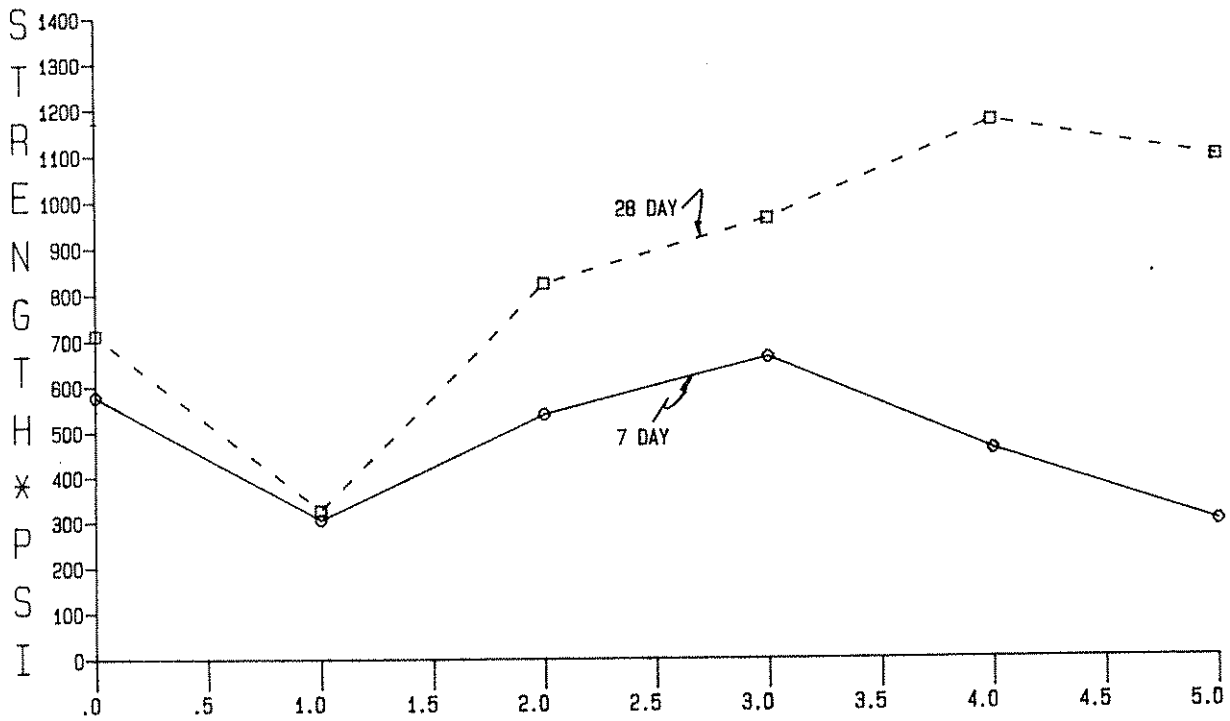
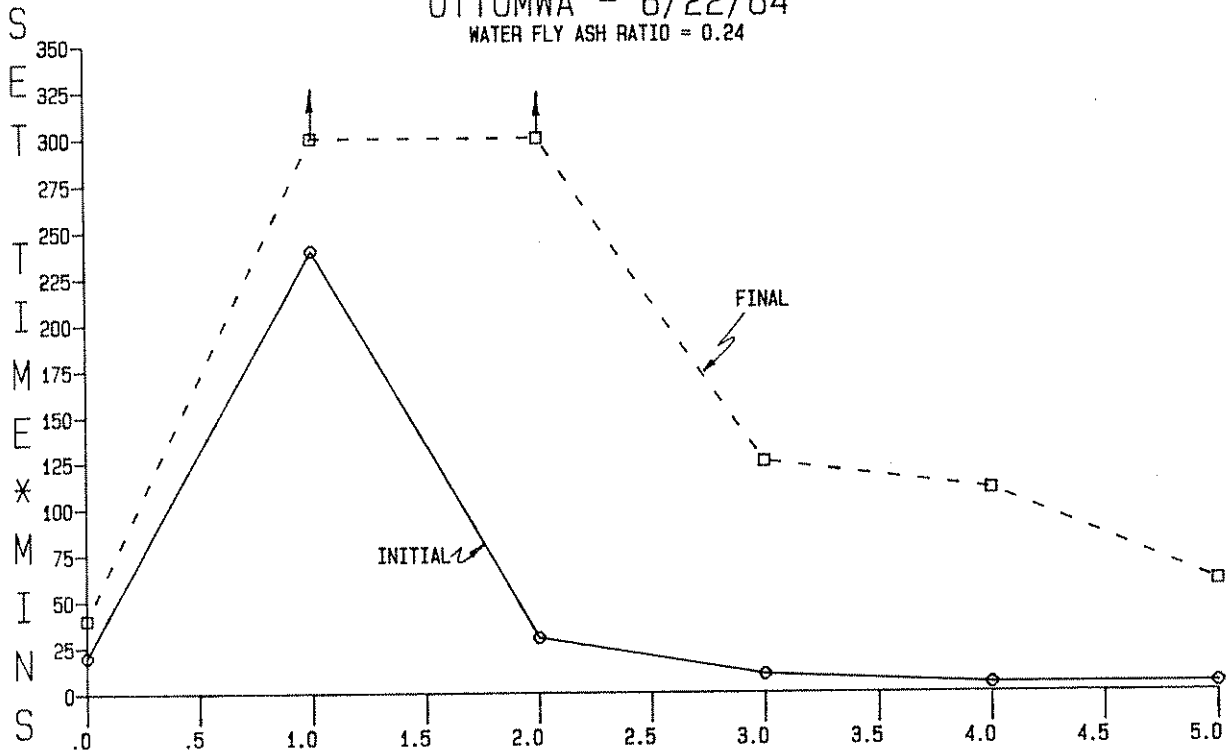
OTTUMWA - 8/22/84  
WATER FLY ASH RATIO = 0.24

OTTUMWA - 8/22/84  
WATER FLY ASH RATIO = 0.30

AMMONIUM NITRATE FERTILIZER, PERCENT

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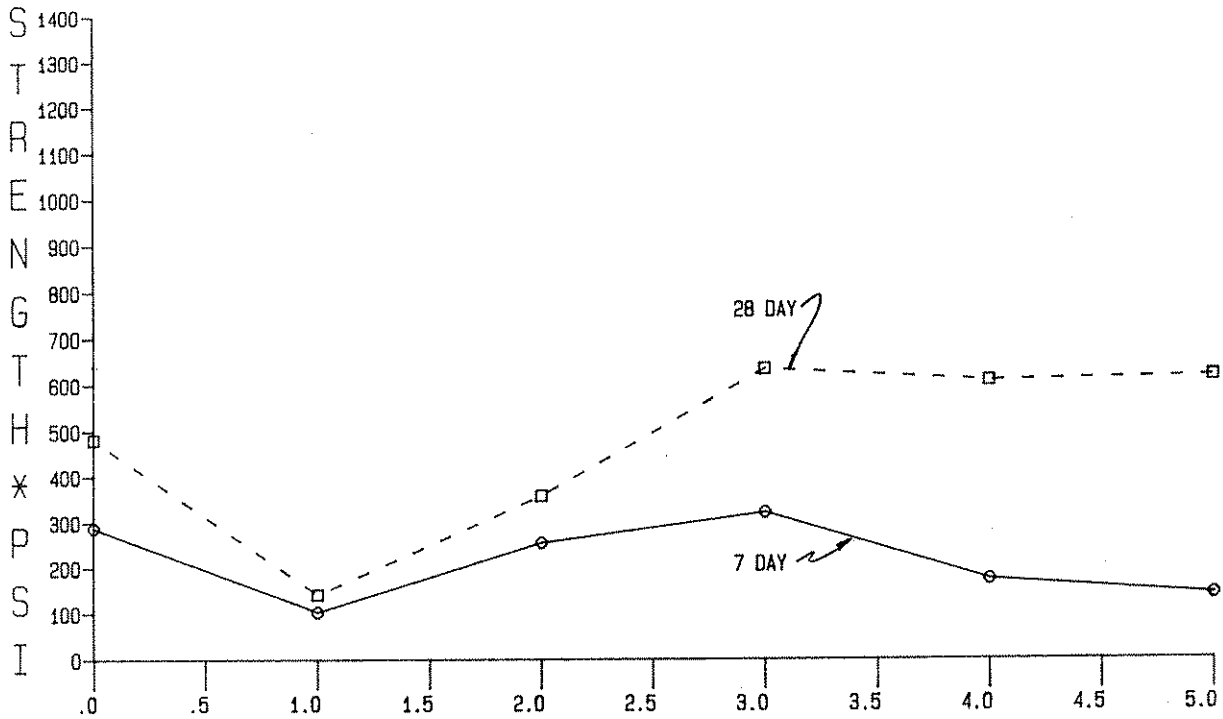
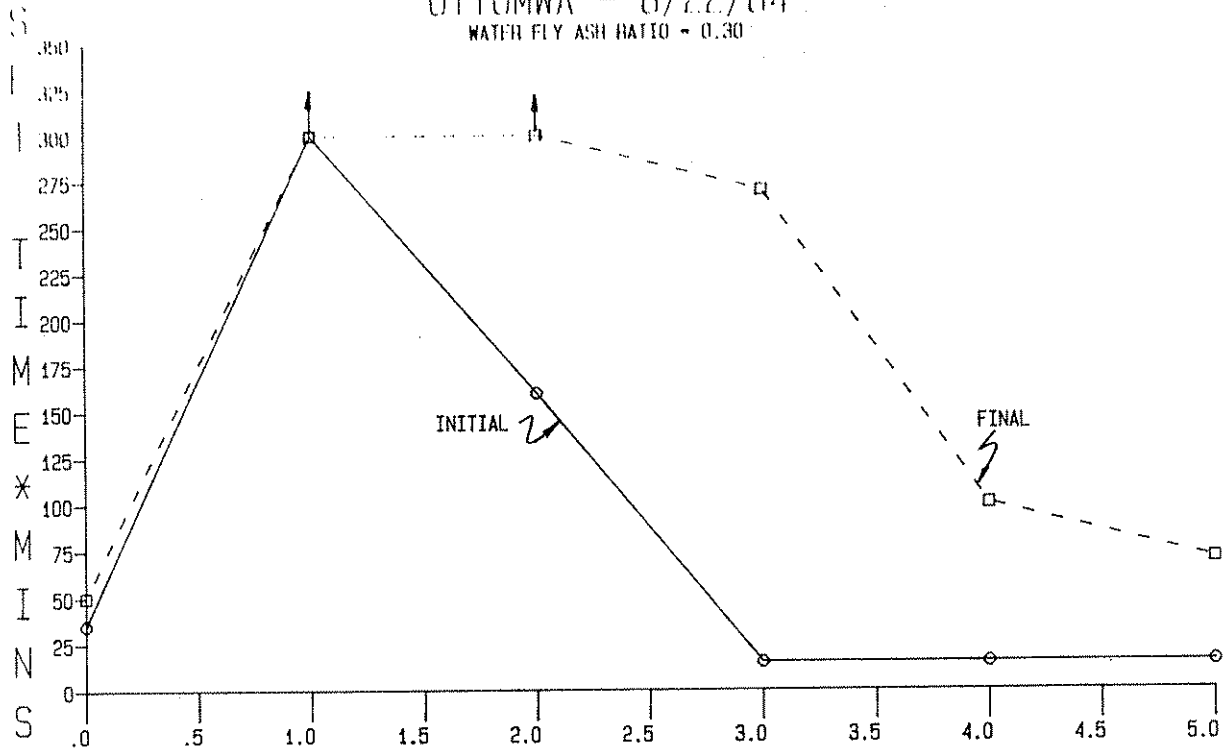
OTTUMWA - 8/22/84  
WATER FLY ASH RATIO = 0.24



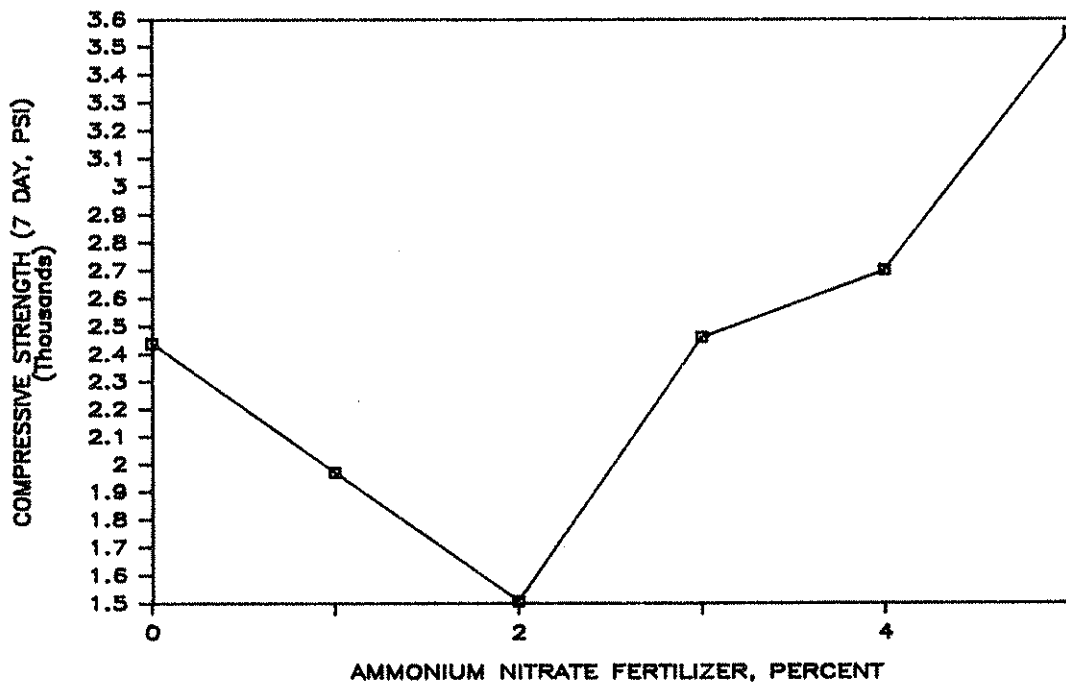
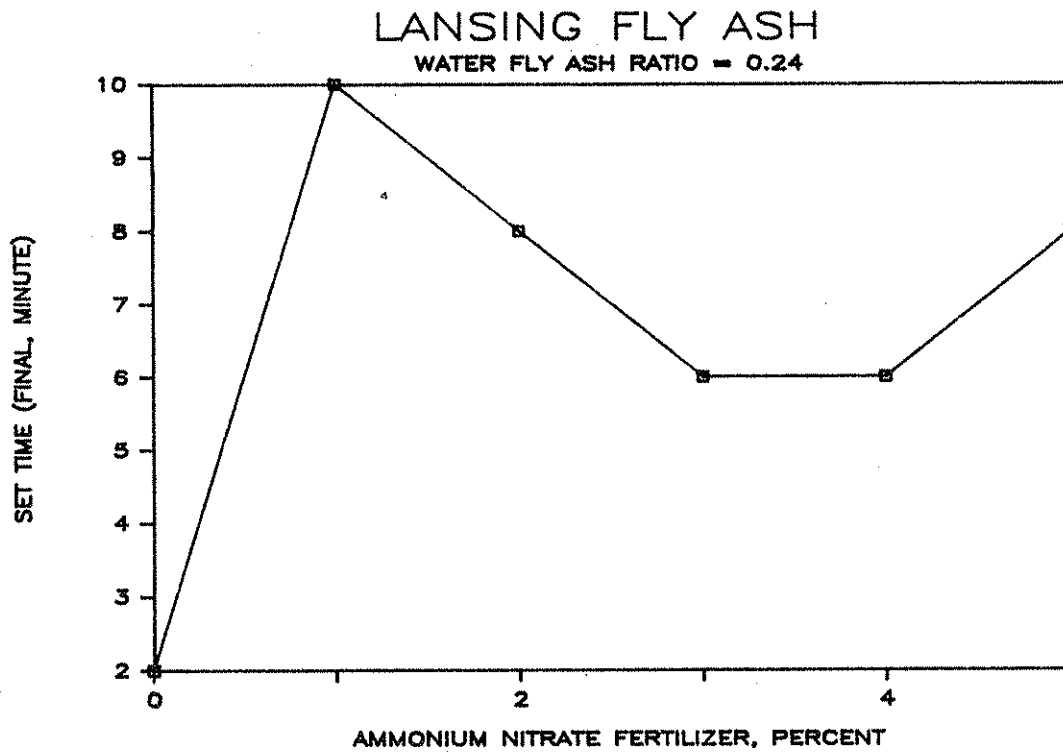
DIBASIC AMMONIUM PHOSPHATE FERTILIZER, PERCENT

