

THES

pt. 1

MICHIGAN STATE UNIVERSITY LIBRARIES



3 1293 01046 8274

LIBRARY
Michigan State
University

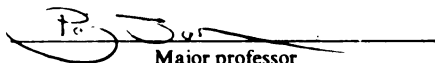
This is to certify that the

dissertation entitled
Recycling of Industrial By-Products in Concrete Construction
&
Flowable Fill:
A Comprehensive Assessment of Mix Design Variables
and Properties
presented by

Augustine Okwuegbu

has been accepted towards fulfillment
of the requirements for

Ph.D. degree in Civil Engineering


Major professor
Dr. Parviz Soroushian

Date October 27, 1993

**PLACE IN RETURN BOX to remove this checkout from your record.
TO AVOID FINES return on or before date due.**

DATE DUE	DATE DUE	DATE DUE
DEC 2 2 1998 10 66 55 57	_____	_____
10 67	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

MSU is An Affirmative Action/Equal Opportunity Institution

ct/circ/datedue.pm3-p.1

**RECYCLING OF INDUSTRIAL BY-PRODUCTS IN
CONCRETE CONSTRUCTION**

&

**FLOWABLE FILL:
A COMPREHENSIVE ASSESSMENT OF MIX DESIGN VARIABLES AND
PROPERTIES**

By

Augustine Okwuegbu

A DISSERTATION

**Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of**

DOCTOR OF PHILOSOPHY

Department of Civil and Environmental Engineering

1993

ABSTRACT

RECYCLING OF INDUSTRIAL BY-PRODUCTS IN CONCRETE CONSTRUCTION

By

Augustine Okwuegbu

Paint sludge powder, a by-product of the painting process in automobile plants, was evaluated as an expansive admixture in Portland cement concrete to compensate for shrinkage effects. The powder dosage needed to satisfy the requirements of expansive cement was determined, and specific powder types presenting the most promise for use in shrinkage compensating concrete were selected.

Chemical and mineralogical characteristics of the powders were correlated to expansive qualities, and it was concluded that calcium compounds in the powder may be the source of expansion.

A comprehensive evaluation of the action of paint sludge powders in concrete mixtures with different raw materials and mix proportions indicated that the powders are capable of performing as expansive admixture in diverse concrete mixtures.

The durability characteristics of concrete materials incorporating the powders under various physical and chemical causes of deterioration were investigated. It was concluded that the powders provide concrete with shrinkage compensating qualities without any adverse effects on long-term properties under severe exposure conditions. Concrete materials incorporating the powders were also found to be environmentally safe.

The research successfully demonstrated the potential for a high-value and large-volume utilization of an industrial by-product in construction applications.

**Copyright
by
Augustine Okwuegbu
1993**

**DEDICATED TO THE MEMORY OF MY MOTHER:
OBAJI CHRISTIANA OKWUEGBU**

ACKNOWLEDGMENTS

The work presented here was conducted under the guidance of Dr. Parviz Soroushian. The author deeply appreciates the contributions of Dr. Soroushian to this work. His great effort and very helpful supervision and advice will never be forgotten, and the author hopes that Dr. Soroushian will continue to be his mentor.

The author also would like to thank Dr. Mark Snyder, Dr. Gerald Ludden and Dr. Thomas Maleck for their help during his Ph.D. program.

The financial support for the performance of this research was provided by the Chrysler Motor Corporation and the Research Excellence Fund of the State of Michigan. The technical support was provided by the Composite Materials and Structures Center at Michigan State University. These contributions are gratefully acknowledged.

I wish to extend my sincere appreciation to Kenneth Nwele, Irene Enigwe, Godwin Afefi and Nwalo Nwankwagu for their continuous encouragement and moral support. My greatest appreciation goes to Dr. Atef Tlili for his honest and sincere contributions throughout this research.

Finally, my deepest appreciation to my father and all members of my family for their prayers, love and support.

TABLE OF CONTENTS

CHAPTER I

INTRODUCTION	1
--------------------	---

CHAPTER II

EXPANSIVE CEMENT CONCRETE	5
2.1 INTRODUCTION	5
2.2 HISTORY AND DEVELOPMENT OF EXPANSIVE CEMENT CONCRETE	8
2.3 PRODUCTION AND COMPOSITION OF EXPANSIVE CEMENT	10
2.3.1 CONVENTIONAL PORTLAND CEMENTS	10
2.3.2 EXPANSIVE CEMENTS	15
2.3.3 FRENCH CEMENT	17
2.3.4 RUSSIAN CEMENT	18
2.4 MECHANISMS OF EXPANSION	19
2.4.1 INTRODUCTION	19
2.4.2 HYDRATION CHARACTERISTICS ASSOCIATED WITH EXPANSIVE ETTRINGITE FORMATION	22
2.4.2.1 PRINCIPAL CONDITIONS FOR ETTRINGITE FORMATION	23
2.4.2.2 GENERAL SOURCES OF PRINCIPAL COMPONENTS	23
2.4.2.3 PRIMARY FACTORS INVOLVED IN PROPORTIONING OF EXPANSIVE CONSTITUENTS ..	24
2.4.2.4 SO ₃ REACTIONS OTHER THAN ETTRINGITE FORMATION	25
2.4.2.5 PRODUCTS OF REACTION CAUSING EXPANSION ..	26
2.4.2.6 ALKALI-CARBONATE REACTION	26
2.4.2.7 HEAT OF HYDRATION	27

2.4.3	THEORIES OF EXPANSION	27
2.4.3.1	CRYSTAL GROWTH THEORY	27
2.4.3.2	THE SWELLING THEORY	37
2.4.3.3	SUMMARY OF THE HYPOTHESES	40
2.4.3.4	SOME RESEARCHERS DISAGREE WITH BROWN'S THEORY (1970)	42
2.4.3.5	THEORIES OF "PROTECTIVE" AND "PARTIAL PROTECTIVE" COATINGS	44
2.5	MIX PROPORTIONING AND MANUFACTURING	48
2.5.1	INTRODUCTION	48
2.5.2	CONCRETE PROPORTIONS	49
2.5.3	EFFECTS OF ADMIXTURE ON EXPANSIVE CEMENT	53
2.5.4	CONSTRUCTION TECHNIQUES	56
2.6	DESIGN WITH SHRINKAGE COMPENSATING CONCRETE	64
2.6.1	INTRODUCTION	64
2.7	APPLICATIONS	81

CHAPTER III

DEVELOPMENT OF EXPANSIVE CEMENT WITH PAINT SLUDGE POWDER .		82
3.1	INTRODUCTION:	82
3.2	PAINT SLUDGE POWDER	83
3.3	OVERALL APPROACH	86
3.3.1	SCREENING OF THE POWDERS	86
3.4	EXPERIMENTAL PROGRAM	87
3.5	RESTRAINED EXPANSION TEST PROCEDURES	89
3.5.1	MORTAR TESTS	89
3.5.2	CONCRETE TESTS	92

3.6	EXPERIMENTAL RESULTS.	94
3.6.1	POWDER PROPERTIES	94
3.6.2	RESTRAINED EXPANSION TEST RESULTS	95
3.6.2.1	MORTAR TEST	95
3.6.2.2	CONCRETE TEST	102
3.6.3	SET TIME	109
3.6.4	FLOWABILITY	113
3.6.5	SLUMP	115
3.6.6	AIR CONTENT	119
3.6.7	UNIT WEIGHT AND YIELD	125
3.6.8	STRENGTH	129
3.6.9	MODULUS OF ELASTICITY	136
3.6.10	FLEXURAL STRENGTH	139
3.7	VARIATIONS BETWEEN SHIPMENTS	144
3.8	SUMMARY AND CONCLUSIONS	151

CHAPTER IV

MECHANISM OF EXPANSION ASSOCIATED WITH PAINT SLUDGE POWDER		152
4.1	INTRODUCTION:	152
4.2	CONTROL OF EXPANSION	154
4.3	POWDER CHEMISTRY	157
4.4	POWDER MINERALOGY	162
4.5	CONCRETE MINERALOGY	167
4.6	ASSESSMENT OF CALCIUM CARBONATE REACTION WITH CEMENT ALKALIS AS THE CAUSE OF EXPANSION.	172
4.7	CHEMICAL AND PHYSICAL PROPERTIES OF THE LOW AND HIGH ALKALI CEMENTS	175
4.8	CONCLUSION	176

CHAPTER V

EFFECTS OF MIX VARIABLES	177
5.1 INTRODUCTION	177
5.2 EXPERIMENTAL PROGRAM	178
5.3 EXPERIMENTAL RESULTS AND ANALYSIS	184
5.4 SUMMARY AND CONCLUSIONS	206

CHAPTER VI

DURABILITY CHARACTERISTICS	207
6.1 INTRODUCTION	207
6.2 EXPERIMENTAL PROGRAM	209
6.3 TEST PROCEDURES	211
6.3.1 ABRASION RESISTANCE	211
6.3.2 CHLORIDE PERMEABILITY	212
6.3.4 DE-ICER SALT SCALING	215
6.3.5 SULFATE ATTACK	217
6.3.3 FREEZE-THAW DURABILITY	219
6.3.6 CORROSION OF EMBEDDED STEEL	223
6.3.7 HOT WATER IMMERSION	225
6.4 EXPERIMENTAL RESULTS AND DISCUSSIONS	225
6.4.1 ABRASION RESISTANCE	225
6.4.2 CHLORIDE PERMEABILITY	227
6.4.3 FREEZE-THAW DURABILITY	229
6.4.4 SCALING RESISTANCE	231
6.4.5 SULFATE ATTACK	231
6.4.6 CORROSION OF EMBEDDED STEEL	233
6.4.7 HOT WATER IMMERSION	234
6.5 SUMMARY AND CONCLUSIONS	236

CHAPTER VII

GENERAL SUMMARY AND CONCLUSIONS	237
7.1 INTRODUCTION	237
7.2 DEVELOPMENT OF EXPANSIVE CEMENT WITH PAINT SLUDGE POWDER	237
7.3 MECHANISM OF EXPANSION ASSOCIATED WITH PAINT SLUDGE POWDER	238
7.4 EFFECTS OF MIX VARIABLES	239
7.5 DURABILITY CHARACTERISTICS	239

APPENDIX I

PRACTICAL GUIDELINES	242
I.1 INTRODUCTION	242
I.2 COST ANALYSIS	242
I.3 TOXICITY CHARACTERISTICS AND LEACHING	243

REFERENCES	245
-------------------	-----

LIST OF FIGURES

Figure 1 (a).	Drying Shrinkage of Concrete made with Type I Cement.	2
Figure 1 (b).	Drying Shrinkage of Concrete made with Expansive Cement.	2
Figure 2.1.	Isothermal Heat Liberation of $C_2A-CASO_4.2H_2O-Ca(OH)_2-H_2$ (Schhwiete et al, 1966)	46
Figure 2.2.	Prediction of member expansion from prism data	69
Figure 3.1	Paint Sludge Powder Production Flow Chart	85
Figure 3.2.	Restraining Mortar Cage Mold (ASTM C-806).	91
Figure 3.3	Restraining Concrete Cage Mold (ASTM C-878)	93
Figure 3.4 (a).	7-Day Restrained Expansion Test Results for Mortar Mixtures (0.5% Powder).	96
Figure 3.4 (b).	7-Day Restrained Expansion Test Results for Mortar Mixtures (1% Powder).	97
Figure 3.4 (c).	7-Day Restrained Expansion Test Results for Mortar Mixtures (1.5% Powder).	98
Figure 3.5 (a).	28-Day Restrained Expansion Test Results for Mortar Mixtures (0.5% Powder).	99
Figure 3.5 (b).	28-Day Restrained Expansion Test Results for Mortar Mixtures (1% Powder).	100
Figure 3.5 (c).	28-Day Restrained Expansion Test Results for Mortar Mixtures (1.5% Powder).	101
Figure 3.6 (a).	7-Day Restrained Expansion Test Results for Concrete Mixtures (0.5% Powder).	103
Figure 3.6 (b).	7-Day Restrained Expansion Test Results for Concrete Mixtures (1% Powder).	104
Figure 3.6 (c).	7-Day Restrained Expansion Test Results for Concrete Mixtures (1.5% Powder).	105