OTTUMWA - 8/22/84

WATER FLY ASH RATIO = 0.30



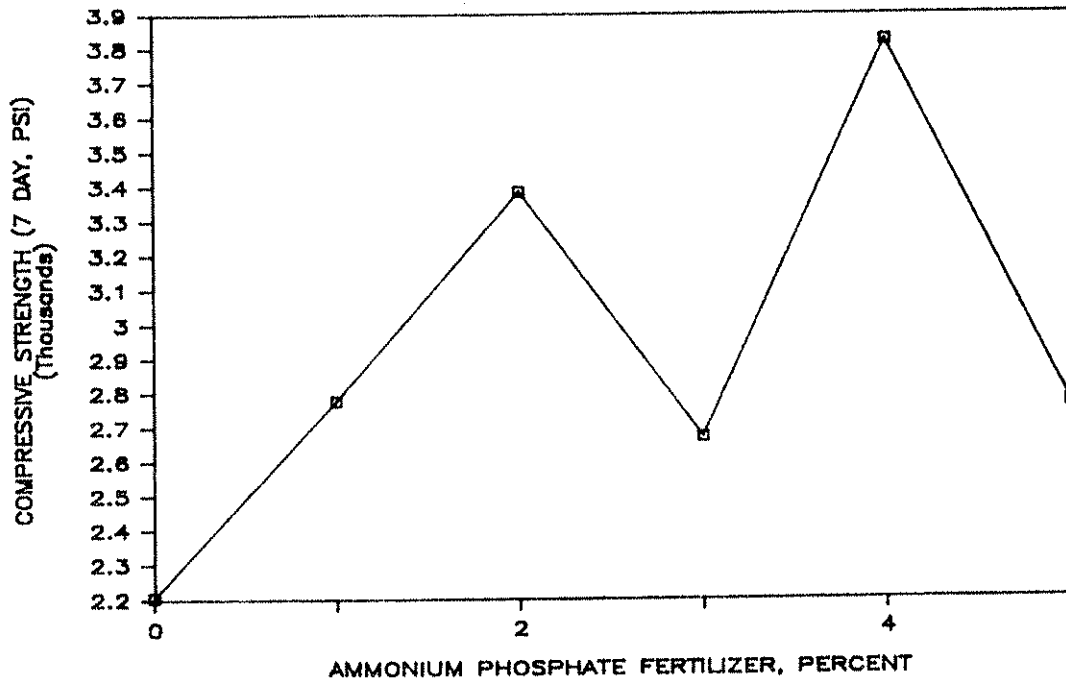
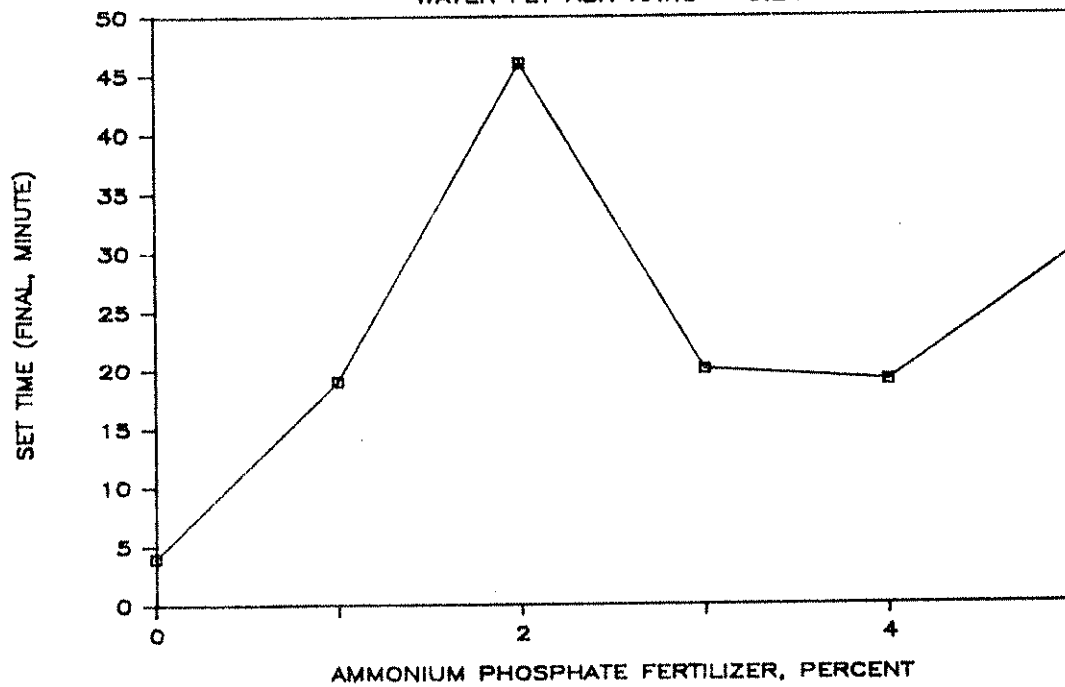
DIBASIC AMMONIUM PHOSPHATE FERTILIZER, PERCENT





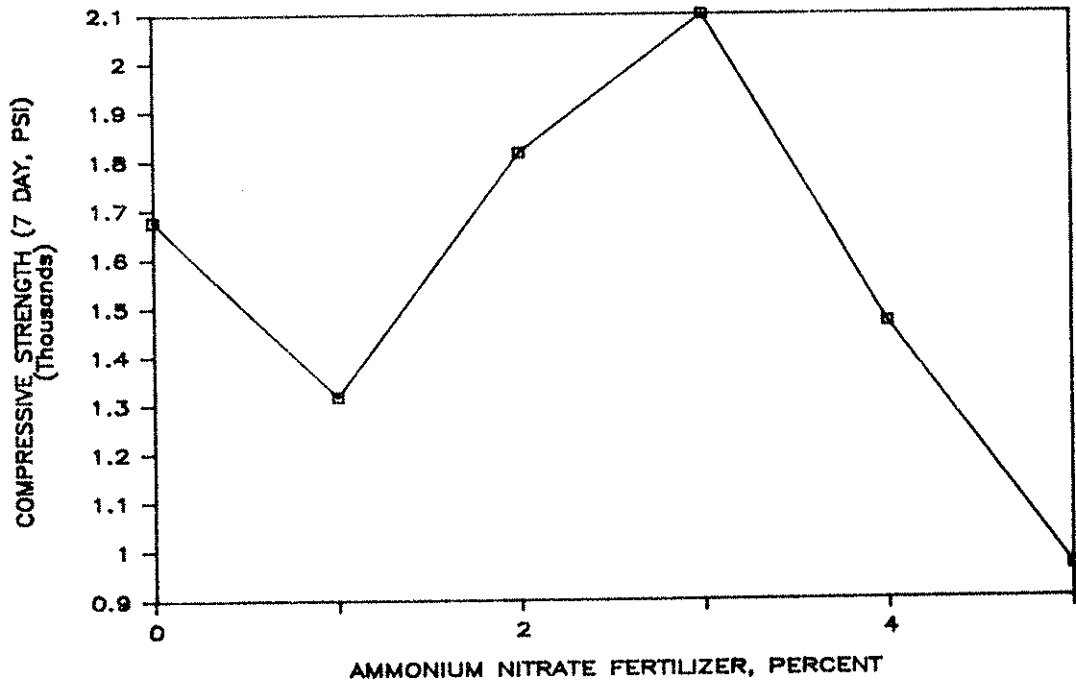
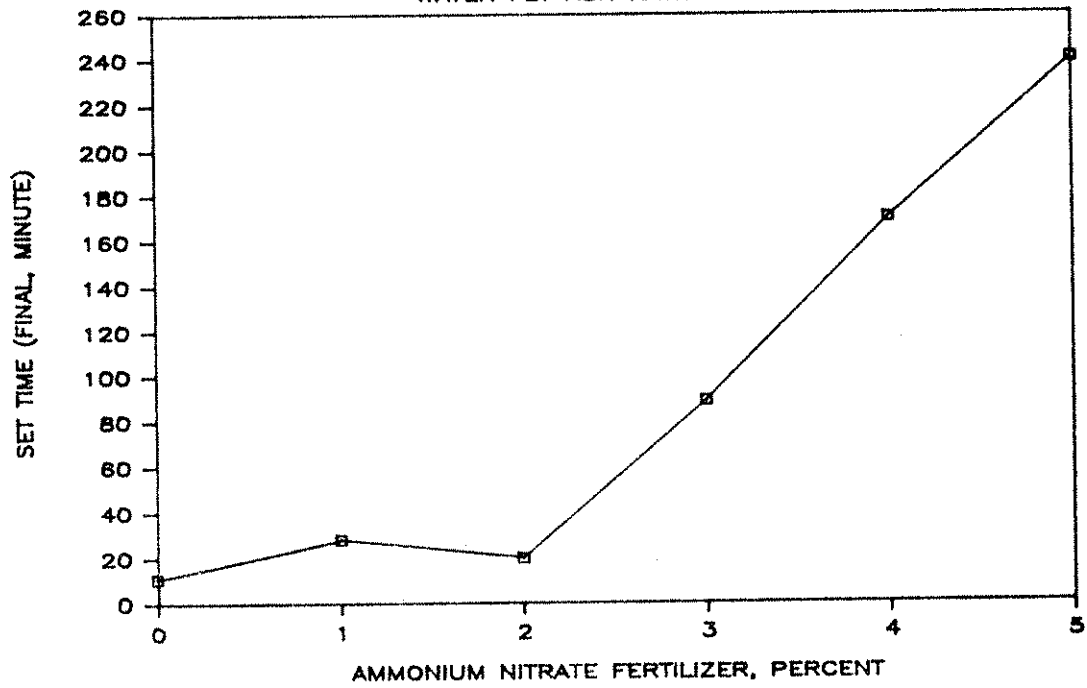
## LANSING FLY ASH

WATER FLY ASH RATIO = 0.24



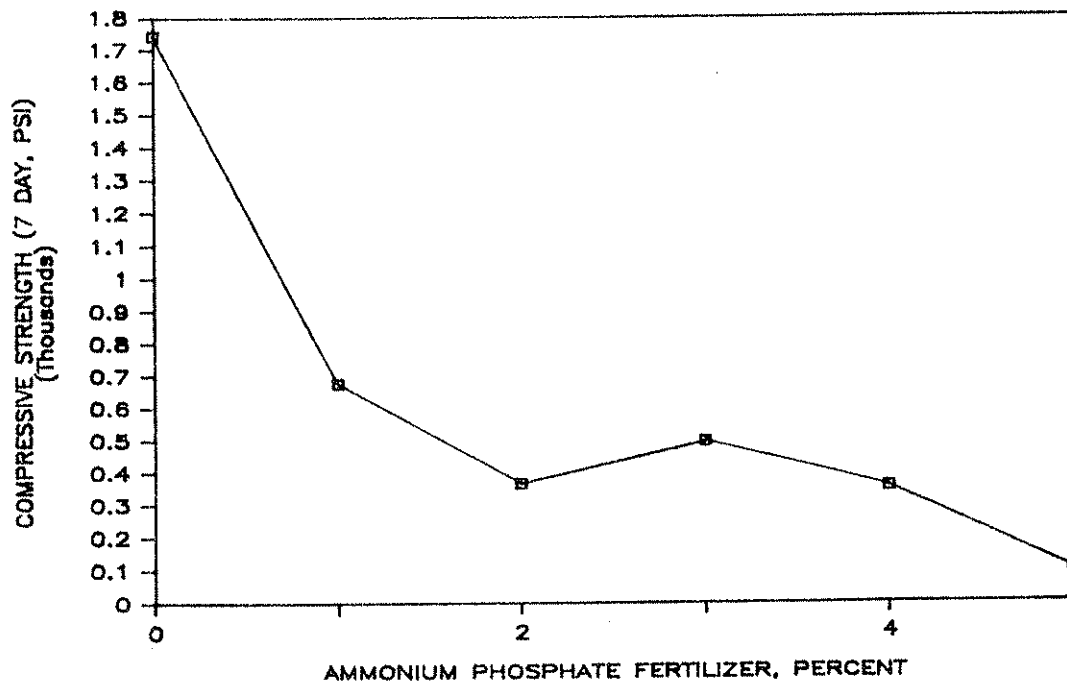
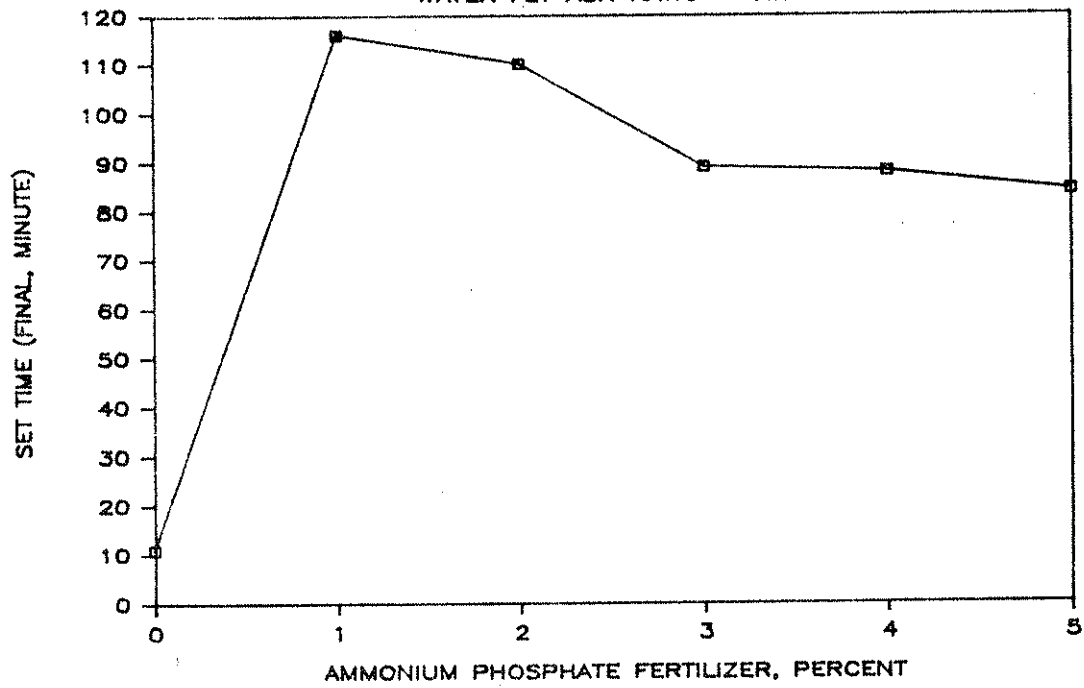
## LOUISA FLY ASH