Figure 3.7 (a). 28-Day Restrained Expansion Test Results for Concrete Mixtures (0.5% Powder).	106
Figure 3.7 (b). 28-Day Restrained Expansion Test Results for Concrete Mixtures (1% Powder).	107
Figure 3.7 (c). 28-Day Restrained Expansion Test Results for Concrete Mixtures (1.5% Powder).	108
Figure 3.8 (a). Final Time of Set Test Results (0.5% Powder)	110
Figure 3.8 (b). Final Time of Set Test Results (1% Powder)	111
Figure 3.8 (c). Final Time of Set Test Results (1.5% Powder)	112
Figure 3.9. Effect of Paint Sludge Powder on Fresh Mix Flowability.	114
Figure 3.10 (a). Slump Test Results (0.5% Powder)	116
Figure 3.10 (b). Slump Test Results (1% Powder)	117
Figure 3.10 (c). Slump Test Results (1.5% Powder)	118
Figure 3.11. Pressure Type Air Meter	121
Figure 3.12 (a). Air Content Test Results, (0.5%)	122
Figure 3.12 (b). Air Content Test Results, (1%)	123
Figure 3.12 (c). Air Content Test Results, (1.5%)	124
Figure 3.13 (a). Unit Weight Result (0.5% Powder)	126
Figure 3.13 (b). Unit Weight Result (1% Powder)	127
Figure 3.13 (c). Unit Weight Result (1.5% Powder)	128
Figure 3.14 (a). Effects of Paint Sludge Powder on 7-Day Compressive Strength (0.5% Powder)	130
Figure 3.14 (b). Effects of Paint Sludge Powder on 7-Day Compressive Strength (1% Powder)	131
Figure 3.14 (c). Effects of Paint Sludge Powder on 7-Day Compressive Strength (1.5% Powder)	132
Figure 3.15 (a). Effects of Paint Sludge Powder on 28-Day Compressive Strength (0.5% Powder)	133

Figure 3.15 (b). Effects of Paint Sludge Powder on 28-Day Compressive Strength (1% Powder)	134
Figure 3.15 (c). Effects of Paint Sludge Powder on 28-Day Compressive Strength (1.5% Powder)	135
Figure 3.16. Various parameters that influence the modulus of elasticity of concrete.	137
Figure 3.17. Modulus of Elasticity Versus Compressive Strength of Paint Sludge Powder.	138
Figure 3.18. Arrangement of the Modulus of Rupture Test	140
Figure 3.19 (a). Flexural Strength of Paint Sludge Powder Concrete (0.5% Powder). . .	141
Figure 3.19 (b). Flexural Strength of Paint Sludge Powder Concrete (1% Powder). . .	142
Figure 3.19 (c). Flexural Strength of Paint Sludge Powder Concrete (1.5% Powder). . .	143
Figure 3.20 (a). Between Shipment Variations in 7-Day Expansion	145
Figure 3.20 (b). Between Shipment Variations in 28-Day Expansion	147
Figure 3.20 (c). Between Shipment Variations in Final Set	147
Figure 3.20 (d). Between Shipment Variations in Air Content	148
Figure 3.20 (e). Between Shipment Variations in 7-Day Compressive Strength	149
Figure 3.20 (f). Between Shipment Variations in 28-Day Compressive Strength	150
Figure 4.1. Effects of Reinforced Steel on Expansion of Concrete and Prestress Developed.	154
Figure 4.2. Effect of Different Curing Procedures on the Expansion of Type M Expansive Cement Concrete.	156
Figure 4.3 (a). Correlations Between Powder Chemistry (Calcium Content, Ca) and 7-Day Restraint Expansion.	159
Figure 4.3 (b). Correlations Between Powder Chemistry (Sulfur Content, S) and 7-Day Restraint Expansion.	160
Figure 4.3 (c). Correlations Between Powder Chemistry (Aluminum Content, Al) and 7-Day Restraint Expansion.	161

Figure 4.4 (a). Powder X-Ray Diffraction Results (PPG-SB)	163
Figure 4.4 (b). Powder X-Ray Diffraction Results (BASF)	164
Figure 4.4 (c). Powder X-Ray Diffraction Results (PPG-WB)	165
Figure 4.4 (d). Powder X-Ray Diffraction Results (PPG-SWB)	166
Figure 4.5 (a). Concrete X-Ray Diffraction Results (PPG-SB)	168
Figure 4.5 (b). Concrete X-Ray Diffraction Results (BASF)	169
Figure 4.5 (c). Concrete X-Ray Diffraction Results (PPG-WB)	170
Figure 4.5 (d). Concrete X-Ray Diffraction Results (PPG-SWB)	171
Figure 4.6 (a). Effects of Cement With Low Alkali Content on 7-Day Restrained Expansion.	173
Figure 4.6 (b). Effects of Cement With High Alkali Content on 7-Day Restrained Expansion.	174
Figure 5.1 (a). Effects and Interactions of Powder Type and content in Determining 7-Day Restrained Expansions.	190
Figure 5.1 (b). Effects and Interactions of Powder Type and content in Determining 28-Day Restrained Expansions.	191
Figure 5.2 (a). The Interaction Between Powder Type and Air content in Determining 7-Day Restrained Expansions.	192
Figure 5.2 (b). The Interaction Between Powder Type and Air content in Determining 28-Day Restrained Expansions.	193
Figure 5.3 (a). Interaction of Different Factors (Powder Content & Cement Type) in Determining Final Set Time.	195
Figure 5.3 (b). Interaction of Different Factors (Powder Content & Aggregate Content) in Determining Final Set Time.	196
Figure 5.3 (c). Interaction of Different Factors (Powder Type & Aggregate Content) in Determining Final Set Time.	197
Figure 5.4 (a). Interactions Between Different Factors (Powder Type & cement Type) in Determining Flexural Strength.	199
Figure 5.4 (b). Interactions Between Different Factors (Powder Type & Aggregate Content) in Determining Flexural Strength.	200

Figure 5.5.	Effect of Powder Content on Slump.	202
Figure 5.6.	Effect of the Dosage of Air Entraining Agent on Air Content.	203
Figure 5.7 (a).	Effects and Interactions of Powder Content and Cement Type in Determining Concrete Unit Weight.	204
Figure 5.7 (b).	Effects and Interactions of the Dosage of Entraining Agent in Determining Concrete Unit Weight.	205
Figure 6.1	Chloride Permeability Test Set Up.	214
Figure 6.2.	Specimen Used in Scaling Test.	217
Figure 6.3.	Creation of hydraulic pressure in frozen cement paste: (a) non-air-entrained paste; (b) air-entrained paste.	222
Figure 6.4.	Arrangement of Molds For Corrosion Specimen	224
Figure 6.5.	Abrasion Resistance Test Results (Means and 95% Confidence Intervals - 3 replications).	226
Figure 6.6	Rapid Chloride Permeability Test Results (Means and 95% Confidence Intervals).	228
Figure 6.7.	Freeze-Thaw Durability Results (Means and 95% Confidence Intervals).	230
Figure 6.8.	Sulfate Resistance Test Results (Means and 95% Confidence Intervals, 3 replication	232
Figure 6.9.	Corrosion Potentials Test Results (Means and 95% Confidence Interval, 3 replications).	233
Figure 6.10.	Hot Water Durability Test Results (Means and 95% Confidence Intervals, 3 replications).	235

LIST OF TABLES

Table 2.1	Oxide composition	7
Table 2.2	Average Composition of Standard Types of Cement (From T.C. Hansen 1966)	15
Table 2.3	Trial Mixture guide for Shrinkage Compensating Cement Concrete	51
Table 2.4	Recommended Slumps at Placements	57
Table 2.5.	Typical Tensile Strain Crack Capacity for Normal Weight Concrete.	75
Table 2.6.	Typical Comparative Residual Strains for Reinforced Normal Weight Concrete.	76
Table 2.7.	Typical Comparative Residual Strains for Reinforced Light-Weight Concrete.	76
Table 3.1.	Mix Designs of Experimental Program	88
Table 3.2.	ASTM C-845 Physical Requirements for Expansive Cement Mortar	89
Table 3.3.	Properties of the Paint Sludge Powders and Type K Cement	94
Table 3.4	Recommended air content of concretes containing aggregates of different maximum size, according to ACI 201.2R-77, (BS 8110: Part 1:1985).	119
Table 4.1.	Variables Affecting Expansive Cements	155
Table 4.2.	Chemical Analysis of the Chrysler Paint Sludge Powders	158
Table 4.3	Chemical and Physical Properties of the Alkali Cement	175
Table 5.1.	Fractional Factorial Design of Experiments.	179
Table 5.2.	Properties of Cement Used.	183
Table 5.3.	Results of Fractional Factorial Design of Experiments.	185
Table 6.1.	Interpretation of the Chloride Permeability Output (AASHTO T-277-831) . .	213
Table 6.2.	Minimum Moist-Curing Times to Develop Salt Scaling Resistance	216
Table 6.3.	ASTM C-876 Half-Cell Test Classification	224
Table 6.4	Scaling Resistance Test Results.	231
Table I.1.	Paint Sludge Powder Toxicity Results	244

CHAPTER I

INTRODUCTION

1.1 INTRODUCTION

Literature review on expansive and shrinkage compensating concrete is presented in the following headings of expansive cement concrete; mechanism of expansion; mix proportioning and manufacturing; design with shrinkage compensating concrete; and applications.

One of the major disadvantages of Portland cement concrete is its susceptibility to tensile cracking when volume contractions associated with drying shrinkage are wholly or partially restrained (Figure 1a). Shrinkage cracking is unsightly and destroys the integrity of concrete; special costly allowances must be made in design and construction of concrete pavements and structures in order to reduce the restraint against shrinkage movements. [1, 10]

Volume expansions during moist curing in ordinary Portland cements are very small; if greater expansions could be reached during hardening (e.g., through the use of expansive cements), the effects of contractions that occur upon drying could be offset (Figure 1b).