WATER FLY ASH RATIO = 0.20



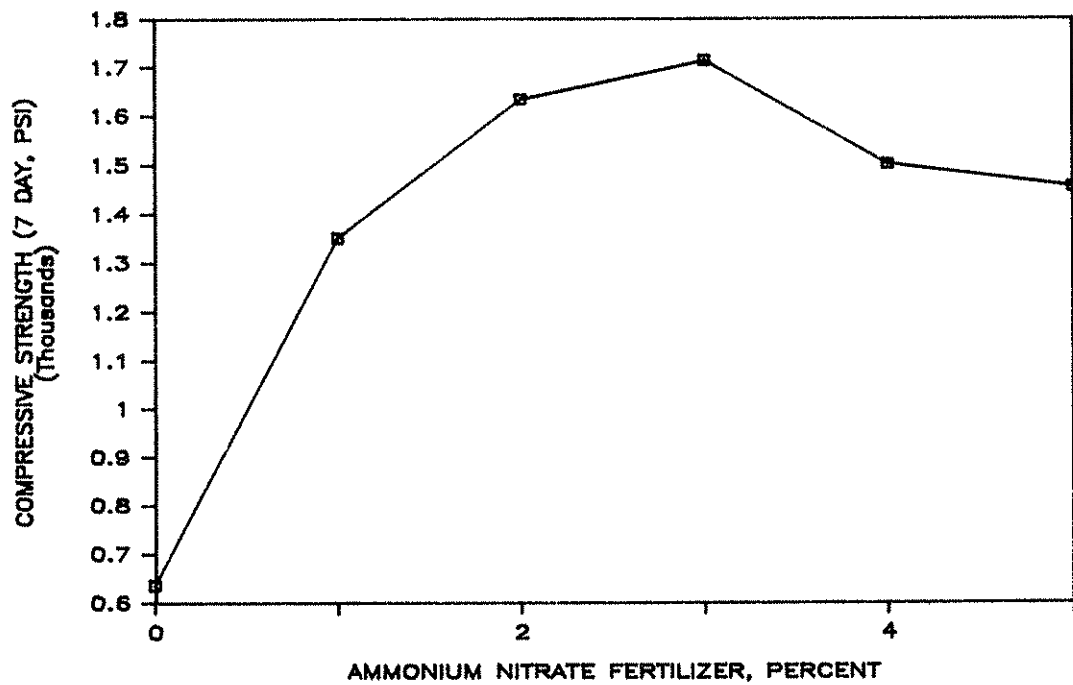
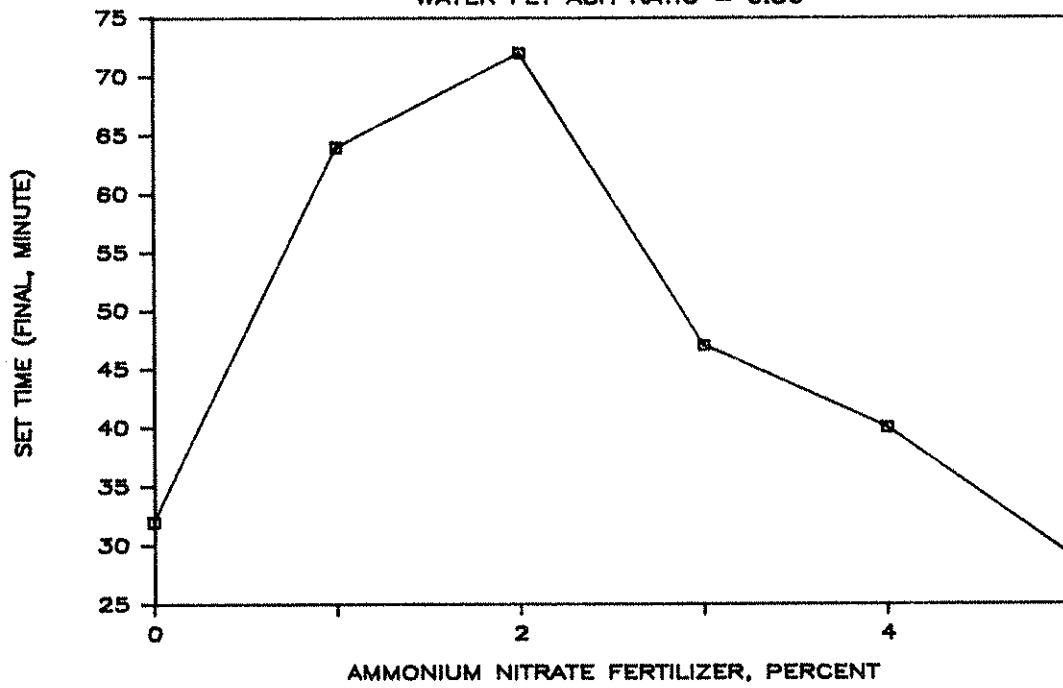
# LOUISA FLY ASH

WATER FLY ASH RATIO = 0.20



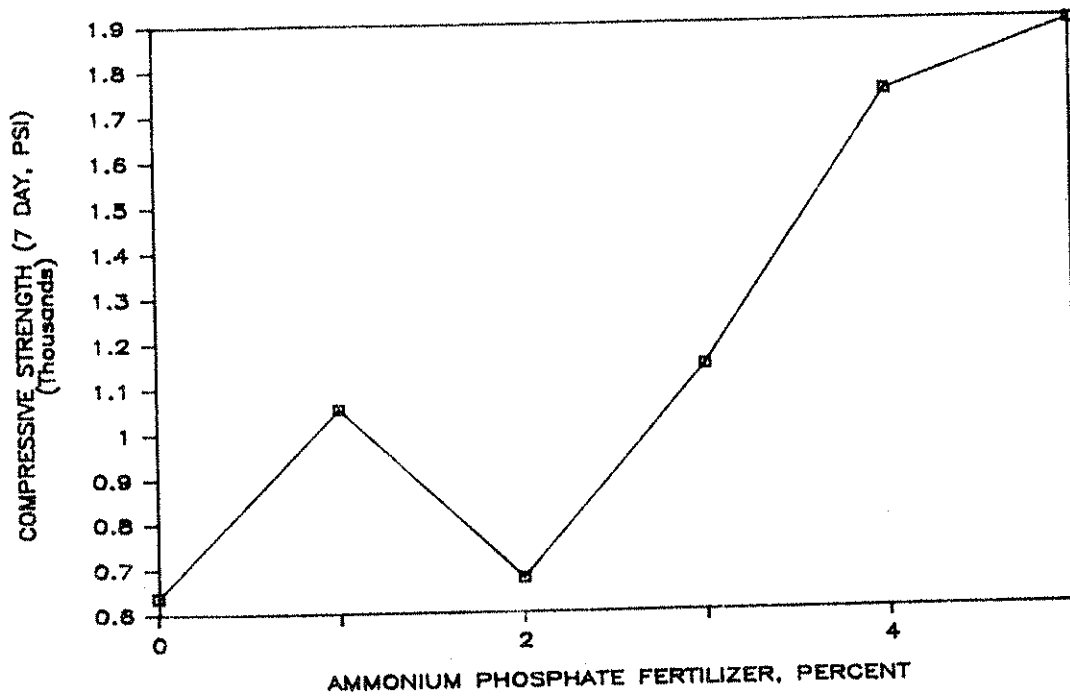
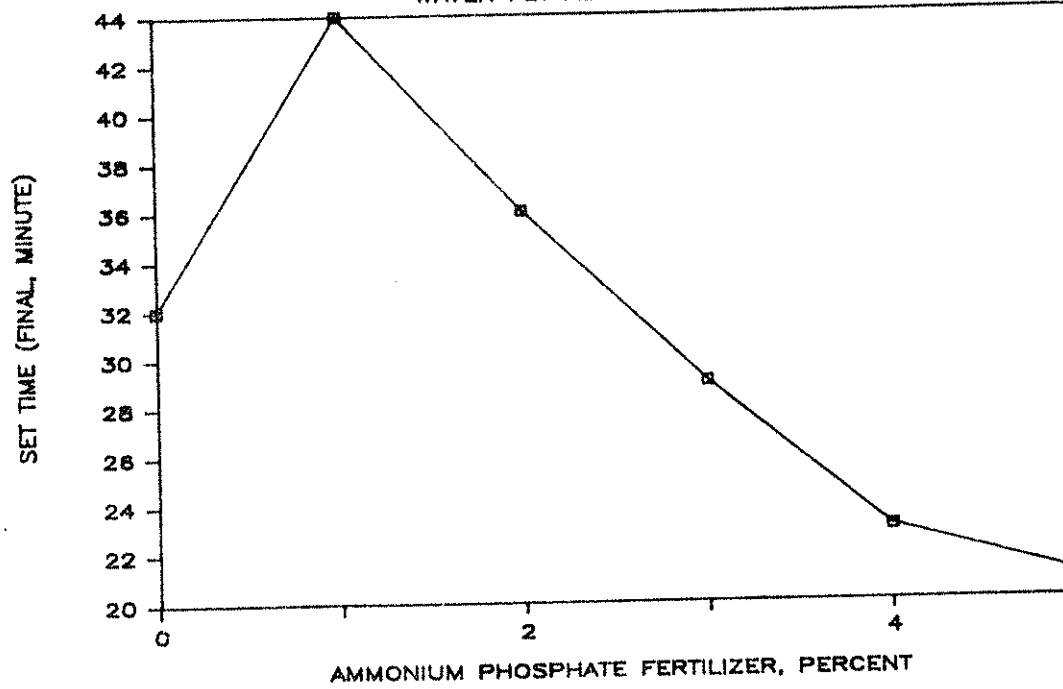
# AMES FLY ASH

WATER FLY ASH RATIO = 0.30



# AMES FLY ASH

WATER FLY ASH RATIO = 0.30



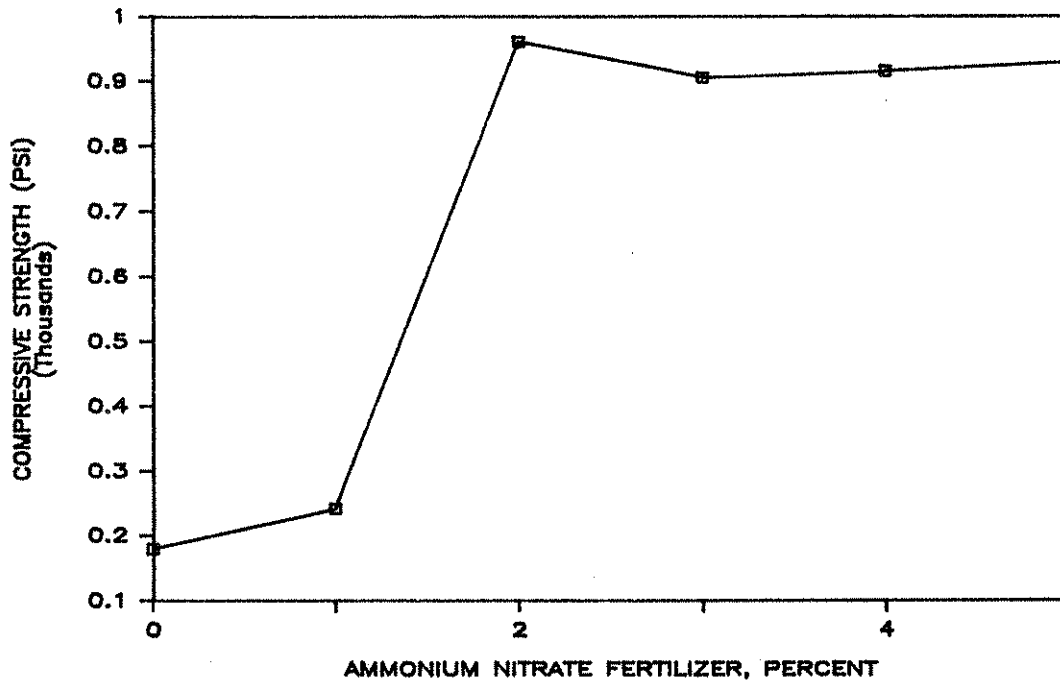
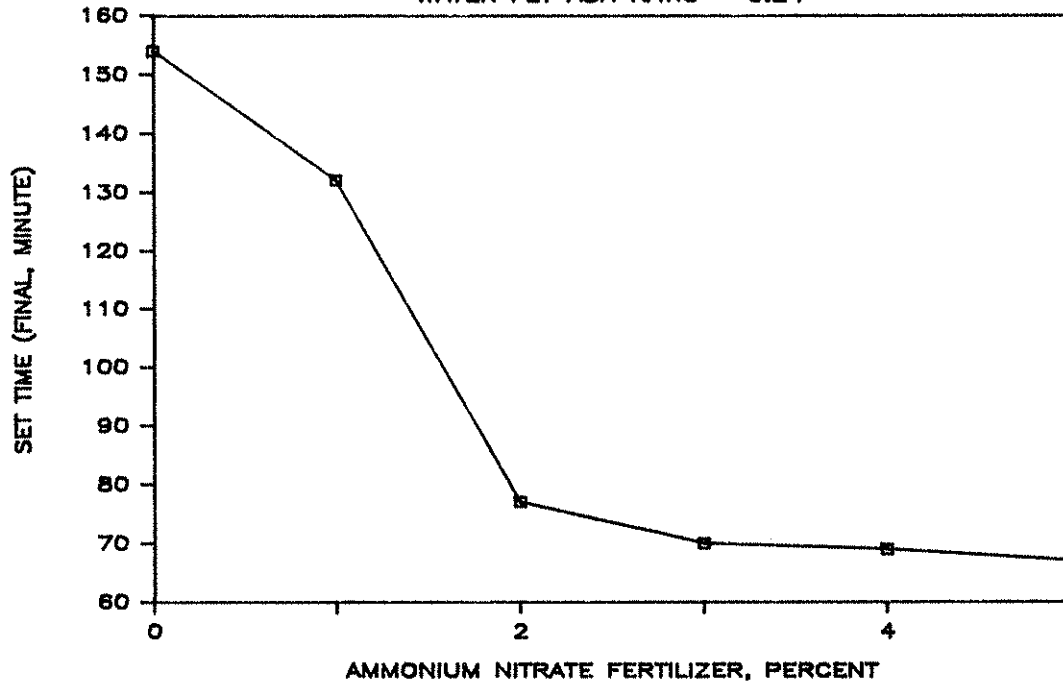
## APPENDIX C

### ADDITIVE CONCENTRATION

C-1

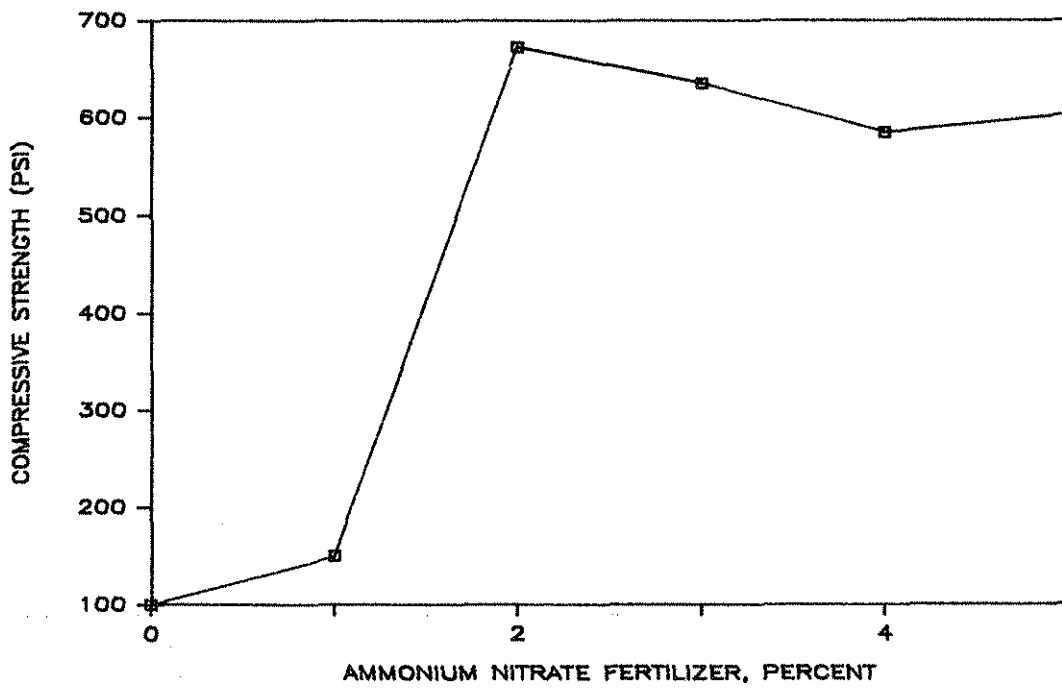
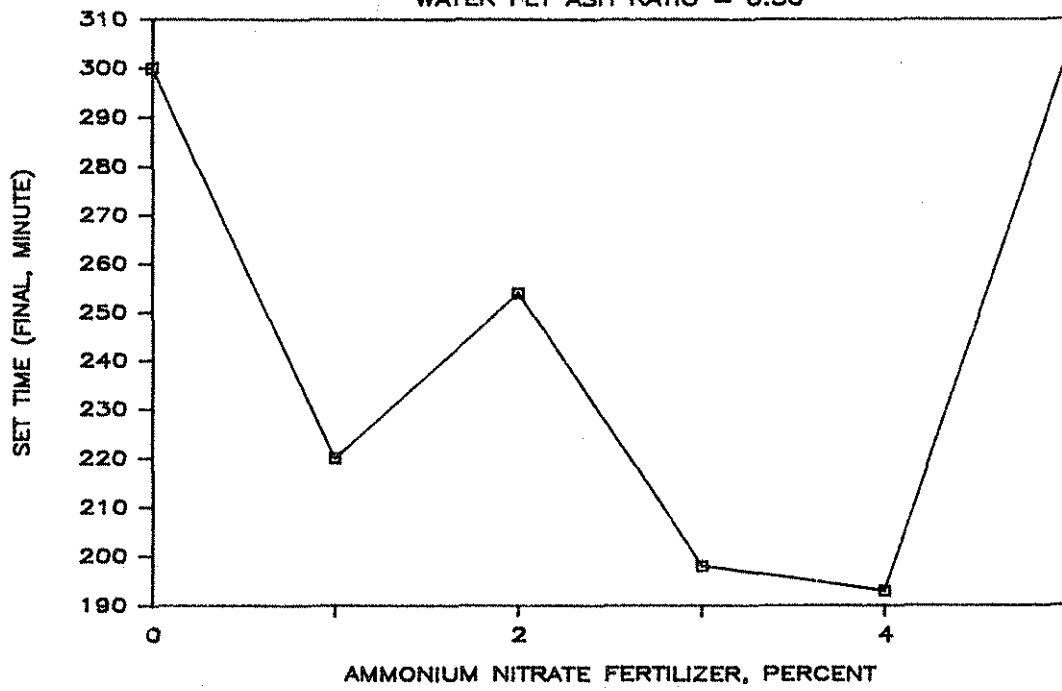
## NEAL #2

WATER FLY ASH RATIO = 0.24



## NEAL #2

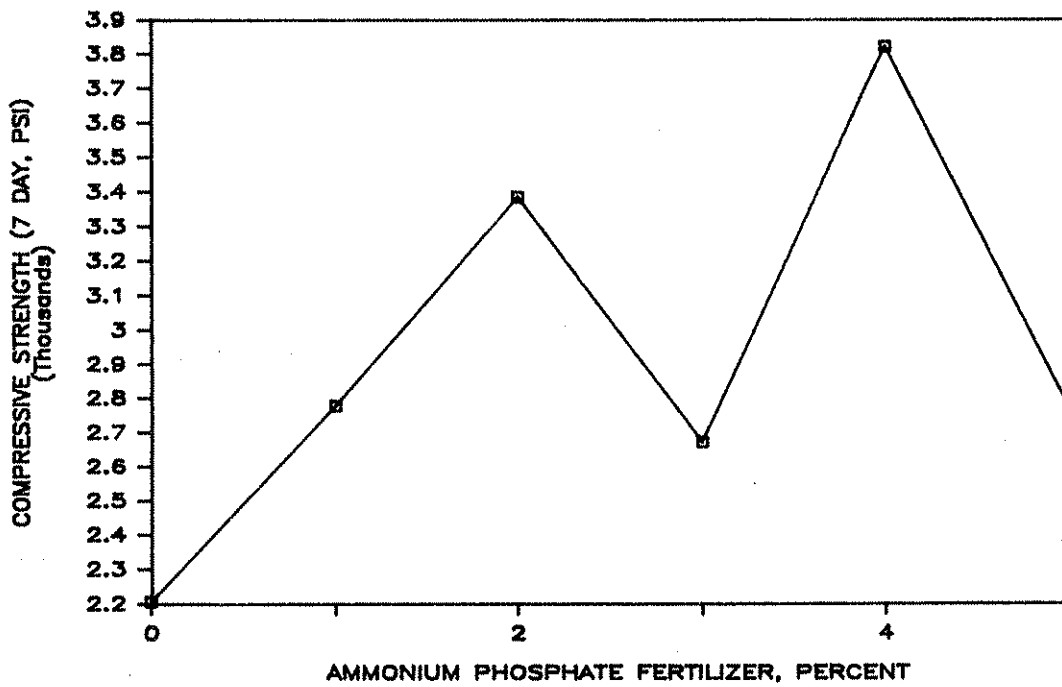
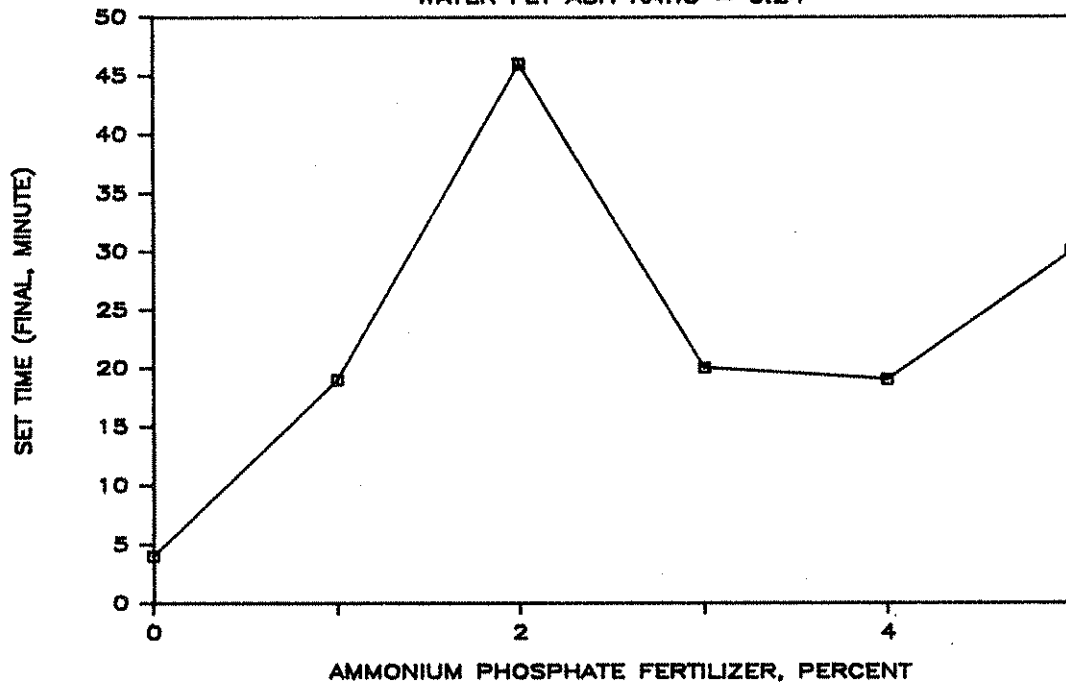
WATER FLY ASH RATIO = 0.30





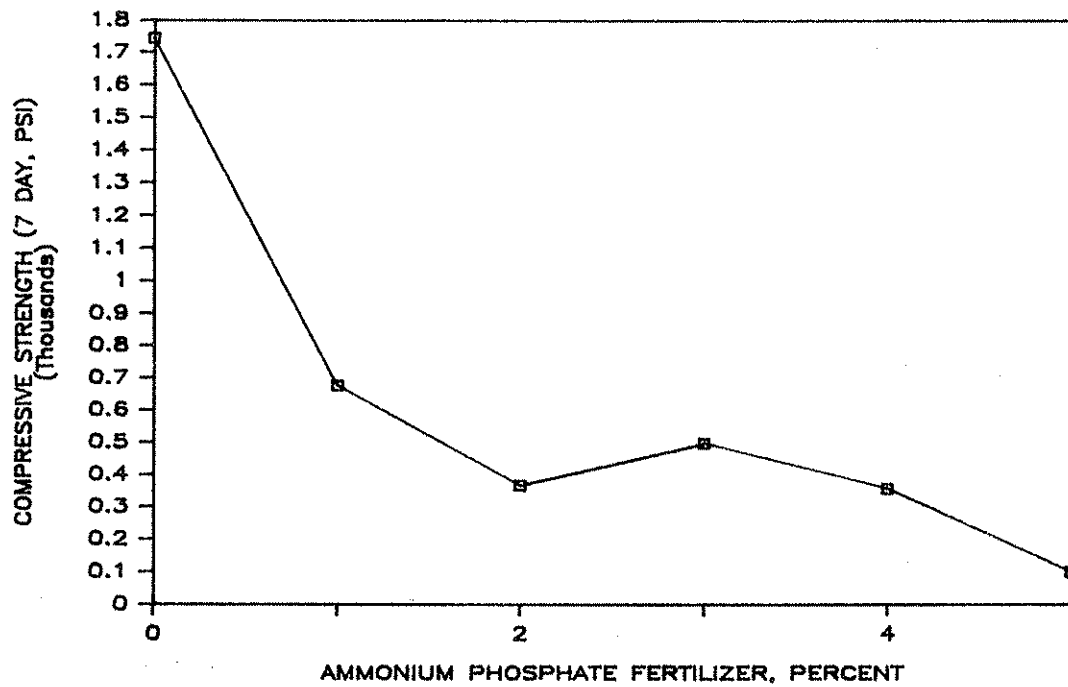
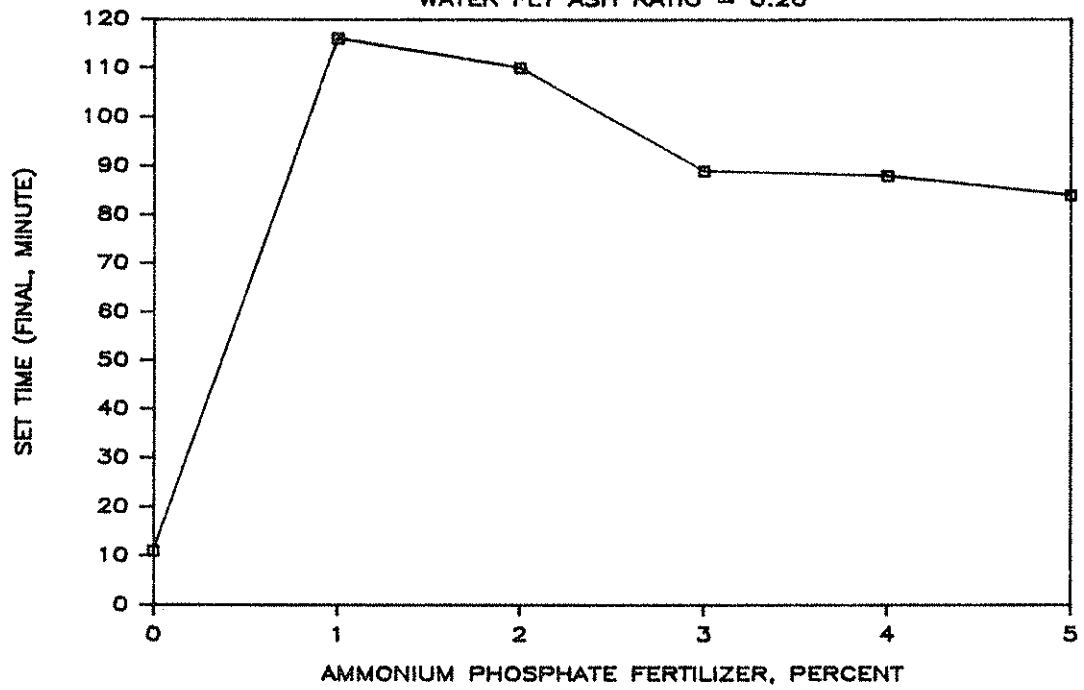
## LANSING FLY ASH