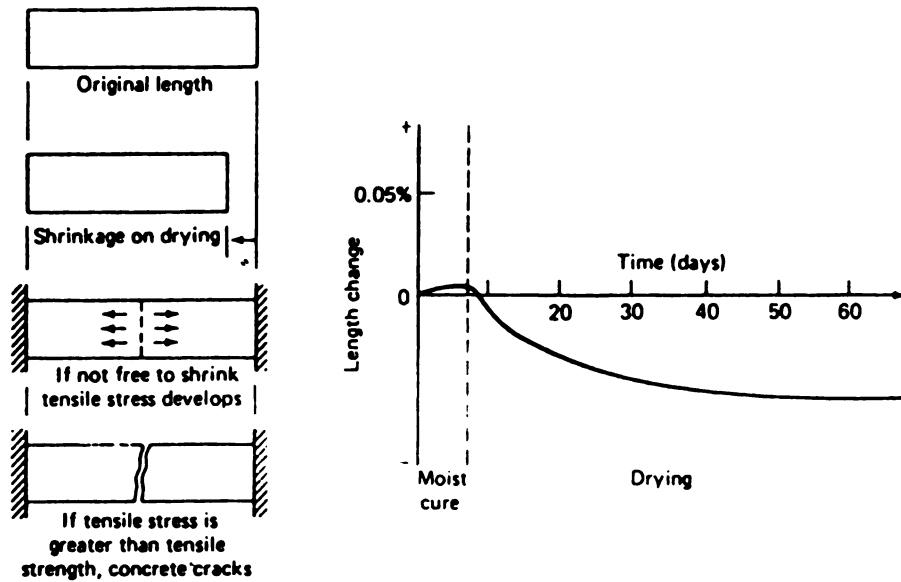


Figure 1 (a). Drying Shrinkage of Concrete made with Type I Cement. [10]

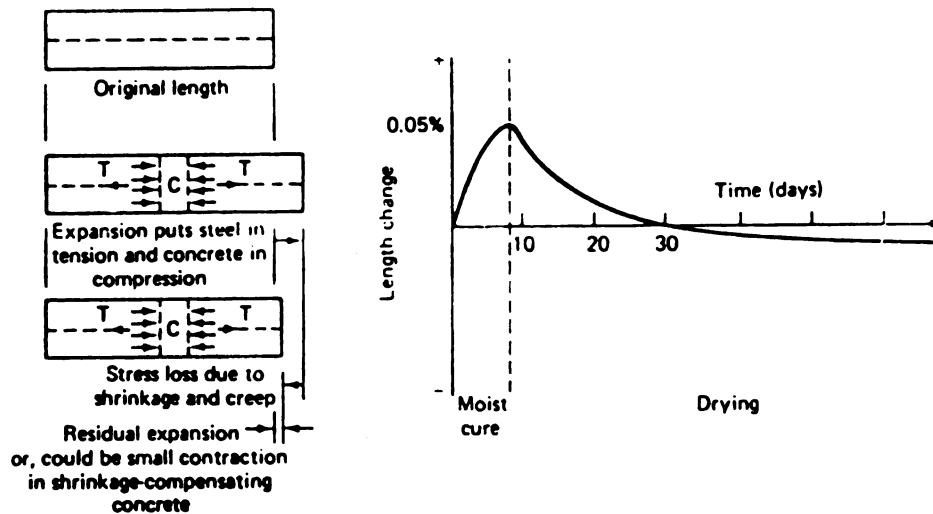


Figure 1 (b). Drying Shrinkage of Concrete made with Expansive Cement. [10]

Expansive cements have been used in a wide variety of concrete structures. One of the more frequent uses in the United States has been in parking structures, to prevent water leaks that can cause damage to cars. The largest such structure is the parking building at O'Hare International Airport in Chicago, which used 120,000 yd³ (90,000 m³) of shrinkage-compensating concrete. In concrete pavements using expansive cement, the shrinkage control joints can be combined with thermal expansion control joints and other construction joints; the elimination of shrinkage control joints is an attractive advantage in the laying of pavements. The largest paving job to date using this material is probably at the Love Field Airport at Dallas-Fort Worth, where more than 150,000 yd³ (1150,000 m³) of shrinkage-compensating concrete was used in taxiway. Use of Shrinkage-compensating concrete in bridge deck overlays can help minimize cracking and control the corrosion of steel in bridge decks. In patching of concrete pavements, where shrinkage can cause separation, shrinkage compensating concrete has found broad applications. [9, 10]

Expansive cement can also be used in concrete structures where water-tightness is an important requirement, such as water storage tanks, swimming pools, and ice rinks. Shrinkage compensating concrete has also been used in tilt-up construction, where residual prestress helps elements withstand the stresses imposed during lifting, and minimizes the separations between elements. It has long been recognized that hydration reactions in general result in an increase in the specific volume of solids. However, the fact that an increase in solid volume occurs does not necessarily mean that an expansion

takes place. As a matter of fact, when all of the reactants, including water, are taken into account, it can be shown that during the calcium sulfoaluminate expansive reaction the reactants undergo a net reduction in volume. Consequently, as Mehta points out, the explanation for expansion associated with ettringite formation is more involved than simple volume increase as the reactants convert into the reaction products.

Perhaps the most generally accepted view of the calcium sulfoaluminate expansive process is one based on the topochemical* growth of crystals of ettringite on the surface of grains of C_3A and other alumina bearing phases. The probable nature of this topochemical* growth is discussed by Schwiete et al: crystals of ettringite growing on the surface of the grain forms an expanding layer which is constantly forced outward by the crystallization pressure of the growing crystals inside the layer. Other consequences of this process appear to include a reduction in the rate of ettringite formation caused by the protection of the C_3A surface by the dense ettringite layer (this mechanism is thought to be responsible for the retarding effect of gypsum in the hydration of Portland cement), and subsequent and perhaps repeated bursting of the layer, allowing at least temporary increases in the reaction rate. [3, 4, 5]

CHAPTER II

EXPANSIVE CEMENT CONCRETE

2.1 INTRODUCTION

ACI Committee 223, in its report (ACI, 1970) on expansive cement concretes, defines the nomenclature pertaining to expansive cement concrete as follows: Expansive cement is a cement which when mixed with water forms a paste that, during and after setting and hardening, increases significantly in volume. [2, 6, 9]

Expansive concretes are divided into two categories, shrinkage-compensating and self-stressing. The ACI definitions are: Shrinkage-compensating concrete is an expansive cement concrete in which expansion, if restrained, induces compressive stresses which approximately offset tensile stresses in the concrete induced by drying shrinkage.

Self-stressing concrete is an expansive cement concrete in which expansion, if restrained, induces compressive stresses of a high enough magnitude to result in significant compression in the concrete after drying shrinkage and creep strains have occurred. [7, 8]

Shrinkage compensating concrete is generally made with one of the three main categories of expansive cement: Expansive component is the material interground with Portland cement clinker to obtain expansive cement. It is made up of the anhydrous calcium sulfoaluminate ($\text{CaO} \cdot 3\text{Al}_2\text{O}_3 \cdot \text{SO}_3$), calcium sulfate (CaSO_4), and free lime (CaO) as well as other known portland cement compounds.

Expansive cement Type K is a mixture of portland cement compounds, anhydrous calcium sulfoaluminate ($4 \text{ CaO} \cdot 3 \text{ Al}_2\text{O}_3 \cdot \text{SO}_3$), calcium sulfate (CaSO_4), and lime (CaO). The anhydrous calcium sulfoaluminate is a component of a separately burned clinker blended with portland cement or, alternatively, it may be formed simultaneously with the Portland clinker compounds during the burning process.

Expansive cement, Type M, is either a mixture of portland cement, calcium aluminate cement, and calcium sulfate or an interground product made with portland cement clinker, calcium aluminate clinker, and calcium sulfate.

Expansive cement Type S is a Portland cement containing a large C_3A content and modified by an excess of calcium sulfate above the usual amount found in other Portland cements. [13, 27]

Recently expansive admixtures have also been developed which convert a conventional concrete mixtures into shrinkage compensating concrete.

Ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) is the phase formed during the hydration of expansive cements which is the source of the expansive force; it is comparable to the natural mineral of the same name. This high calcium sulfoaluminate is also formed by sulfate attack on mortar and concrete, and was designated as 'cement bacillus' in older literature." All expansive cements consist predominantly of Portland cements, about 94 to 70 percent. The chemical compositions are similar to those of portland cement except for higher sulfate contents (about 4.5 to 6.5 percent S), generally higher alumina contents (about 5 to 9 percent) and generally lower silica contents. The lower the amount of the

expansive agent the greater is the similarity in compositions. Representative oxide compositions of the three types of shrinkage-compensating cements currently being produced in the United States are given in the following Table.

Table 2.1 Oxide composition [3]			
Property	Type of Cement		
	K	S	M-X
SiO ₂ , %	21.6	18.3	18.4
Al ₂ O ₃	4.8	8.8	7.7
Fe ₂ O ₃	2.8	1.9	2.1
CaO	62.4	63.7	61.1
MgO	0.7	1.1	3.0
SO ₃	6.2	5.3	4.7
Na ₂ O	0.18	0.04	0.04
K ₂ O	0.18	0.3	0.04
LOI	1.06	-	-

Three types of shrinkage-compensating cements are characterized by different alumina-bearing agents, C_4A_3S in Type K, $CA=C_{12}A_7$ (calcium aluminate cement) in Type M-X, and C_3A in Type S. The sulfate is present as gypsum, hemihydrate, anhydride and, in part in Type K cement, as C_4A_3S . There remains little doubt that ettringite formation is the source of expansive energy according to the reaction, $6C+A+3S+32H-C_3A\cdot H_{32}$. No one of the oxides actually occurs as a reactant except water for in part. Phase rule dictates expression of the reactants as oxides in a general reaction. [21, 28, 30]

2.2 HISTORY AND DEVELOPMENT OF EXPANSIVE CEMENT CONCRETE

The transition from science to engineering was made when it was first suggested to use the disruptive expansive component and put its forces to work towards chemical prestressing. Candlot, in 1890, discovered ettringite in the form of the reaction product of tricalcium aluminate and calcium sulfate in an aqueous solution. He reported it as $5.5 \text{ CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2.5 \text{ SO}_3 \cdot 59\text{H}_2\text{O}$. This has been referred to as "Candlot's Salt."

Two years later, in 1892, Michaelis suggested that ettringite was responsible for the destructive expansion of Portland cement concretes in the presence of environments containing sulfates (ACI. 1970). He reported the compound discovered by Candlot to be $6 \text{ CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{SO}_3 \cdot 30\text{H}_2\text{O}$, which has the same composition and structure as the naturally occurring mineral ettringite as reported by Lehman in 1847. The first realization of potential uses for ettringite in offsetting the drying shrinkage is credited to French researchers in Lafarge Cement Company in France in the 1920's. But their tests proved to be unsuccessful. [1, 20]

Lossier, in 1925, introduced expansive cements. His objective was chemical prestressing of concretes. The findings were not introduced to the market since expansion was uncontrollable. In 1958, Alexander Klein (Klein and Troxell, 1958), at the University of California in Berkeley, introduced anhydrous calcium sulfoaluminate-bearing cement-- $\text{C}_4\text{A}_3\text{S}$, as reported by Fukuda (1968). Its three major characteristic XRD peaks were reported to be $23.72^\circ 2\theta$, $33.82^\circ 2\theta$, and $41.62^\circ 2\theta$, with relative intensities of 100, 30, and 25 percent respectively. Klein suggested that upon hydration,

C_4A_3S is better for controlling the formation of ettringite. Its successful field trials, conducted in 1963, laid the foundation to fulfill the promising advantages of expansive cements. [42]

Type K expansive cement is named after Alexander Klein who patented it in 1961. Seven years later, in 1968, Type S expansive cement was introduced by Greening from Portland Cement Association. In 1970, Universal-Atlas Division marketed Type M expansive cement which was produced by Monfore a decade earlier.