WATER FLY ASH RATIO = 0.24



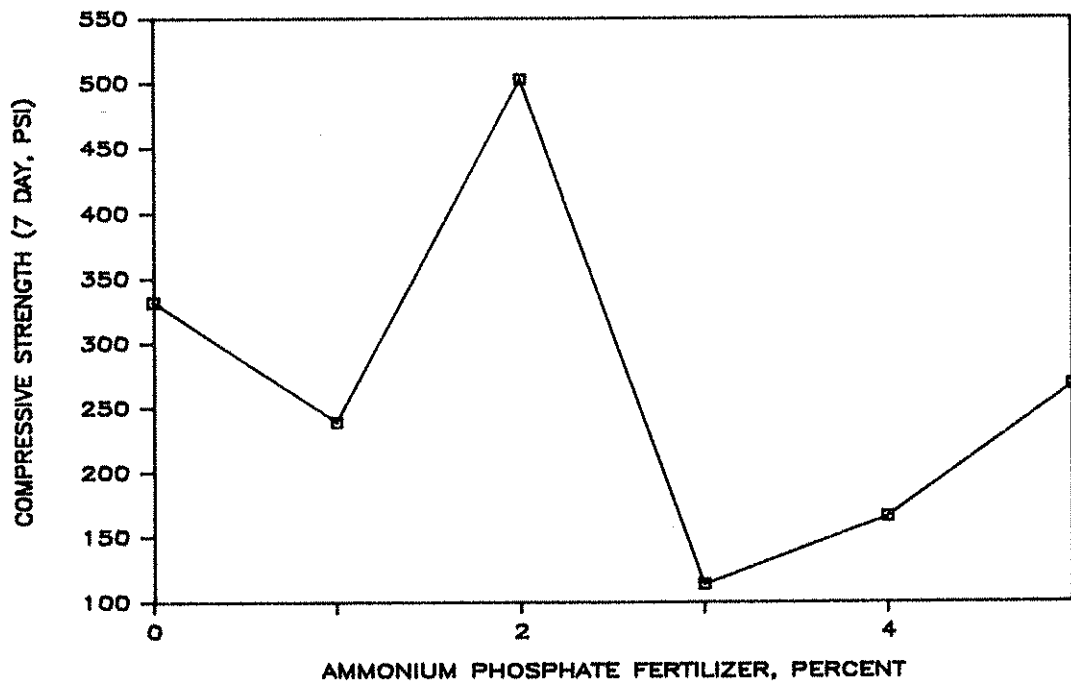
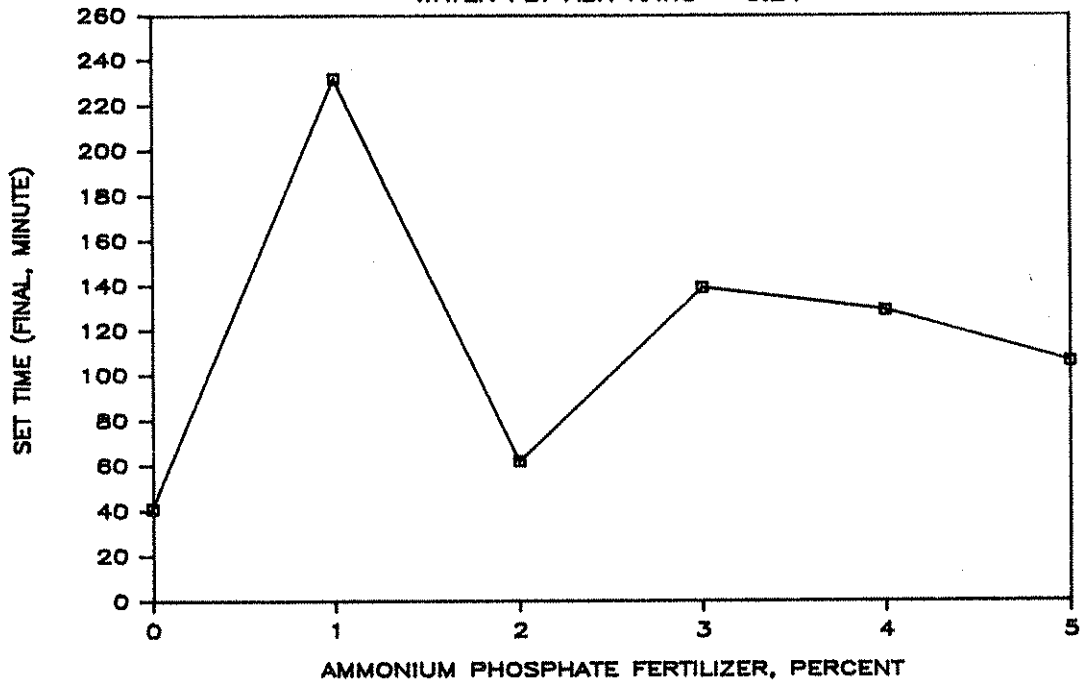
## LOUISA FLY ASH

WATER FLY ASH RATIO = 0.20



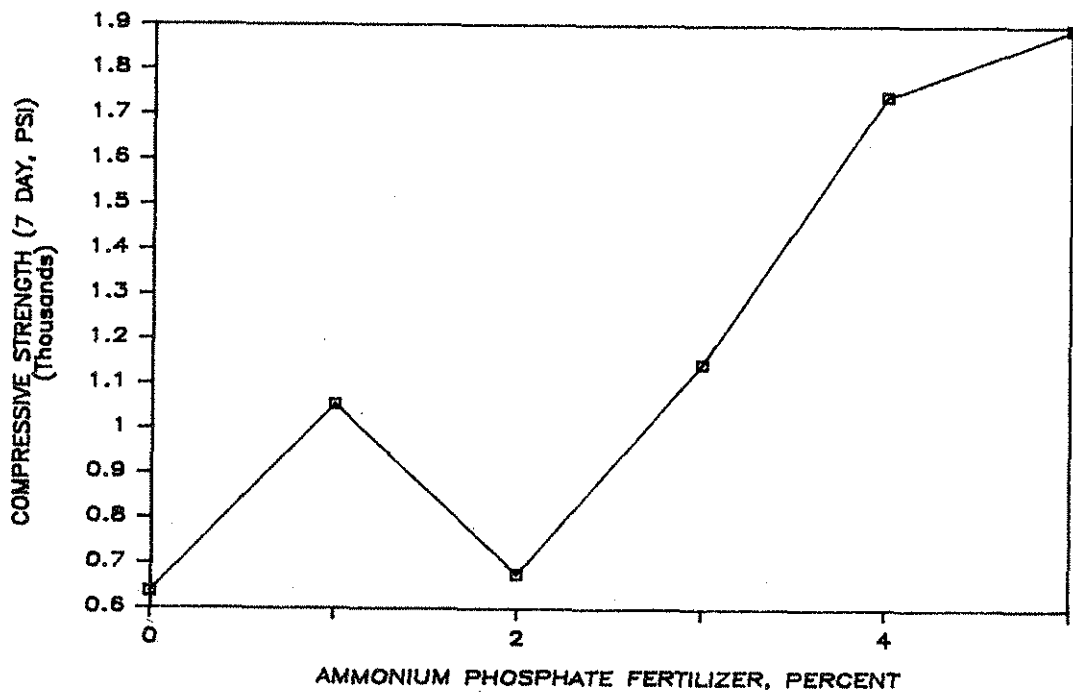
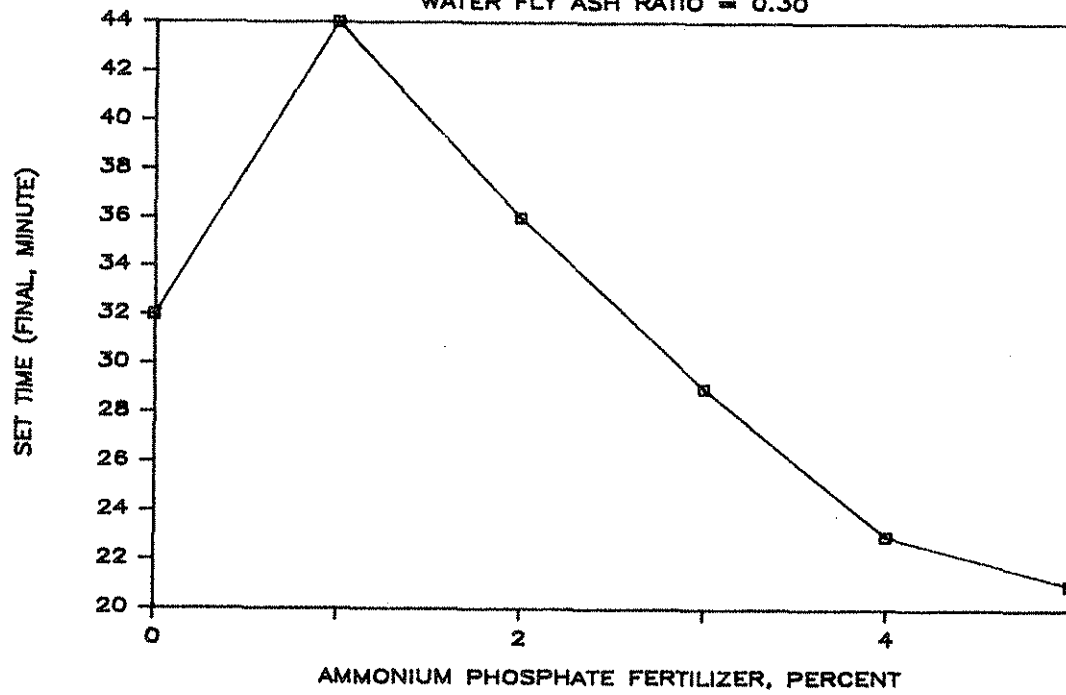
## OTTUMWA FLY ASH

WATER FLY ASH RATIO = 0.24



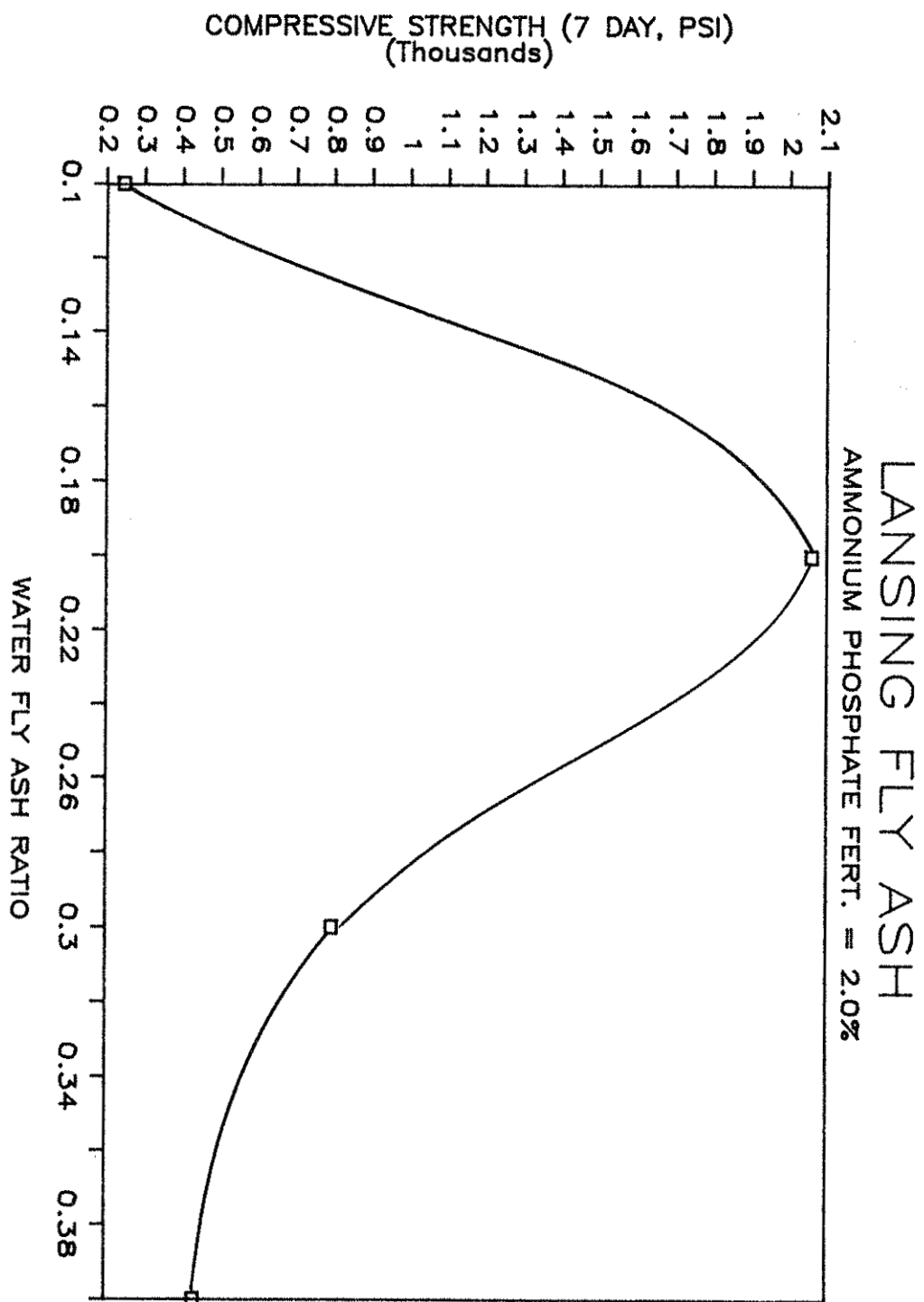
C-6

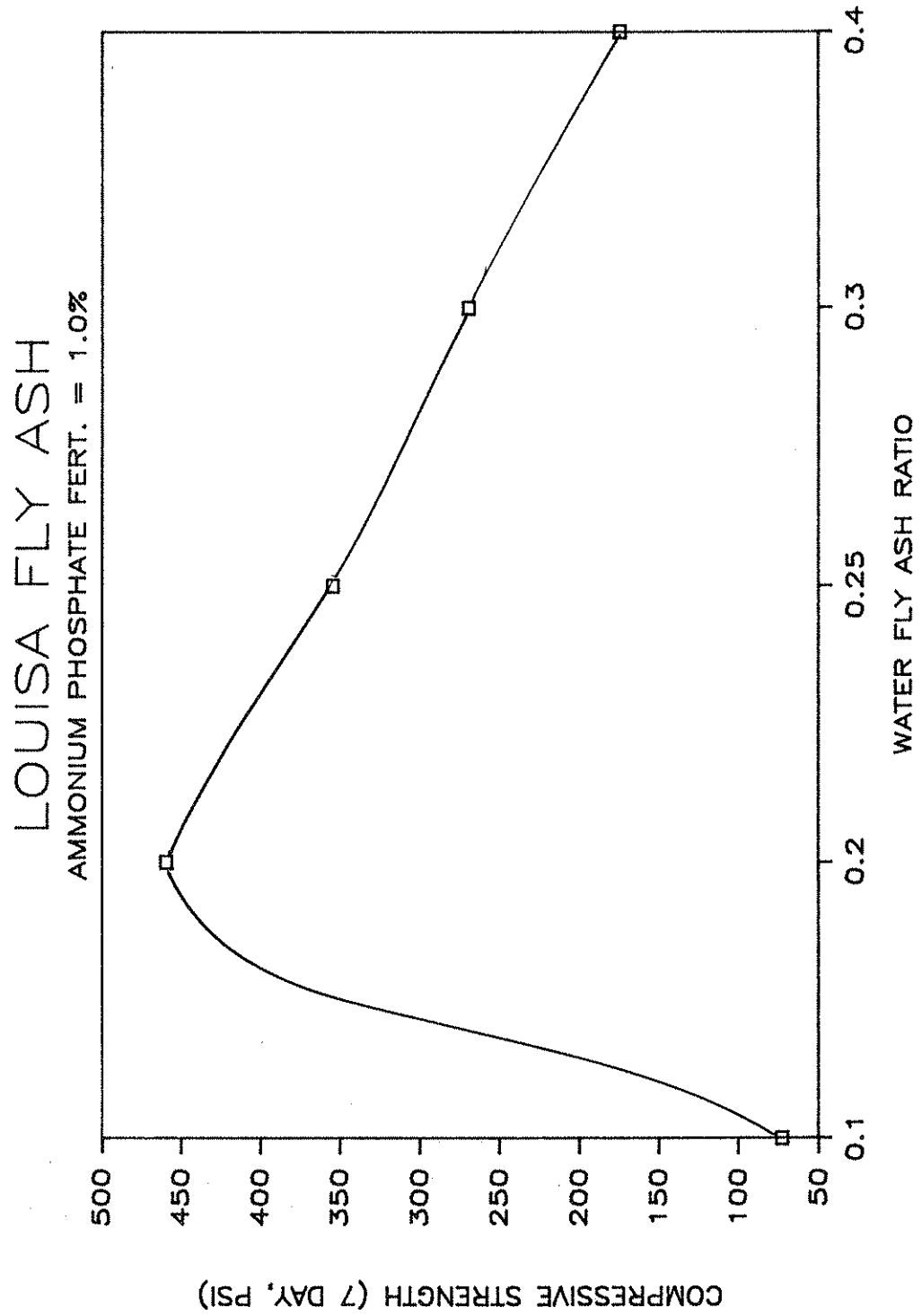
# AMES FLY ASH WATER FLY ASH RATIO = 0.30