The Soviets were also involved in extensive research on expansive cements. They developed a water-proof expansive cement (WEC) in 1942. This was done by dry-grinding of high alumina cement (HAC), gypsum artificially made hydrocaluminate which is a dry mixture of HAC and lime ground together. This was used for repair purposes because of its water-tightness and quick-hardening characteristics. They also developed a self-stressing cement by using finely ground portland cement, HAC, and gypsum. However, this was uneconomical because it had to be cured by a hydrothermal process (Kerdegari, 1978).

Expansive cements were introduced in Japan in 1965, and the amount of usage has been gradually increasing ever since. The development of expansive cement in Japan has been very similar to that of Type K expansive cement which expands by formation of ettringite. However, there is also a different type of expansive component which expands by the action of a specially processed calcium oxide (Kokubu, 1973)--a form of free-lime expansion. [22, 23, 48]

2.3 PRODUCTION AND COMPOSITION OF EXPANSIVE CEMENT

Before proceeding to a description of various types of expansive cement, this section presents an overview of portland cement production.

2.3.1 CONVENTIONAL PORTLAND CEMENTS

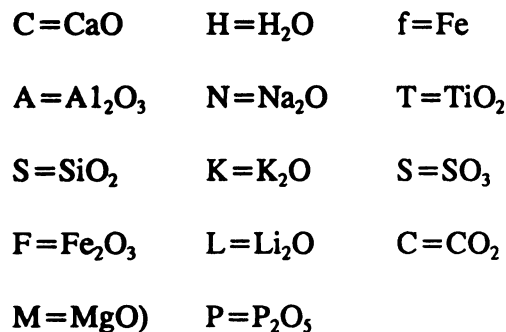
Portland Cement is made from raw materials containing Lime, silica, and alumina. Natural calcareous deposits, such as limestone and shell beds, provide the lime while natural argillaceous deposits, such as clay, shale and slate, provide the silica and alumina.

These natural deposits also contain iron, which gives the grayish color of the cement. They also contain magnesia, alkalies, phosphates, and other impurities. Some of these have deleterious effects if they appear in large quantities (Troxell et al, 1968).

The natural raw materials, based on chemical analyses, are proportioned to a desired composition and ground in either dry or slurry form (dry or wet, respectively). The prepared mixture is fed into a kiln and heated to temperatures ranging from 1600 °C to 1900 °C (2912 °F to 3452 °F). The high temperature in the kiln produces reactions among the lime-silica-alumina-iron-impurities under oxidizing conditions. The product, called clinker, contains main phases such as tricalcium silicate ($3\text{CaO} \cdot \text{SiO}_2$), dicalcium silicate ($2\text{CaO} \cdot \text{SiO}_2$), tricalcium aluminate ($3\text{CaO} \cdot \text{Al}_2\text{O}_3$), and tetracalcium aluminoferrite ($4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$). Some minor constituents, such as free lime (CaO), periclase (MgO), and alkalies are also included. [9, 19, 51]

After burning, the clinker is cooled, stored, and then ground with a small amount of calcium sulfate mineral, for example, gypsum, to give cement controlled setting characteristics. Usually the fineness ranges from 2,000 to 4,000 cm²/g specific surface.

Chemical constituents of cement can be represented as follows:



Using these abbreviations, the four main components of portland cements are:

Tricalcium silicate (3CaO.SiO₂=C₃S)

Dicalcium silicate (2CaO.SiO₂= C₂S)

Tricalcium aluminate (3CaO.Al₂O₃= C₃A)

Tetracalcium aluminoferrite (4CaO.Al₂O₃.Fe₂O₃=C₄AF)

The Bogue equations are well known to cement chemists, and they are used to calculate with reasonable accuracy the amount of each of the major phases in Portland cement by knowing the oxides from chemical analyses. They are as follows:

$$C_3S = 4.0710C - 7.6024S - 1.4297F - 6.7187A$$

$$C_2S = 8.6024S + 5.0683A - 3.0710C$$

$$C_3A = 2.6504A - 1.6920F$$

$$C_4AF = 3.0432F$$

If the chemical analysis data includes the contributions from gypsum, then the Bogue equations for C_3S and C_2S can be modified by subtracting $2.8516S$ from C_3S and adding $2.1512S$ to C_2S (Kerdegari, 1978). If the ferrite phase composition does not occur as C_4AF , then the main error would be in C_3A value and a modified Bogue equation is introduced, assuming the composition to be $C_4A_{0.72}F_{1.28}$ (Midgley, 1964). Copeland and Kantro (1964) reported that the ferrite phase generally can vary in composition from C_6AF_2 to C_6A_2F .

Kantro et al (1964) determined quantitatively the amounts of the major phases in Portland cements by using the x-ray diffraction methods. A major advantage over the Bogue equations is that using the x-rays gives information on the individual phases actually existing in the cement. For example, the Bogue equations are based on the assumption that the clinker has reached equilibrium in the kiln and also that the A/F mole ratio of the ferrite phase is unity (C_4AF). It is also assumed that the silicate phases are free from other constituents such as alumina. The use of XRD does not require these assumptions and can give more accurate values of the existing amounts of the phases. It was found that the potential C_3A contents, as indicated by the Bogue equations, are higher than the x-ray values in most cases simply because some of the alumina is embedded in the silicate phases and the Bogue equations are assigning these alumina to the C_3A phase. Nevertheless, except for C_3A , the compositions obtained from x-ray analysis did not differ significantly from those obtained from Bogue's potential compound composition. [4, 9, 14, 20, 22]

In recent times, the content of lime has been increasing and silica has been decreasing in the composition of Portland cement. This is a result of improvements in the methods of manufacture by which cements of higher lime contents can be produced without reducing the soundness. The purpose is that higher lime content in the kiln during the heating process will increase the tricalcium silicate content of cements. This will result in a more rapid development of strength during the hydration process (Lee, 1956). The calculated potential compound compositions are a valuable information for determining and comparing the general characteristics of Portland cements, and they are extremely important to cement chemists and manufacturers.

Since C_3A , C_3S , C_2S , and C_4AF have different crystalline structures, they combine at different rates with water to produce different products serving different purposes. Studies on pure compounds indicate that the rate of hydration of C_4AF is the largest, followed by C_3A , C_3S , and C_2S , respectively (Brunauer, 1957). Hydration of CS usually takes a few months while C_2 sometimes takes years to complete its reaction (Copeland et al, 1960). Thus, cements containing different proportions of these compounds would behave differently.

There are five major commercially-available portland cements, all classified according to the relative proportions of the major compounds C_3S , C_2S , C_3A , and C_4AF . Table 2.2 (from T. C. Hansen, 1966) describes the five types with approximate percent of potential compound compositions. [13, 43]

Portland cement Types I and II are usually used for regular concrete structures. If the content of C_3A and C_3S increases, the cement becomes Type III. In the presence of large amounts of C_3A and C_3S the cement hardens at early ages and for this reason Portland cements that are low in C_3A and C_3S are used for mass concrete where the amount of heat liberated by phases such as C_3A and C_3S (due to their high heat liberation upon hydration with water) become of importance when thermal stresses are developed in pipes and reinforcements embedded in the mass concrete. This type of cement is called Portland cement Type IV or low-heat Portland cement. Portland cement Type V or sulfate resisting portland cement contains low amounts of C_3A , C_4AF , and MgO and is used where there is a danger of sulfate attack by water carrying sulfates, for example, sea water. [5, 47]

Table 2.2 Average Composition of Standard Types of Cement (From T.C. Hansen 1966)					
ASTM Type	I Standard	II Moderate Heat	III Rapid Hardening, High Early Strength	IV Low Heat	V Sulphate Resistant
Applications	Regular Concrete Structures	Mass Concrete	Winter Concrete	Mass Concrete	Sulphate Water Sea Water
	<u>Percent</u>	<u>Percent</u>	<u>Percent</u>	<u>Percent</u>	<u>Percent</u>
C ₃ S	53	47, max 50	58	26, max 35	max 50
C ₂ S	24	32	16	54, min 40	
C ₃ A	8	3, max 8	8, max 15	2, max 7	max 5
C ₄ AF	8	12	8	12	

2.3.2 EXPANSIVE CEMENTS

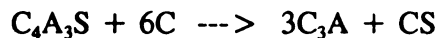
Expansive cements are also available in addition to Portland cement types I through V.

The three types of expansive cements used in the United States are differentiated by their alumina-bearing phases. For example, Type K expansive cement which contains the expansive clinker is characterized by C₄A₃S. Similarly, Types M and S are characterized by CA+C₁₂A₇ and a high percentage of C₃A, respectively.

Type K expansive cement which is usually produced by intergrinding ordinary Portland cement clinker, expansive clinker (containing C₄A₃S), and a sulfate source (like anhydrite or gypsum) is the one mostly utilized in the US. The expansive clinker

is burnt separately from raw materials containing lime, alumina, and sulfates in rotary **kilns** in the same manner as ordinary portland cement clinker, at a temperature of **approximately 1300 °C (2372 °F)**, which is 300 to 600 °C (572 to 1112 °F) less than **ordinary** portland cement clinkers. [9, 11, 12]

The sulfate acts as a fluxing agent and at this temperature, the formation of C_3A **will** form at the expense of C_4A_3S and free lime, according to the chemical equation **below** (Mehta and Polivka, 1974):



The clinker is cooled and then crushed to a fineness equivalent to the Portland **cement** clinker. The crushing energy it requires to obtain the similar fineness is less than **that** required for portland cement clinker (Burciaga, 1977).

On the assumption of chemical equilibrium inside the kiln, and on the further **assumption** that no C_3A is present, Mehta (1974) computed the following equations to **determine** the compound from oxide analysis:

$$C_3S = 4.070C - 7.6S - 2.239A - 4.287F - 2.851S$$

$$C_2S = 8.600S + 1.689A + 3.235F + 2.150S - 3.070C$$

$$C_4A_3S = 1.995A - 1.273F$$

$$CS = 1.700S + 0.284F - 0.445A$$

$$C_4AF = 3.043F$$

The CaO (C) which is to be used in the equations is the total CaO minus free CaO **(uncombined)** as determined by the chemical analysis.

Alexander Klein (1958) developed similar equations earlier but he assumed C_4A_2F to occur instead of C_4AF . However, on the basis of x-ray diffraction, Mehta showed that the composition of the ferrite phase is closer to C_4AF . The expansive clinkers in the United States are developed to contain eight percent to 50 percent C_4A_3S (Mehta and Polivka, 1974). They also contain CS which helps to keep the C_4A_3S stable during the burning process. Percentages of oxides and calculated compound compositions typical of commercially available Type K expansive clinkers were given earlier in Table 2.2, based on Mehta's equations. [5, 45, 52]

2.3.3 FRENCH CEMENT

Initially, the expansive cement produced in France by the Lafarge Company was a mixture of Portland cement, gypsum and aluminous cement. It was not successful since the reaction was erratic and difficult to control.

Later developments by Lossier used a mixture of Portland cement, a ground clinker, and a stabilizer. The clinker was obtained by burning a mixture of gypsum, bauxite, and chalk. The stabilizer was blast furnace slag and was used to control the reaction by combining with the CH, reducing its concentration in the liquid phase and thus affecting the formation of the trisulfate hydrate. The cement was not successful due to difficulties in controlling and reproducing the expansion. Originally, the ground clinker was thought to be an anhydrous calcium sulfoaluminate. However, in 1952, Lafuma (29) reported results of X-ray analysis of the clinker and found no anhydrous

calcium sulfoaluminate (ternary C-A-S compound) but only a mixture of free anhydrous **CS**, calcium aluminated (mainly C_3A_3 and C_2S (dicalcium silicate)). Based on later **information**, the temperature of the clinker burning must have been below that required **for the** formation of the ternary compound. However, Lafuma concluded wrongly that **no benefit** is achieved by separate clinker burning and that the same trisulfate hydrate **expansion** characteristics could be obtained by using a mixture of high-alumina cement **and** calcium sulfate. [3, 5]

2.3.4 RUSSIAN CEMENT

A number of expansive cements have been used in Russia. Cements with low **expansion** had an expansive component made of a mixture of calcium aluminate hydrate, **obtained** by the interaction of lime on aluminous cements or on calcined clay, and **gypsum**. In 1957, Mikhailov reported cements with high expansion used for chemically **prestressed** concrete. These were made by intergrinding aluminous cement, Portland **cement** and either gypsum or plaster of Paris. Because of the large amount of A ions **available**, immediately on hydration, the monosulfate (C_4ASH_{12}) is formed rather than **the trisulfate** ($C_6AS_3H_{32}$). Thus the initial expansion is low and the danger of disruption **exists** on recrystallization to the trisulfate hydrate. Mikhailov achieved proper control **by using** a very low water-cement ratio (e.g., 0.16), permitting the material to harden **at ambient** temperature for up to 24 hours and then subjecting the hardened structure to **hydrothermal** treatment for some 6 hours at about 70-100 °C. This treatment is

effective because with the disruptive formation of the trisulfate hydrate, the hydration of the Portland cement is accelerated, due to the high temperature, and a balanced strength-expansion relation is achieved. However, the type of hydrothermal treatment required limits the structural use of this expansive cement. [3, 4, 5, 7]

2.4 MECHANISMS OF EXPANSION

2.4.1 INTRODUCTION

The same reactions that cause damage to the concrete, when allowed to progress uncontrolled, also are responsible for the expansion associated with expansive cements. Further, although these reactions are accompanied by a chemical shrinkage, that is, a decrease in the overall volume of the reacting system (solids + water), there is a very significant increase in the original solid volume. There is sufficient evidence that a topochemical* reaction mechanism is the main reason for the observed expansion.

To produce expansion without cracking, the time at which the expansive reaction takes place during the hardening process is critical. It must occur at a time when the concrete has lost its plasticity and achieved a certain rigidity and strength. If it occurs before this time, a plastic deformation, rather than an expansion, of the material will result. On the other hand, the concrete matrix must not be too stiff to accommodate some limited expansion, as otherwise the expansion would lead to cracking. When the concrete loses this flexibility, the expansive reactions also must cease. [1, 5]

The formation of ettringite is the process employed most commonly to produce expansion in expansive cements. For reasons discussed above, it is important to control this process so that the bulk of ettringite is formed after the setting of the concrete, and yet while the concrete matrix is still flexible enough to accommodate the expansion.

To produce ettringite in expansive cements, calcium sulfate is used as the source of sulfate ions, whereas different calcium aluminate phases may be used for supplying the aluminum ions. However, due to the differences in their reactivities, not all calcium aluminates perform equally to produce expansion without cracking. Calcium hydroxide, produced by the hydration of the calcium silicate phases of cement, or added to the system externally, also plays an important role by facilitating the topochemical* mechanism of the expansive reaction as well as by controlling (retarding) its kinetics.

From among the various aluminate phases, tetracalcium trialuminosulfate, C_4A_3S , appears to be the most suitable source of aluminum ions for the expansive reaction. Most of this phase hydrates in the desirable time range of several hours to several days, after mixing with water. Monocalcium aluminate, CA , hydrates rather rapidly initially in the presence of gypsum or lime, but a significant fraction of it reacts shortly after setting, that is, at a time which is suitable for the expansion process. This reaction ceases within a few days. Contrary to the reactions of these aluminates, tricalcium aluminate, C_3A , produces ettringite in very early stages of hydration, that is, at a time when this reaction does not cause an expansion. Later, although the reaction may continue for an extended period of time even after the concrete has lost its ability to

expand without cracking, it progresses at a very slow rate. Finally, the reaction of the $C_{12}A_7$ phase is much too rapid to be used in expansive cements.

The hydration reactions of CaO and MgO to produce $Ca(OH)_2$ and $Mg(OH)_2$, respectively, also are expansive in nature, and may be employed in expansive cements. However, it is rather difficult to control the kinetics of these reactions in such a manner that the expansion is not accompanied by a cracking. [13, 20]

It is a well known fact that expansion in sulfoaluminate - type expansive cements (types K, M and S) is associated with the formation of a calcium sulfoaluminate hydrate, ettringite. C_4A_3S in Type K, CA and $C_{12}A_7$ in Type M and C_3A in type S are the major phases responsible for the formation of ettringite.

Another type of expansion is in the form of free-lime expansion. This CaO-type expansive cement was introduced by Kawano, Hitotsuya and Mori. With adequate control of hydration rate, it shows superior property when compared to the sulfoaluminate-type expansive cements.

A review of literature on sulfoaluminate-type expansive cements, specifically Type K, shows that understandings of the morphology of ettringite formation and the mechanism of expansion remain controversial issues.

The numerous theories and models aimed at explaining the mechanism of formation of ettringite and expansion due to ettringite can be divided into two major hypotheses: the Crystal Growth Theory and the Swelling Theory.

According to the first hypothesis, expansion is caused by the growth of ettringite crystals which form on the surfaces of the expansive particles or in the solution. The growth of these crystals, commonly referred to as crystal growth, is responsible for the crystallization pressure and, hence, expansive force.

According to the second hypothesis, expansion is caused by the swelling of the ettringite particles that are of colloidal size. These particles are commonly referred to as gel. This gel has a large specific surface area and, similar to C-S-H gel, takes up water to produce overall expansion by swelling. The formation of this gel occurs by a through-solution mechanism due to reaction between the expansive particles and the surrounding solution. Moreover, if Ca(OH)_2 exists in the solution, then ettringite particles will be colloidal in size. But if Ca(OH)_2 is absent, then ettringite particles will be larger in size. It is only in the former case that expansion can occur. [46]

2.4.2 HYDRATION CHARACTERISTICS ASSOCIATED WITH EXPANSIVE ETTRINGITE FORMATION

The hydration chemistry of expansive cements is not yet fully understood. It is generally recognized, however, that formation of ettringite, $(3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{CaSO}_4\cdot 32\text{H}_2\text{O})$ is the source of the expansive force and is a factor common to most commercially available expansive cements.

2.4.2.1 PRINCIPAL CONDITIONS FOR ETTRINGITE FORMATION

As shown by the formula, four components, CaO, Al_2O_3 , SO_3 , and H_2O , constitute ettringite. The reaction involved in the formation of this phase, reduced to its simplest form, is: $6\text{CaO} + \text{Al}_2\text{O}_3 + 32\text{H}_2\text{O} \rightarrow 3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$.

The three components other than water may originate from a large variety of reactants. The materials must be either soluble or at least slightly soluble, or form soluble to slightly soluble hydration products. A second requirement is that the aqueous phase (the solution formed when the mixing water reacts initially with the materials) contain concentrations of CaO, SO_3 , and Al_2O_3 in sufficient amount for stabilization of ettringite. This second condition is provided in the expansive cements under consideration as long as SO_3 is available to the solution in amounts equal to, or exceeding, the solubility of ettringite. [42, 46, 49]

2.4.2.2 GENERAL SOURCES OF PRINCIPAL COMPONENTS

Lime, as $\text{Ca}(\text{OH})_2$, required for chemical combination originates by hydrolysis of alite ($3\text{CaO} \cdot \text{SiO}_2$), belite ($2\text{CaO} \cdot \text{SiO}_2$) and hydration of free CaO in both the expansive component and portland cement. Calcium sulfate, when present, supplies the CaO associated with the SO_3 . The Al_2O_3 is obtained from CA and C_{12}A_7 of the calcium aluminate cement, and C_3A and $\text{C}_4\text{A}_3\text{S}$ of the Type K cement. Still other Al_2O_3 -bearing materials (bauxite, burned kaolin, slag, etc.) have been used in experiments as source of

Al_2O_3 but are not considered in this report. The SO_3 in practice is supplied either as gypsum or anhydrite, or partially by $\text{C}_4\text{A}_3\text{S}$ when the latter is present. It follows on the basis of the primary chemical requirements that many combinations of the above materials would serve for formation of ettringite. The choice in practice is limited to combinations of available materials that are productive of the desired degree of expansion at the right time. [1, 2, 3]

2.4.2.3 PRIMARY FACTORS INVOLVED IN PROPORTIONING OF EXPANSIVE CONSTITUENTS

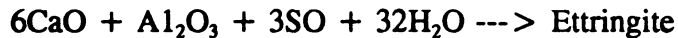
The proportioning of the expansive constituents in different cements is not based on the amounts represented by the theoretical ratio of C_6AS_3 . The important requirement to be fulfilled in the choice of proportioning the materials is that the CaO , SO_3 , and especially the Al_2O_3 , become available for ettringite formation at the right time. Ettringite starts to form during mixing and continues to form during subsequent water curing until the SO_3 or Al_2O_3 is exhausted. A major part of ettringite must form after attainment of a certain degree of strength, otherwise the expansive force will dissipate in deformation of a still plastic or semiplastic concrete and place no stress on the restraint provided. If, on the other hand, the ettringite continues to form rapidly for too long a period after the major part of strength has developed through cement hydration, disruptive expansion of the hardened concrete may occur.

Most of the expansive reaction of formation of ettringite must therefore cease before development of high strength through hydration has occurred. Experience has

shown that some expansive forces may continue to develop over the "ettringite formation period" without major deleterious effect on strength. This period includes the time of continuing hydration of the cement with substantial strength development. Continuing expansion may cause microcracking in the paste, but such microcracks are being continually sealed with new hydration products, provided sufficient moisture is available. Aroni proposed a multistage expansion theory which describes a number of cycles of expansion, internal restraint, local disruption, and revitalized expansion. [51, 54]

2.4.2.4 SO₃ REACTIONS OTHER THAN ETTRINGITE FORMATION

If the reaction:



represented the consumption of all Al₂O₃ and SO₃, and these having been present in the theoretical ratio in the hydrating cement, the problems of control would be relatively simple. Some of the SO₃, however, combines with the hydrating silicates. The distribution of SO₃ and Al₂O₃ between ettringite and the hydrated calcium silicate requires special care in proportioning. At the present time, control is based on securing expansion of desired amounts and at predetermined time by careful control of the proportions of the cement mixture established in laboratory tests. [2, 13]

2.4.2.5 PRODUCTS OF REACTION CAUSING EXPANSION

The mechanism of expansion of cement pastes containing C_4A_3S , CA, or a higher than usual C_3A content is often attributed to ettringite formation and also infrequently to the formation of calcium aluminate monosulfate. Hansen et al¹⁶ proposed that the ettringite crystals form directly on the surfaces of the C_3A grains without the latter entering the solution. In the normal process of hydration, the residual C_3A grains would be completely surrounded by hydration products and growth of the ettringite crystals formed in such sites would develop expansion stresses. Similar reaction mechanisms could apply to C_4A_3S and CA as well. The reactions involving C_4A_3S in ettringite formation have been discussed in detail by Mehta and Klein. Mehta concluded that ettringite, not monosulfate, was the expansive phase.