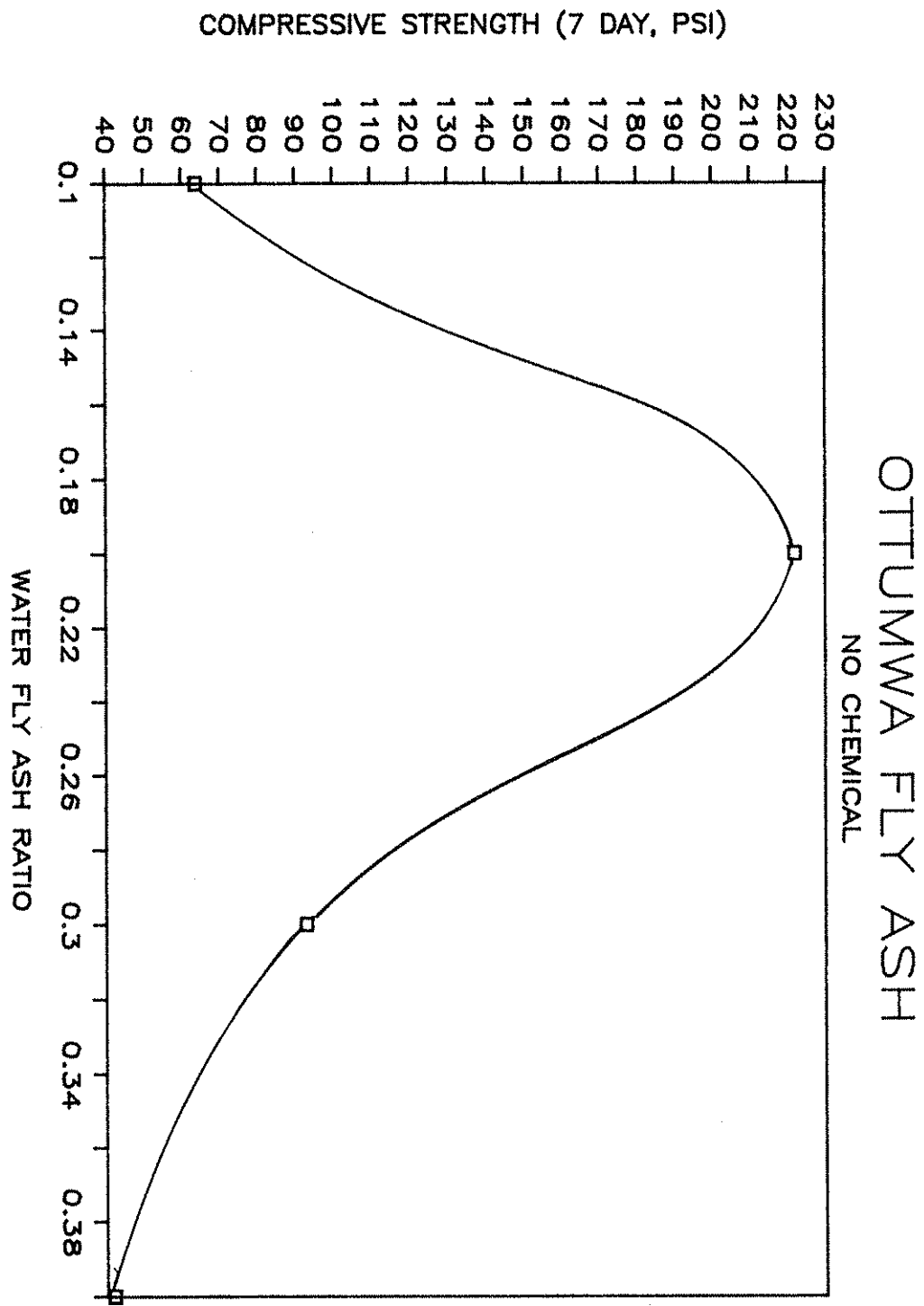


## APPENDIX D

### EFFECT OF WATER/CEMENT RATIO ON STRENGTH

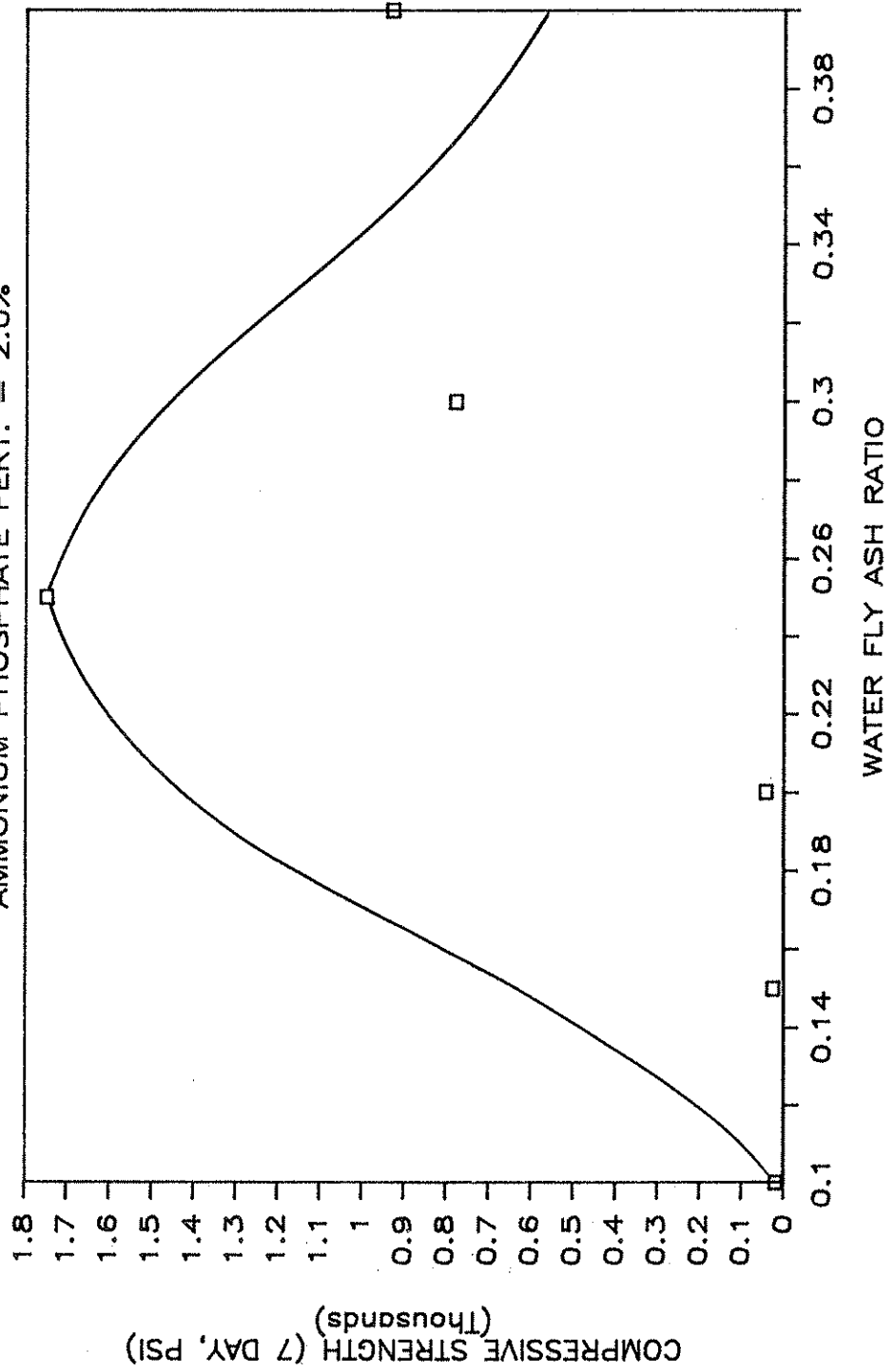








AMES FLY ASH  
AMMONIUM PHOSPHATE FERT. = 2.0%

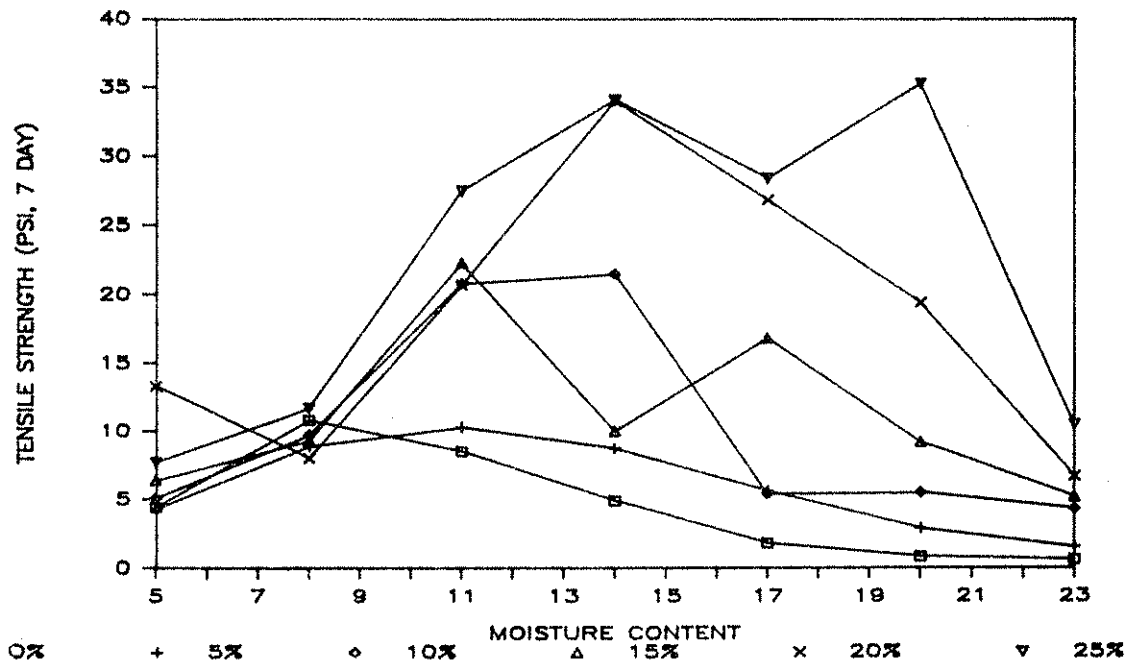
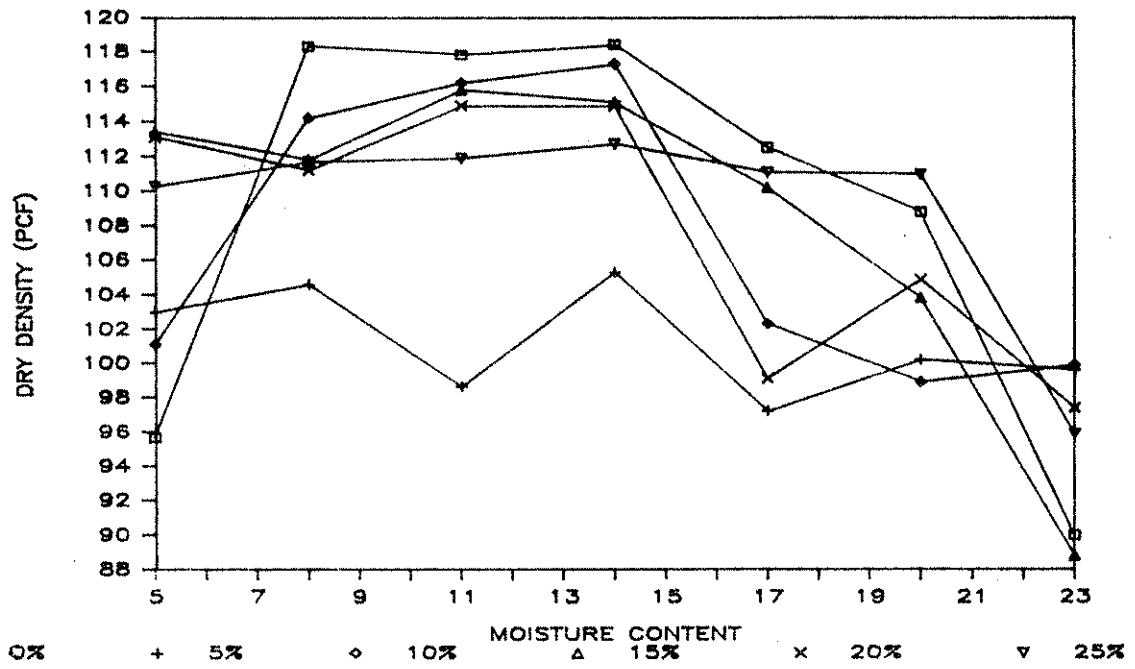