Unequivocal proof has not yet been established on the mechanism of formation of the expansive phases resulting in expansive forces. It is, however, probable that ettringite crystals when surrounded and restrained by reaction products grow in size and thereby are responsible for the development of stresses. [21, 35]

2.4.2.6 ALKALI-CARBONATE REACTION

Some reactive aggregates tend to produce expansive products as a results of reactions with cement alkalis. Alkali-carbonate reactions is one of the forms of alkali-carbonate reactions.

According to Tauget et al, the microstructure of the reactive carbonate rock is characterized by a distribution of rhombic crystals of dolomite in a matrix composed of clay and fine grained calcite (calcium carbonate); the clayey part exists as a network. The alkali solution penetrates through the channels of the clay network and reacts with the dolomite crystals. The expansion is caused by the alkali ions and water molecules migrating into the restricted space and by the growing and rearranging of the products from dedolomitization. [23, 37, 38, 40, 55, 56]

2.4.2.7 HEAT OF HYDRATION

Available information on the nature of hydrating compounds and ASTM C-186, indicates that all types of expansive cement may be expected to have significantly higher heats of hydration at early ages, and slightly higher heats of hydration at later ages compared to portland cements.

2.4.3 THEORIES OF EXPANSION

2.4.3.1 CRYSTAL GROWTH THEORY

Since, in chemically prestressed expanding concrete, restraint is a vital ingredient, we are dealing with a phenomenon of crystal growth under pressure. Basic physico-chemical knowledge of this phenomenon could help us understand, control and predict related behavior of expanding concrete.

Becker and Day appear to have been the first to demonstrate conclusively that a crystal can grow and lift an object when loaded with a weight. According to the Riecke principle, a crystal under linear pressure has a greater solubility or lower melting point than an unstressed one. According to Correns, the pressure which a growing crystal can exercise depends upon the supersaturation of the surrounding medium. Thus we would expect the expansion under stress, in expanding concrete, to depend on the state of supersaturation. Also, restraint and pressure will cause not only visco-elastic deformations but will interact with and influence the very cause of expansion, the crystal growth.

Hansen discussed crystal growth as a source of expansion in portland-cement concrete and described the work of Taber, who undertook an extensive early investigation of the behavior of crystals under different environments. Some of Taber's conclusions were: [1, 2, 3]

- (a) A crystal will grow in a direction in which external forces oppose growth if the surface on which the forces are acting is in contact with a solution that is supersaturated with respect to it. As the solubility increases with pressure, for any increase in the forces opposing growth a corresponding increase in the concentration of the solution is necessary for the growth to continue.
- (b) If a crystal is subjected to a greater pressure in one direction than in others, the surfaces that are under the lesser pressure will tend to limit the

concentration of the solution and prevent it from becoming supersaturated with respect to the surfaces that are rendered more soluble because of the greater pressure. In order that growth may also take place on the surface subjected to the greater pressure, some means must be provided for maintaining the supersaturation of the solution in contact with them.

- (c) In porous materials the solutions in subcapillary pores are not readily nucleated. Therefore, the solution in such pores will be supersaturated with respect to crystals growing in larger pores. Hence, the crystals in the larger pores can receive material from the smaller pores, can grow at the interface between the liquid and the crystal, and can exert pressure during this growth.

Richards and Helmuth (1974) have applied this theory to study the mechanism of expansion in expansive cements. In their studies they assumed the expanding particles C_3A and C_4A_3S to be spherical and of average composition. These particles are surrounded by a solution of uniform thickness containing dissolved sulfates, calcium hydroxides, and water.

Hydration is assumed to proceed at equal fraction rate according to the equation bellow:



Moreover, they assumed that as soon as reaction starts, immediately the surfaces of the expanding particles become covered by a dense coating product--ettringite.

Further hydration, which occurs topochemically,* increases the thickness of this coat or layer. When the layer exceeds the surrounding solution thickness, it will push other particles apart, resulting in overall expansion. This is the crystal growth theory. Expansion will continue until either the expanding particles or the dissolved sulfates will deplete.

Following these assumptions, the Micromechanical Model was developed to calculate geometrically the final magnitude of expansion. This magnitude depends on the final thickness of the ettringite coating and on the number of expansive sites. These two factors are influenced by:

- (1) chemical composition
- (2) water-cement ratio
- (3) particle-size distribution of the expanding particles (PSD)

This model predicted that there is an optimum PSD resulting in the highest magnitude of expansion. The model also predicted that increasing the amount of sulfate (e.g., gypsum) at the expense of Portland cement clinker while keeping the amount of expansive clinker constant increases the magnitude of expansion. [2, 3]

One disadvantage of the model is that it does not have a time factor. For example, it does not say anything about the kinetics of ettringite formation and how they would affect the time of expansion. Schwiete et al (1966) also stated that ettringite crystals are formed on the surfaces of the C_3A particles without the C_3A being dissolved beforehand. They showed that these crystals form in the first few seconds on the

surfaces of the C_3A particles. Since the volume of the ettringite formed is greater than that of the portion of the C_3A used, the ettringite layer will not remain on the surface of the C_3A particle. It will eventually peel off. This is the crystallization pressure associated with the formation of ettringite.

Bentur and Ish-Shalom (1974. I., II.) also applied the crystal growth theory to study the expansion mechanism of pure C_4A_3S particles. Similar to Richards and Helmuth, they assumed the ettringite to form topochemically* on the surfaces of C_4A_3S particles which they also took to be spherical. Unlike the micromechanical model, these particles were considered to be of equal diameters. In addition, they assumed the ettringite layer to be made of fine intermeshed crystallites enclosing pores between them. This was again different from the micromechanical model which assumed the ettringite layer to be dense and without any pores. [1, 5]

Bentur and Ish-Shalom showed that expansion does not start instantaneously but at a critical degree of hydration, a_{cr} , when the first contact is formed between the expanding spheres. Further hydration produces more ettringite which will push against other particles, thus producing expansion. Probably the time at which the critical hydration occurs corresponds to the time it takes for the ettringite crystals, according to the micromechanical model, to surpass the solution of thickness.

Kalousek and Benton (1970) in their study of the mechanism of sea water attack on cement pastes have attributed expansion to the ettringite crystals that grow in confined sites and which exert expansive forces against the surrounding walls. Unlike the

micromechanical model, they stated that there is no correlation existing between the amounts of ettringite precipitated and expansion. It is only a part of the increase of ettringite which is effective in causing expansion, the remainder depositing in voids. Kalousek and Benton divided the ettringite crystals into two categories-the iron-ettringite and the aluminum-ettringite. The iron-ettringite, which they called the F-rich ettringite, is a product of the ferrite phase. These crystals did not grow in size, and if they did, the amount was small. Without crystal growth, expansive forces were not developed and the paste did not expand. The aluminum-ettringite, which they called the F-poor ettringite, is a product of the aluminate phase. These crystals grew in size by deposition of the participating ions from the surrounding supersaturated solution. Their growth, when confined by hydration products, was the source of energy causing expansion.

Budnikov and Dravchenko (1968), in their studies on expansive cements, have also attributed increase in volume of hardened cement paste to the formation of crystals. They also indicate that expansion occurs when the growth of crystals, which occurs at a "linear period," moves the obstacles apart. [14]

Expansion depends, in their opinion, on the number of crystallization nuclei in a unit of volume and also on the growth speed of the crystals. If the number of crystallization nuclei is large per unit volume, despite a great number of these expansive sites, expansion will be small. However, if the number is small per unit volume, expansion will be large, despite a lesser number of these expansive sites. In addition, expansion will also be large if the growth of crystals is more prolonged.

There exists a similarity between Budnikov-Kravchenko's and Richards-Helmuth's ideas. The number of crystallization nuclei (expansive sites) probably corresponds to the number of expanding particles in the micromechanical model. For example, a large number of nuclei relates to a large number of expanding particles per unit volume (fine cements). The micromechanical model also predicts that for this case, expansion will be small.

Similar to Aroni et al, Budnikov and Kravchenko (1968) stress the importance of the kinetics of ettringite formation. For example, if the ettringite crystals form too quickly, as in the case of fine expansive cements, then the magnitude of expansion is not very large. This is because the cement has not hardened sufficiently to provide for expansion. But if the ettringite crystals form later, then expansion will be relatively larger, provided that the cement has gained considerable hardness. [6, 13]

The factors which influence expansion, as they report, are the composition of the expansive cement, the fineness and components of cement, the temperature of the environment, the admixtures modifying the structures of the newly-formed hydrates, etc. However, they state that the nuclei form both in the solution and on the surfaces of the cement grains since the concentration of the solution on the surface of the particle is largest. So, to interpret their saying, they probably believe in both topochemical* and through-solution modes of formation of the ettringite crystals, but do not say in particular which mode of formation produces expansion.

The mode of formation of ettringite was studied extensively by Nakamura et al (1968). In their studies on the expansibility of pure mixtures of C_4A_3S - $Ca(OH)_2$ - $CaSO_4$ system, they reported that the condition of formation of ettringite seems to be dependent upon the concentration of SO_4^{2-} ions around the C_4A_3S particles, regardless of the presence or absence of $Ca(OH)_2$. They suggested that two types of stoichiometric reactions occur: [13, 14, 20]



Both reactions produce ettringite, but owing to the presence of $Ca(OH)_2$, the first reaction occurs earlier.

In the presence of $Ca(OH)_2$, when Ca^{2+} ions were relatively in abundance, ettringite, as they report, crystallized as fine grains coating the surface of C_4A_3S particles. In agreement with Richards-Helmuth and Bentur-Ish-Shalom, this coat developed during the process of hydration and began to act on the adjacent particles to produce expansion. The magnitude of expansion increased with the increasing amount of $Ca(OH)_2$. Similar to Kalousek and Benton (1970), they reported that expansion was not necessarily dependent upon the amount of ettringite that was formed. In the absence of $Ca(OH)_2$ (the second reaction) no expansion was produced even though ettringite was formed. In this reaction, when Ca^{2+} ions were relatively scarce, ettringite formed as large crystals in the solution.

Nakamura et al also observed that in pure mixtures of C_4A_3S - $Ca(OH)_2$ - $CaSO_2$, expansion also was attained even when $Ca(OH)_2$ was absent from the pure mixture. This was probably because $Ca(OH)_2$ was supplied by the C_3S and C_2S particles existing in the Portland cement. Hence, $Ca(OH)_2$ is needed to produce expansion.

Finally, Nakamura et al (1968) reported that expansion begins only when the cement obtains considerable hardness to accommodate expansion. This is in agreement with Aroni et al and Budnikov and Kravchenko.