## APPENDIX E

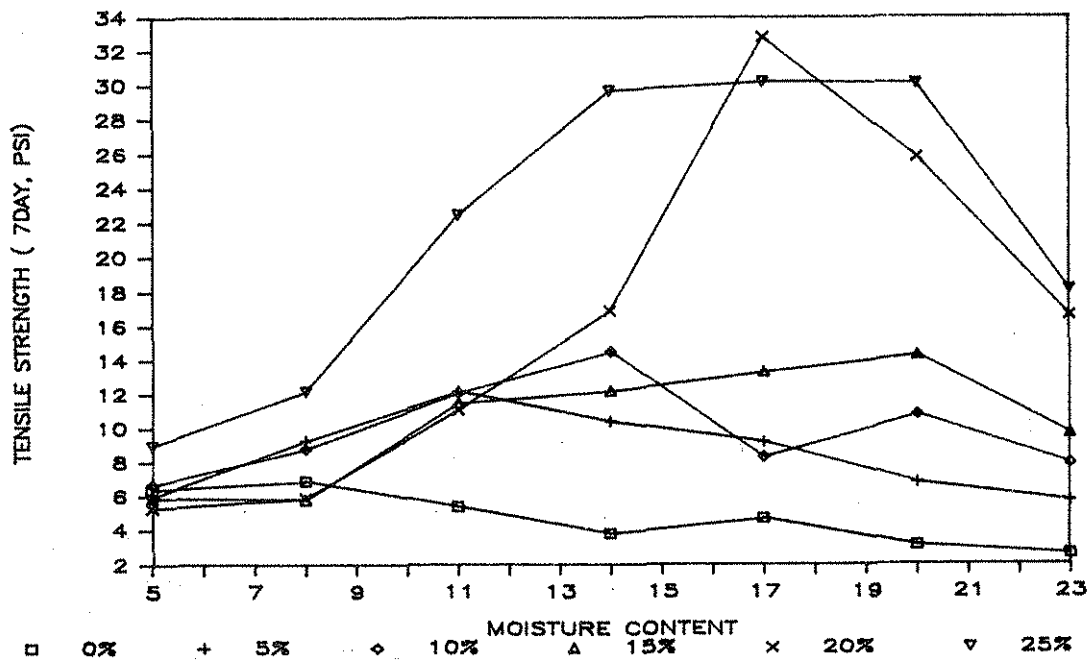
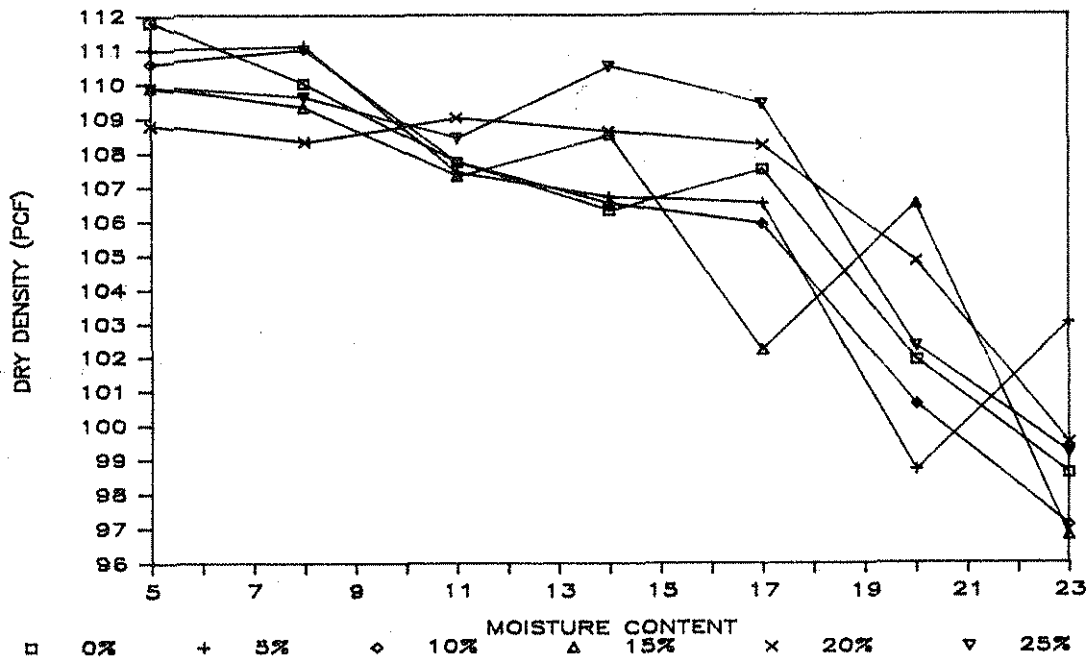
### THE NINE SOIL BLENDS

E-1

Blend #1

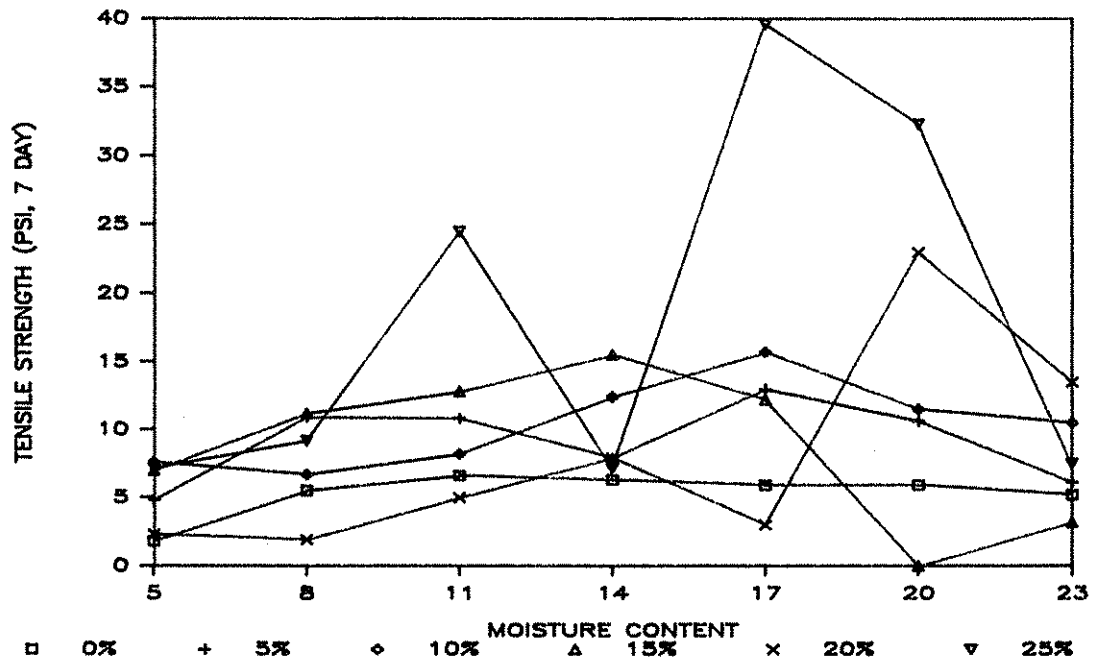
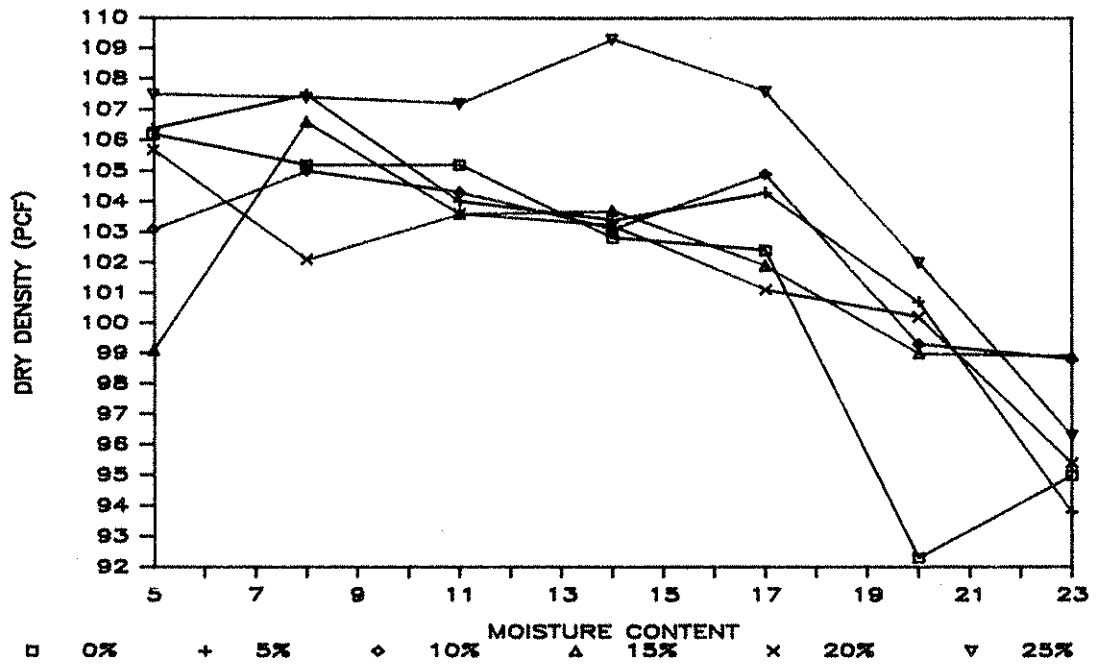


E-2  
Blend #2



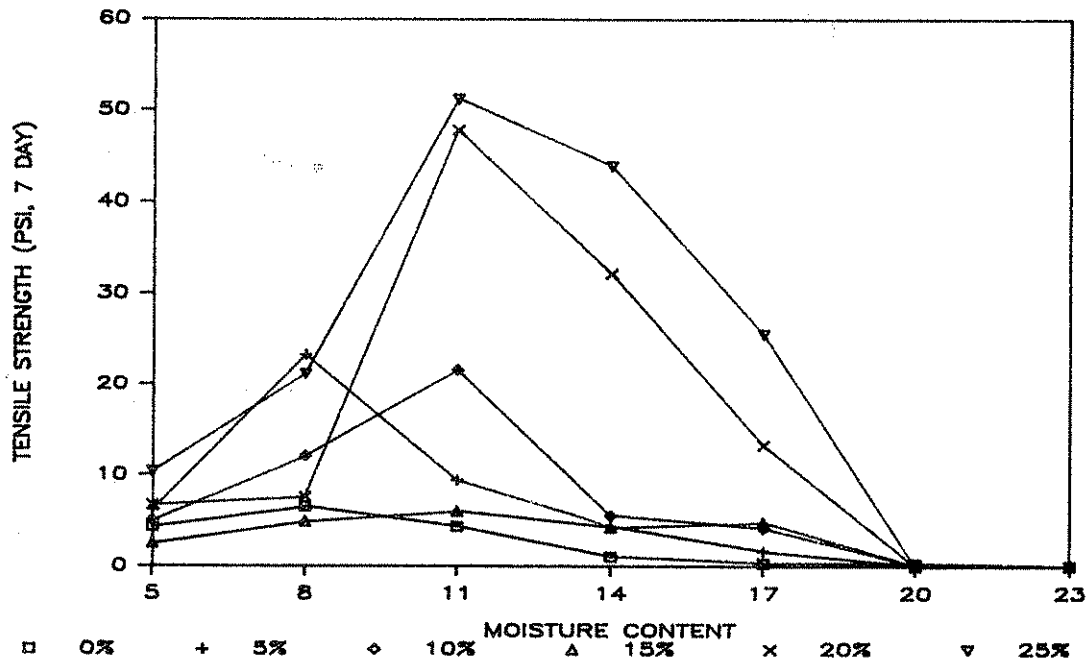
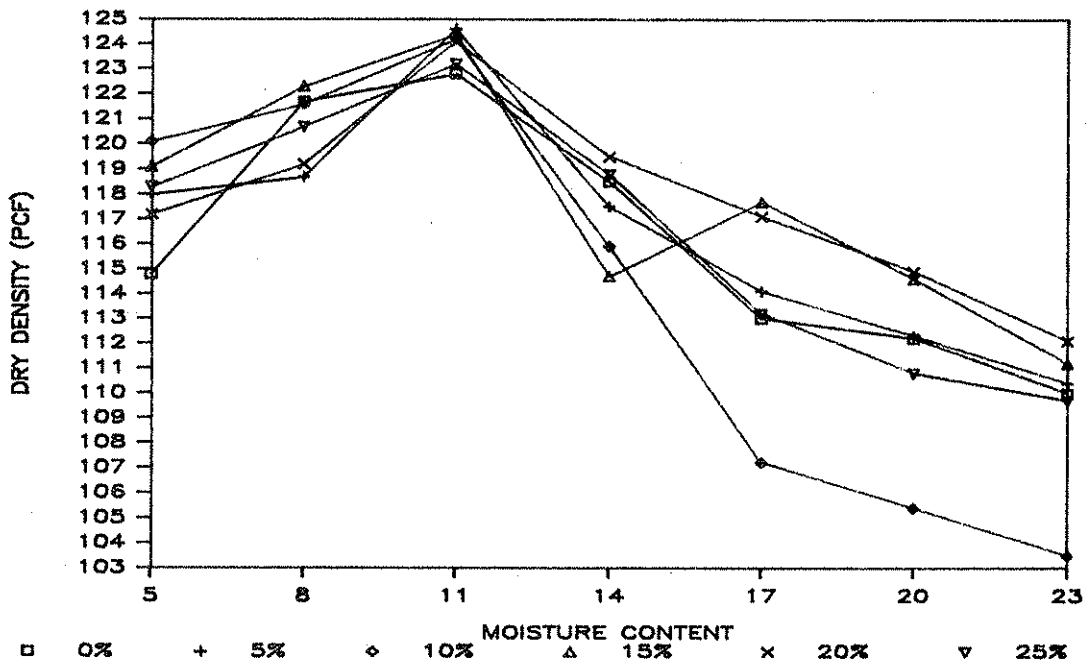
E-3

Blend #3



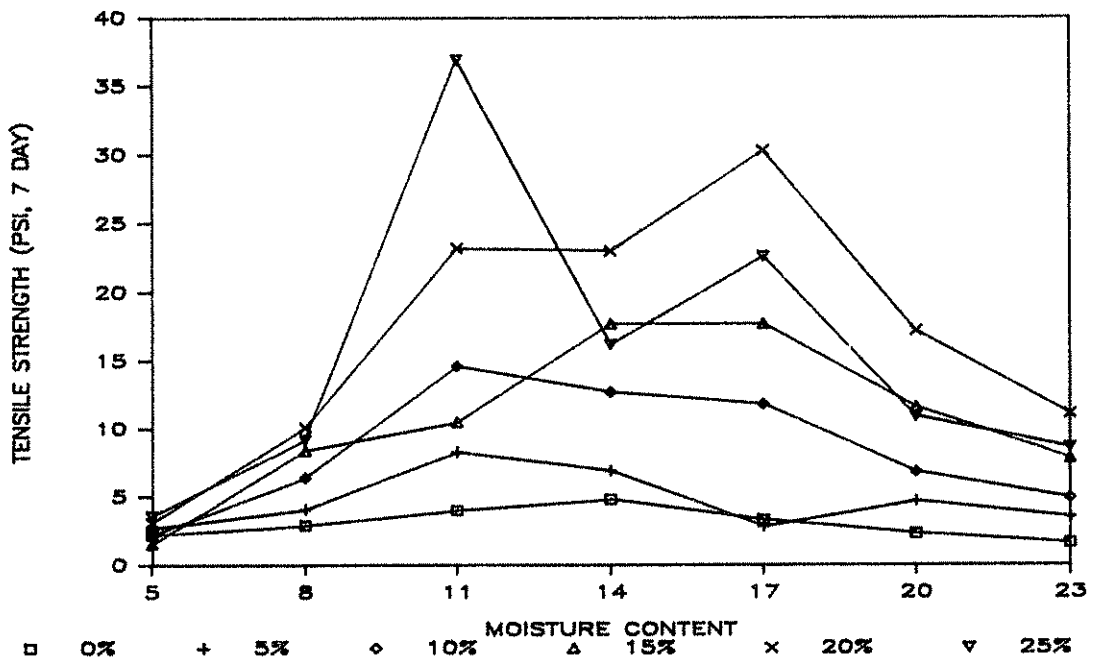
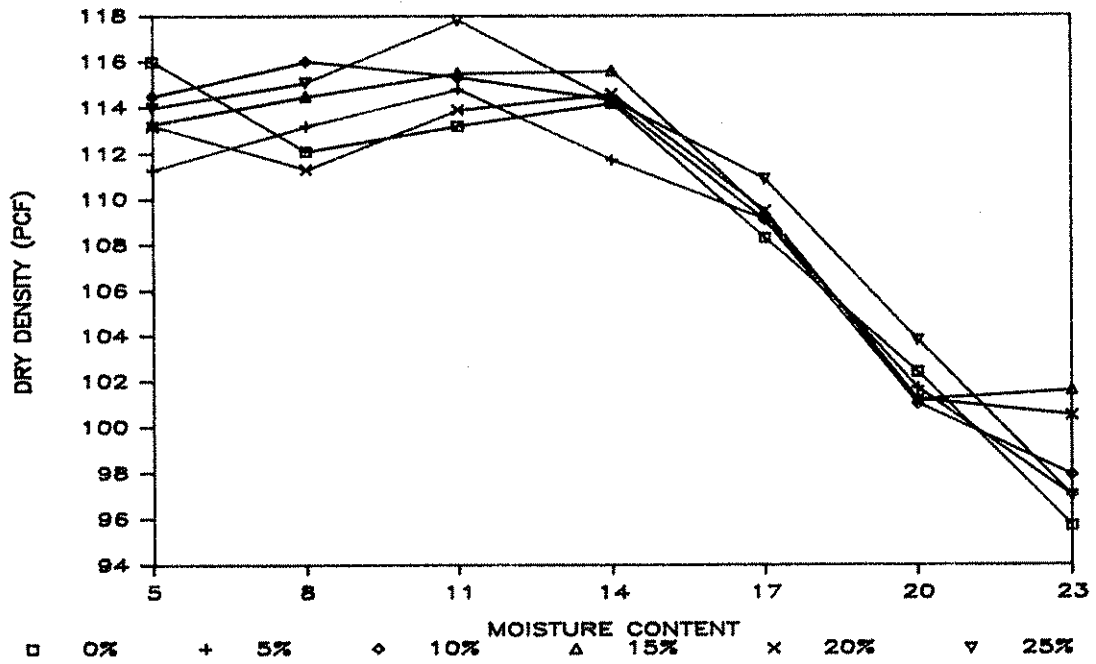
E-4