Along this line, the effects of lime concentration on the mode of formation of ettringite and on the resulting expansion was studied by Okushima et al (1968). In their studies, they carried out physicochemical investigations by means of optics, electron microscopy, x-ray diffraction, and pore-size distribution to study the mechanism of hydration, hardening, expansion, and shrinkage of expansive cements. Similar to the previous investigators, they associated expansion with formation of ettringite.

Okushima et al (1968) also observed very little expansion when the relative concentration of lime (Ca^{2+}) was low. This observation was similar to the findings of Nakamura et al (1968). In addition, they report that ettringite can form either on the surface of C_3A particles or in the solution. In the case of C_4A_3S alone, $C_3A.CS.H_{12}$ (monosulfate) and $Al(OH)_3$ are produced and this causes ettringite to crystallize out in the solution. But in the presence of lime, C_4A_3S hydrates quickly and becomes covered with a fine coating of ettringite crystals without changing the outer shape of the grain. The coating then begins to expand. This once more is a property of topochemical* reaction and crystal growth.

In their XRD analysis, they show that ettringite is produced as long as gypsum is available. However, at a certain point of hydration, monosulfate comes into existence just when the concentration of ettringite starts to drop. This probably indicates that monosulfates form at the expense of ettringite. Furthermore, expansion, as reported, seemed to be mainly accompanied by an increase in the volume of pores.

Okushima et al attribute the irreversible expansion of expansive cements to physicochemical factors as follows:

"In the hydration of CSA-cement (calcium-sulfate-aluminate cement), CaO and CaSO_4 are considered the outer reactants because their rate of dissolution is larger than that of aluminates such as C_3A and $\text{C}_4\text{A}_3\text{S}$, and the surface of aluminate grain is covered with a less permeable layer of coating. The outer reactant permeates through this layer to the interface of the inner reactant. The inner product is rapidly formed by interaction between the diffused materials and the inner reactant, which also has a large rate of dissolution. The accumulation of hydrates begins inside of the grain because of the low solubility of the aluminate sulfate hydrates formed."

This is a property of topochemical* reaction. Note that the "less impermeable layer of coating," probably ettringite, permits CaO and CaSO_4 to diffuse and reach the surfaces of the C_3A and $\text{C}_4\text{A}_3\text{S}$ particles. In other words, ettringite does not act as an impermeable membrane. Along this line, Okushima et al state that the driving force of expansion is due to the pressure created by growth of crystals (probably ettringite) of products under compressive stresses. These crystals do not form a semi-permeable membrane. For this reason, the solution will not produce osmotic pressure which can be a source of expansion.

Finally, in discussing Jorgensen's (1961) work, they conclude that the degree of expansion depends on the size of the crystals produced. For example, expansion is bigger when small crystals are formed. [1, 2, 3, 21]

2.4.3.2 THE SWELLING THEORY

The second hypothesis expansion produced by swelling or water-adsorption characteristic of ettringite--was proposed by P.K. Mehta.

Additionally, there are researchers who disagree with the first hypothesis. Levi Brown (1970), in discussing Kalousek and Benton's paper (1970), challenges the hypothesis that crystal growth creates pressure and believes that it is non-existent in the sense that Kalousek and Benton describe.

Brown states that crystal growth from solution is not a source of significant energy release and the thermodynamic property associated with pressure of crystal

growth is only the heat of solution. He, therefore, rejects the theory that growth of crystals from solution can cause physical damage. Brown further states that crystals can grow in solutions as long as the necessary ingredients are available, but when the crystals become confined, there will be no more "feeding solution" and growth in that direction will stop. To support his argument, he continues:

"If there was such a mechanism as Pressure of Crystal Growth, there would be no such thing as Portland cement concrete. A significantly large proportion of the normal hydration product is coarsely crystalline (e.g. calcium hydroxide). The mix water almost instantly becomes saturated with calcium hydroxide. Though saturated, continuing hydrolysis of the lime silicates pushes lime into the solution, which likewise forces precipitation and crystal growth out of the solution.

If ever there was a situation of crystal growth from solution in a confined space, that would be it. Yet, in Portland cement systems such growth has never been seen as damaging to those confining walls. In fact, autogenous volume change in lime silicate hydrolysis is always shrinkage."

On this basis, the second school of thought -- expansion caused by swelling of ettringite crystals -- was proposed by P.K. Mehta. On the basis of experimental evidence, Mehta (1973) introduced a hypothesis on the mechanism of expansion caused by formation of ettringite crystals. First, he suggested that the mode of formation of ettringite follows a through-solution** mechanism. Second, in the presence of saturated $\text{Ca}(\text{OH})_2$, the rate of hydration of the aluminates decreases significantly (see Steinour, 1958, and Kerdegari, 1978). This causes the ettringite crystals to be colloidal in size. [20, 21]

Since colloidal particles have a large specific surface area, water molecules will be adsorbed on the surfaces of ettringite crystals. This will generate strong swelling pressure of the colloidal ettringite crystals and leads to an overall expansion of the system without any change in the crystal lattice of ettringite (Mehta, 1973). In the absence of lime, the aluminate phase will hydrate rapidly. This will form large, long-lathed ettringite crystals. These crystals will not produce expansion because they have a low specific surface area, attracting lesser amounts of water molecules to their surface. In this experiments, Mehta studied the hydration of $\text{C}_4\text{A}_3\text{S-CSH}_2$ systems with and without quick lime. The examination of the microstructure led him to the same conclusions as those drawn by Okushima et al (1968) concerning the ettringite crystal sizes. Scanning electron micrographs indicated that in the presence of lime, ettringite crystals were about one micron in length and 1/4 micron in width. In the absence of lime, they were about six to eight microns in length and 1/2 to one micron in width.

Mehta also obtained similar results when CA was used instead of C_4A_3S .

In another experiment, Mehta and Hu (1978) observed that an increase in the amount of adsorbed water corresponds to an increase in volumetric expansion.

2.4.3.3 SUMMARY OF THE HYPOTHESES

There are two major hypothesis concerning the mechanism of expansion in expansive cements. According to the first theory, expansion occurs because of crystallization pressure exerted by the growth of the ettringite crystals coating the surfaces of the expanding particles. This theory tends to stress the importance of the sizes of expanding particles in controlling the magnitude and rate of expansion. Expansion, furthermore, is controlled by the number of crystallization nuclei and the speed of crystal growth.

According to the second theory, expansion occurs because of swelling of colloidal ettringite crystals which form in a through-solution mechanism. This theory unlike the first one, does not consider the size of the expanding particles to be important. Instead, it stresses that it is the existence of lime which affects the size of ettringite crystals and, therefore, the rate and magnitude of expansion.

Hence, both theories agree that the size of the ettringite crystals affects the expansion. It is further shown that the particle size distribution and fineness of the expanding particles control the number of nuclei and crystal sizes and, therefore, the expansion behavior of (says Type K) expansive cements.

It seems that both theories agree that lime influences expansion. In numerous investigations the following have been demonstrated by the two theories:

- (1) In the presence of lime, sulfates, water, and expanding particles, ettringite is formed as small crystals and their formation contributes to expansion.
- (2) In the absence of lime, the sulfates and water hydrate with expanding particles to give large ettringite crystals which do not contribute to expansion.

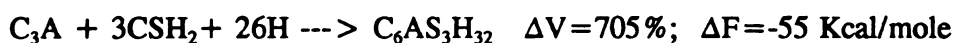
However, a major disagreement exists between the two theories. According to the first theory, hydration is more favorable and quicker in the presence of lime and the mode of formation of ettringite is topochemical*. Type K cements should, therefore, follow this mechanism since lime is never absent during their hydration. Only in the absence of lime would the ettringite crystals grow in locations away from the surface of the expanding particles, for example, through-solution mechanism.

According to the second theory, ettringite always forms in a through-solution mechanism, regardless of the presence or absence of lime. Furthermore, unlike the first theory, the rate of hydration of the expanding particles is believed to be smaller in the presence of lime.

2.4.3.4 SOME RESEARCHERS DISAGREE WITH BROWN'S THEORY (1970)

It would be appropriate to include Kalousek and Benton's rebuttal to Levi Brown in reference to his rejection of their hypothesis concerning pressure created by growth of crystals. Kalousek and Benton, in their paper, attribute expansive stress to the conversion of free energy of reaction to work. They state that in any chemical equilibria, thermodynamics is not concerned with the path of the reactions going from initial to final product. A reaction, in their opinion, may either occur topochemically,* in a through-solution** manner, a solid-state diffusion mechanism, or a combination of two or three of the processes. However, the free energy changes, in going from initial to final products for each reaction, are independent of the path of the reaction. [2, 3]

They considered the free energies (ΔF) and potential volume changes (ΔV) for the hydrations of CaO, MgO, and $C_3A + 3CSH_2$ according to the following stoichiometry:



They stated that these products can cause expansion simply because the ΔV values, which are the ratios of the volume of the hydrate less the volume of the anhydrous phase to the volume of the anhydrous phase, being positive, which means an increase in volume, result in an expansive stress against the surrounding walls of the

hydration products. The source of the expansive stress is the conversion of free energy of the reaction to work. In the case of the crystallization of ettringite, where Ca^{2+} , SO_4^{2-} , and OH diffuse to the original sites of C_3A , Kalousek and Benton believe that the ettringite, because it occupies volume larger than that which was occupied by C_3A , develops expansive stresses against the surrounding walls of the cement gel and, therefore, produces expansion by crystallization pressure.

The $\text{Ca}(\text{OH})_2$ produced by the hydrolysis of CS and B- C_2S , as they believe, is different from that produced by the hydration of lime because crystals of the former grow only in either the voids of the paste in which they can grow with no restraint or in nuclei surrounded by the cement gel, whereas crystals of the later, because they are pervious to water and because they closely surround the CaO particles, can exert expansive stresses similar in nature to the ettringite crystals. [2]

Finally, they state that the free energy of the reactions, which provides an approximate estimate of the amount of work expected, far exceeds the work of expansive disruption even for as little as 0.02 mole of ettringite in 1" x 1" x 12" concrete. Therefore, growth of these crystals in confined sites, regardless of the mechanism of formation, will produce expansion.

2.4.3.5 THEORIES OF "PROTECTIVE" AND "PARTIAL PROTECTIVE" COATINGS

This last section on the theories of expansion discusses the theories of "protective" and "partial protective" coatings which are discussed in detail by Kerdagari (1978). These theories are associated with the hydration of conventional cement, which involves ettringite formation without any significant expansion tendencies.

When water is added to cement, a complex reaction takes place. The result is a paste which remains in plastic state for a certain period of time called the dormant period. This is followed by a stiffening period when the paste gradually loses its plasticity. The cement then becomes brittle even without an increase in strength. After the setting of cement, a hardening process takes place when the cement will gradually gain strength (Popovics, 1979).

The setting period, which follows the dormant period, has two stages--initial setting and final setting. The initial setting corresponds to the beginning of stiffening while final setting corresponds to the disappearance of plasticity. The setting process should not start too early because the freshly mixed concrete must remain in a plastic state in order to be transported, placed, compacted, and finished. Also, if the setting time is too long, it is undesirable because this will cause a useless delay in strength development after finishing (Popovics, 1979).