Blend #4



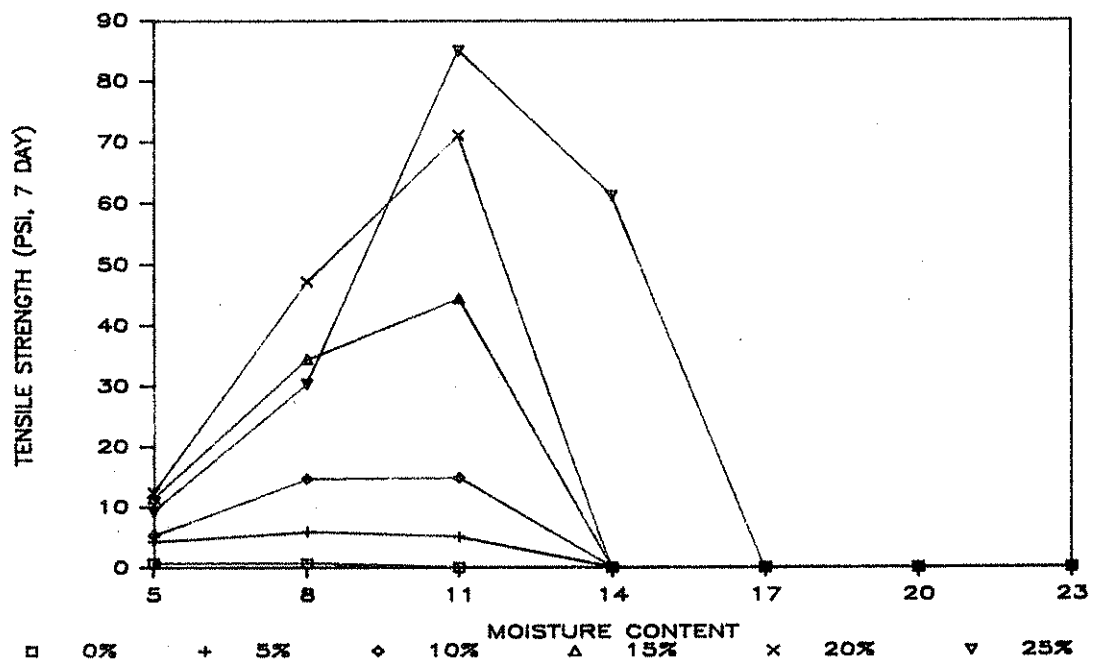
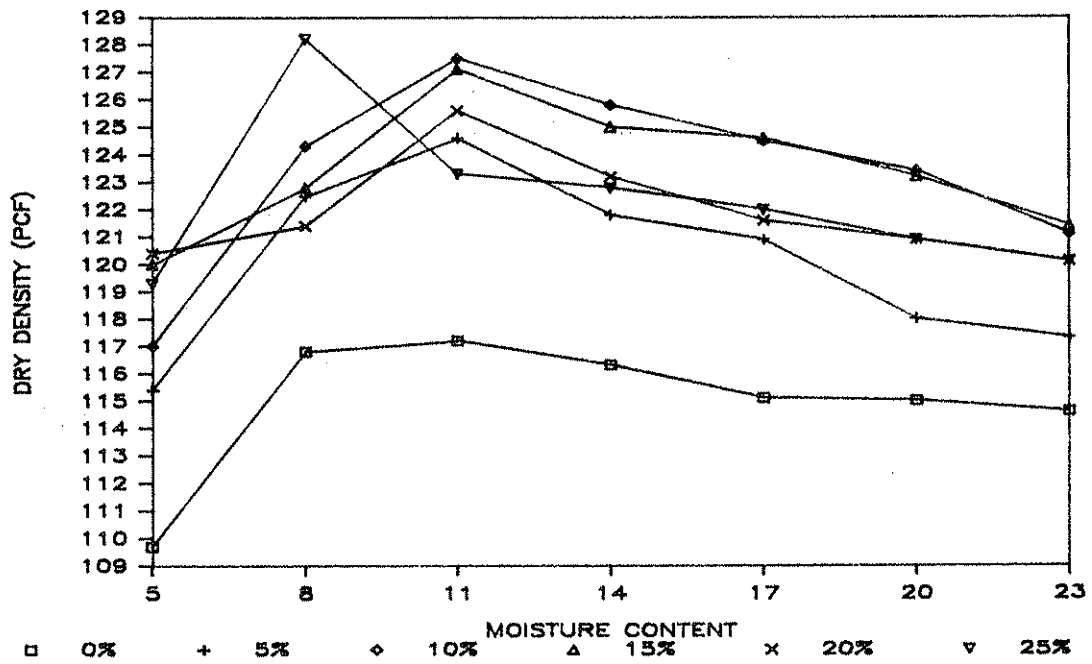
E-5

Blend #5



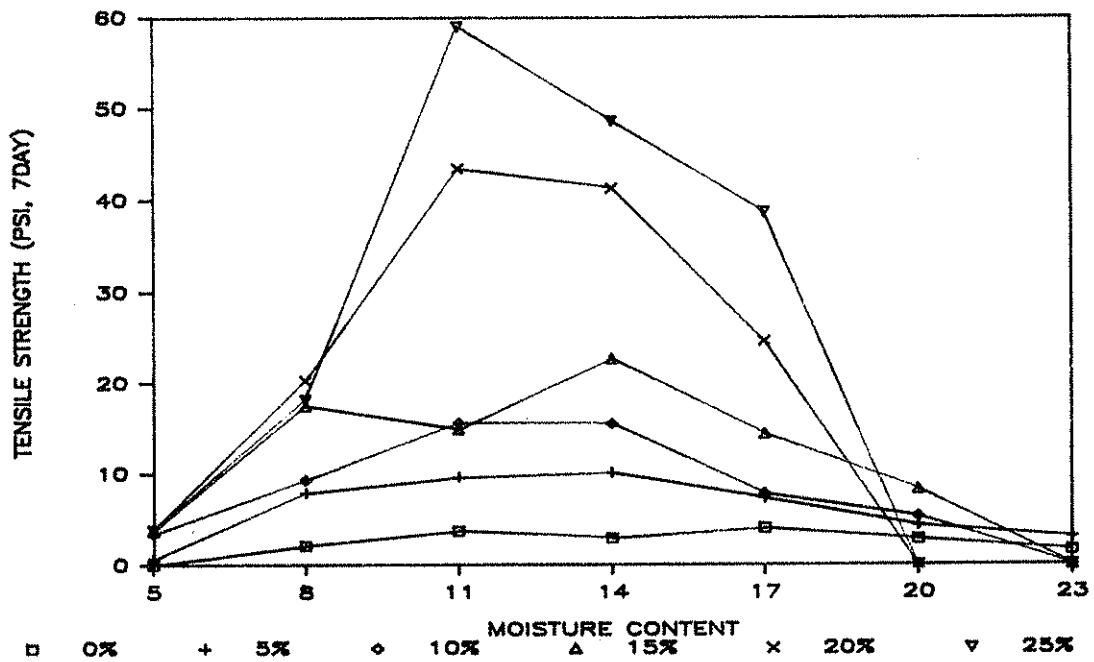
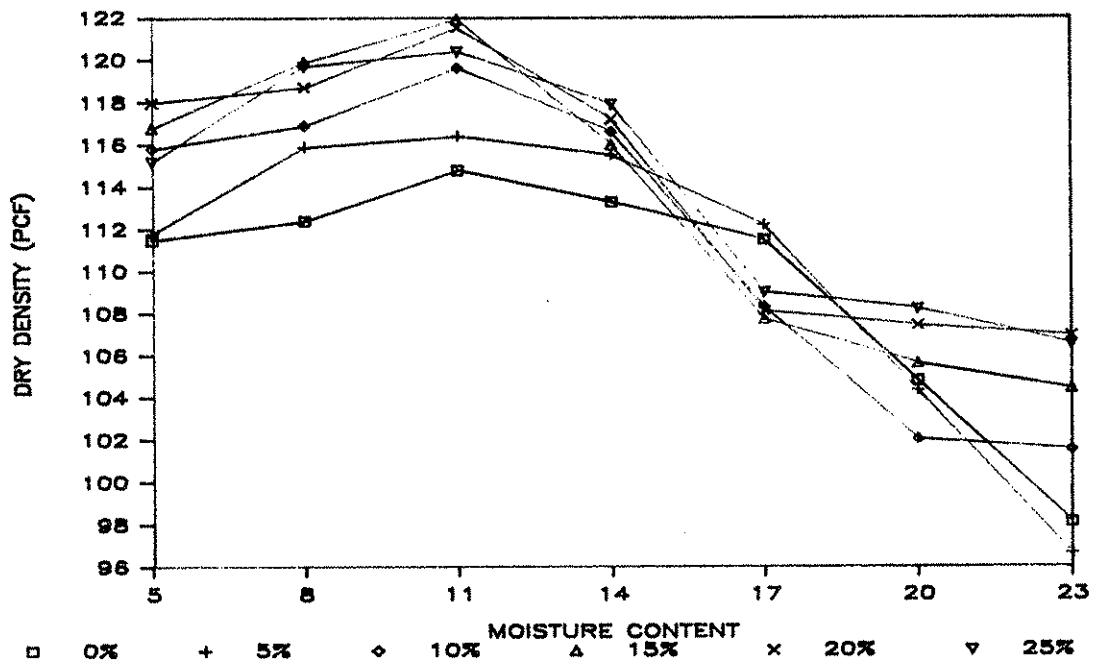
E-6

Blend #6



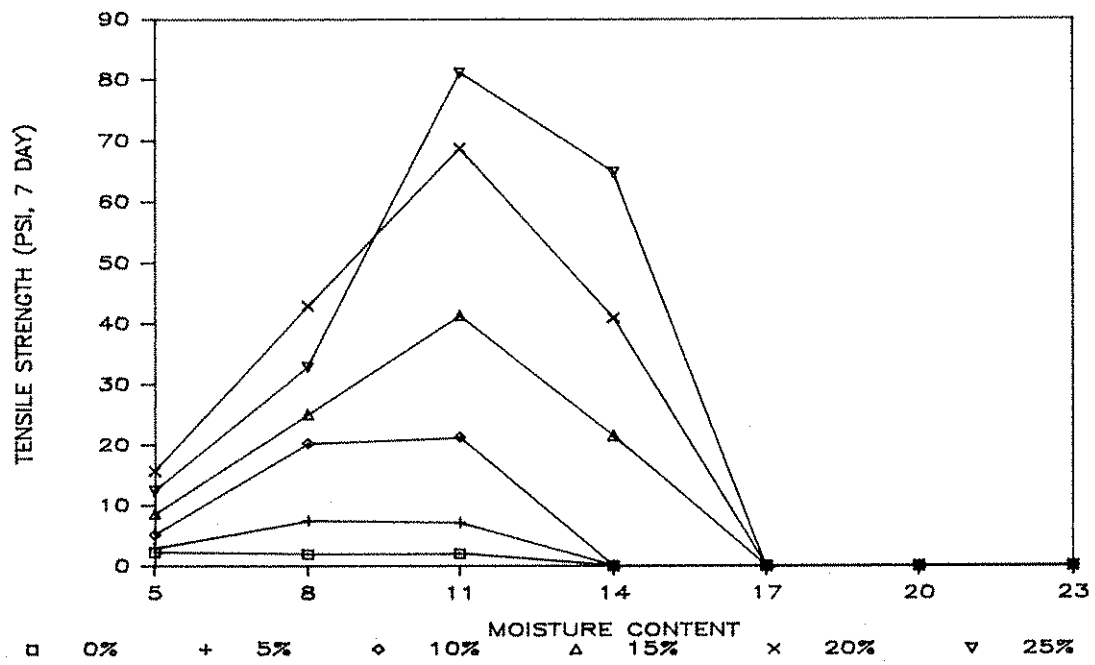
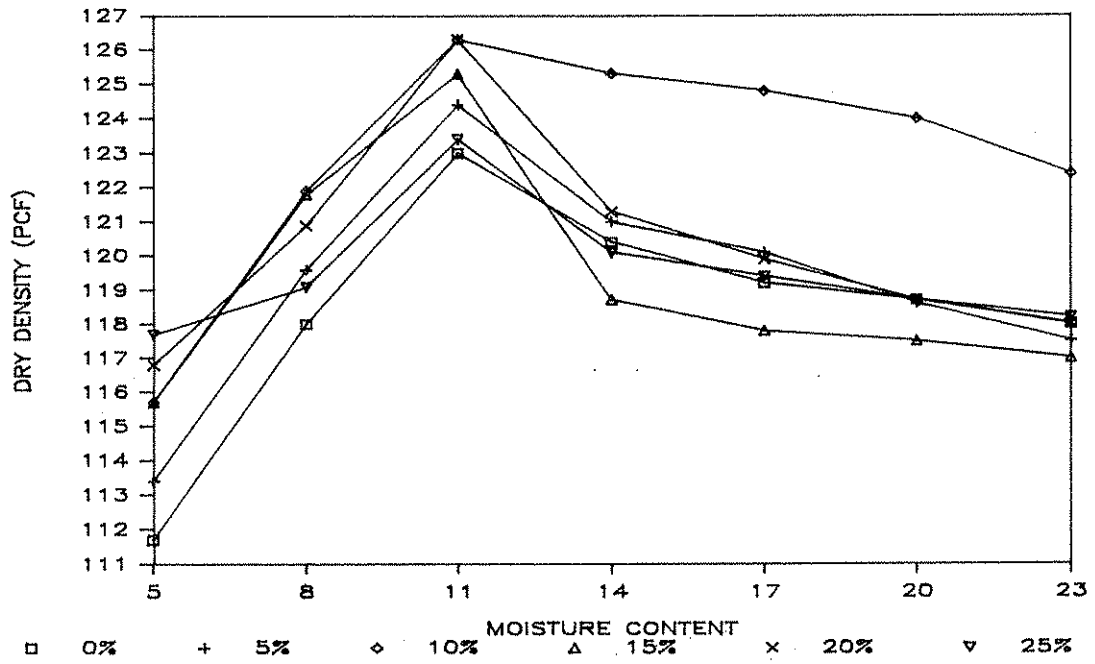


E-7  
Blend #7



E-8

Blend #8



E-9

Blend #9