When Portland cement and water are first brought in contact, a rapid reaction takes place. The velocity of this reaction was of interest to Lerch (1946). (Lerch's work

is discussed in detail by Kerdegari, 1978.) Lerch studied the rate of heat liberation of cement versus time. This is a measure of the speed of reaction. For example, the higher the rate, the more violent the reaction. In his studies he showed that in the absence of sulfates, C_3A reacts violently with the water (to produce hydrated calcium aluminates). Therefore, the time of initial setting will be very short, usually less than one hour (Popovics, 1979). This undesirable phenomenon is called flash set. It involves the evolution of a large amount of heat and the set cannot be overcome by remixing the paste (Popovics, 1979). [1, 2, 3]

In the presence of sulfates, Lerch found that the rate of heat liberation of C_3A is significantly lowered. Instead of the violent formation of hydrated calcium aluminates, ettringite will form with less violence. This increases the time of initial set and prevents flash set.

The mechanism by which sulfates operate can occur in one of two ways:

- (1) Ettringite forming uniformly on the surfaces of the expanding particles interferes with the ease of access of the reactants and, thus, offers impedance or retardation to further reaction. This is the idea of "protective" or "partial protective" coatings.
- (2) The nature of formation of ettringite crystals, regardless of where or how they form, is not violent. During this calm period, ettringite will continue to

form until the sulfates are consumed by the C_3A particles.

Then, when the solution no longer contains sulfate, the rate once again increases sharply as hydrated calcium aluminates begin to be formed.

It is difficult to see how expansion can be produced by ettringite crystals if the theories of "protective" and "partial protective" coatings are true. If the ettringite crystals stop further hydration of the expanding particles, no more ettringite will be produced to cause expansion.

Furthermore, Schwiete et al (1966) described the work of Stein (1963), (Figure 2.1), who also studied the rate of release of heat (cal/sec g C_3A) versus time (hours) for three grams of C_3A , 0.4 grams gypsum, and two cc 0.0196M $Ca(OH)_2$ solution.

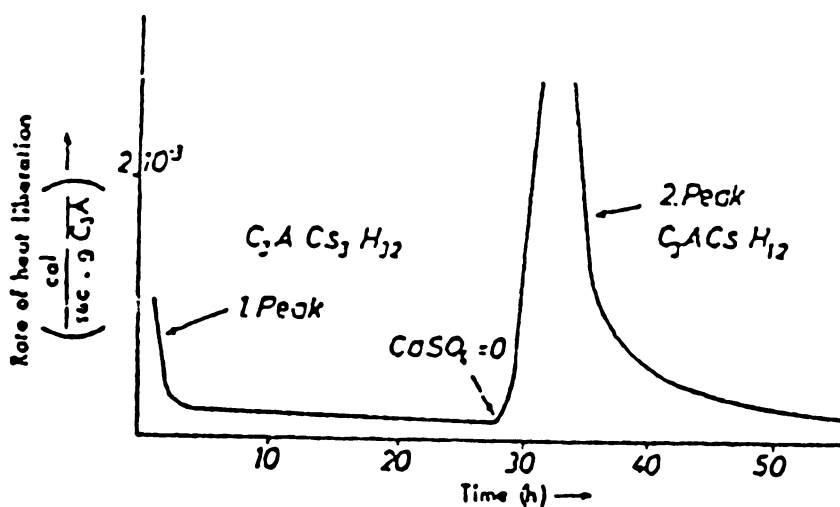


Figure 2.1. Isothermal Heat Liberation of $C_3A-CASO_4 \cdot 2H_2O-Ca(OH)_2-H_2O$ (Schhwiete et al, 1966).

Their findings are as stated below:

"After the first large peak, the rate of release of heat decreases continuously until the gypsum has reacted completely, then it becomes very large again and decreases in the further course of reaction. Ettringite is formed until the added gypsum is completely used up. After the second large peak in the rate of release of heat, Stein radiographically observed tricalcium aluminate monosulfate hydrate. Stein concludes that, in the period in which the gypsum is effective in retarding the hydration of C_3A , ettringite is formed on the surface of the C_3A grain..."

It has, however, been suggested that if the ettringite is forming a protective coating on the surface of C_3A , then it must not allow such a sudden or rapid increase in the rate of heat liberation when $CaSO_4$ concentration becomes zero. This occurs around 28 hours as shown in Figure 2.1. If this coating prevents the water and sulfate from reaching the surface of C_3A , then it should also prevent the water only from reaching the surface once sulfate is exhausted. Hence, "protective" and "partial protective" coating theories are questionable. [1, 2, 3]

* (*"A topochemical reaction," is a reaction between a solid particle and a surrounding solution in which the hydration product is formed on the surface of the particle*).

** (*"A through-solution mechanism," is a process of reaction between a solid particle and a surrounding solution in which ions of the solid particle diffuse away from the surface into the solution, where they nucleate particles of the product*).

Hence, topochemical* reaction of the expanding particles with the surrounding solutions will be associated only with the growth of ettringite crystals which do coat the expanding particles but do not prevent the unhydrated surface from further hydration. This is in agreement with Okushima et al (1968).

2.5 MIX PROPORTIONING AND MANUFACTURING

2.5.1 INTRODUCTION

The maximum expansion that can develop in Type-K expansive cement paste, mortar, and concrete is a function of several parameters such as composition and relative proportions of expansive cement ingredients, type and duration of curing, and the degree and direction of restraint.

The ability to predict the magnitude of expansion is of importance when planning the commercial applications of expansive cement. Such predictions, if successful, can facilitate optimization of mixture proportioning based on economic, engineering, and performance considerations. Expansion in Type-K expansive cement mixtures develops as a result of physicochemical processes occurring during hydration, setting, and hardening. Expansion subsides when the expansive components of the mixture deplete. By knowing the quantitative relationship between composition and relative proportions of the expansive cement ingredients on one side, and the attainable amount of expansion on the other, one can tailor the mixture proportion to achieve any practical level of expansion. This can be especially important in systems that benefit from expansion

forces to achieve the so-called chemical prestressing effect.

As with Portland cement concrete, shrinkage-compensating cement concrete cannot be expected to properly perform its design function without proper mixture proportions. Correct mixture proportioning is necessary to insure (1) practicality of construction or field placability, and (2) adequate expansion, strength, and hardened material properties at minimal cost. The effects of mixture proportions on expansion, drying shrinkage, internal thermal stresses for massive placements, rate of strength gain, and other properties not necessarily indicated by compressive strength should be taken into account in selecting mixture proportions.

In general, concrete mixture proportions which work well with normal Portland cements will also produce good expansive concrete of similar quality, although a minor increase in mixture water may be necessary and an increase in cement content for lean mixtures may be needed to obtain the full expansive movement desired. [40, 48]

2.5.2 CONCRETE PROPORTIONS

Aggregates: Selection of fine and coarse aggregate with regard to quality and proportions for use in shrinkage compensating concrete should follow accepted procedures used with Portland cement concrete. Fine and coarse aggregates from a known source which have performed satisfactorily in concrete may be used in the same proportions previously established.

Cement content: As with conventional concrete, the selection of an appropriate shrinkage-compensating cement content to meet specified concrete strength requirements should be based on test of concrete mixtures containing the materials to be used in the construction. The required strength may then be interpolated from the combined use of a water-cement ratio versus compressive strength curve and a cement factor versus compressive strength curve.

Water requirement: The water requirement of some shrinkage-compensating concretes may be as much as 10 to 15 percent more than conventional Type I and Type II portland cement concretes. Increased water requirements can be attributed to variations in the hydration rates which are influenced by the chemical composition of the cement and such physical properties as cement fineness, concrete temperatures, and mixing procedure. The additional water combines with the expansive component at a very early age so the free water available at time of placement is approximately the same as for Portland cement concrete. Trial values of water cement ratio for achieving different strength levels are presented in Table 2.3. [9, 32]

Table 2.3 Trial Mixture guide for Shrinkage Compensating Cement Concrete [9]

Compressive Strength at 28 days psi, (Mpa)	Absolute Water-Cement Ratio, by Weight	
	Non-air-entrained concrete	Air-entrained Concrete
6000 (41.4)	0.42 - 0.45	-
5000 (34.5)	0.51 - 0.53	0.42 - 0.44
4000 (27.6)	0.60 - 0.63	0.50 - 0.53
3000 (20.7)	0.71 - 0.75	0.62 - 0.65

The evidence is that in general the lower the water-cement ratio of the mix, the greater the expansion of expanding concrete, but in some cases the relationship between water-cement ratio and expansion is not clearly defined. Water-cement ratios as low as 0.16 are reported to be used in Russia in pneumatically applied expanding mortars. A number of reasons have been suggested for the observed behavior:

- (1) Low water-cement ratio will increase the concentration of calcium ions which is conducive to trisulfate hydrate formation and consequent expansion.
- (2) Low water-cement ratio will result in low permeability which impedes entrance of water particularly after 24 to 48 hours. This will reduce expansion and, if this factor is operative, it is apparently not as strong as the factors leading to increased expansion.

- (3) High water-cement ratio will increase the internal water-filled volume. This will provide more room for internal expansion and will reduce the observed external expansion.
- (4) High water-cement ratio might be expected to increase the amount of trisulfate hydrate formed during storage in the molds, and consequently to decrease the amount formed during subsequent curing in water and therefore the expansion. [32]

Low water-cement ratio leads to high expansion and this will tend to decrease the strength. However, apparently, the decrease is more than compensated by the higher strength of the tobermorite gel. Thus strength of expanding concrete also increases with decrease of water-cement ratio.

Cement Content: Test results indicate that an increase in cement content increases the expansion and self-stress of uniaxially and biaxially restraint specimens. Increase in cement content has also been shown to increase both the compressive strength and modulus of rupture of specimens with uniaxial restraint.

Type of Aggregate: In conventional concrete, the effect of aggregate is well known. In expanding concrete, two important additional effects are present:

- (1) The stiffness, size, shape and surface texture of the aggregate influence the internal restraint against expansion.
- (2) The porosity and water retention capacity of the aggregate (such as lightweight expand shale aggregate) could significantly affect the internal curing and therefore the expansion and shrinkage characteristics.

The investigation of the effect of aggregate type on the properties of expanding concrete was the principal aim of Aroni's tests. He used three types of aggregates: lightweight expanded shale (maximum size 5/8 in.), crushed granite, and river gravel (both with maximum size 3/4 in.). The experimental results demonstrated the advantageous use of high porosity aggregate. The internal distribution of water caused the early-age expansion to be a higher percentage of the eventual total expansion than with regular types of aggregate. This, together with a probable reduction of moisture and expansion gradients in the specimens and better internal curing, increased the compressive strength of the material. Also, better behavior on drying was observed with the internal water reservoir causing continued expansion and compensating for more of the shrinkage. [52, 53]

2.5.3 EFFECTS OF ADMIXTURE ON EXPANSIVE CEMENT

The effects of admixtures on expansion have been studied to a limited extent. In one study admixtures reduced the amount of expansion while in another admixtures had little or no effect. A discussion of the field experience with admixtures is presented below.

Many types of commercial admixtures have been included in various concrete mixes. The majority of these admixtures have been of the water-reducing retarder type. Lignin and sodium gluconate-based admixtures have generally been used in normal recommended dosages. During warm weather, relatively large dosages have been added

to the mix to delay initial concrete setting times, and use in winter has not created any difficulties with concrete.

Where freezing and thawing is considered a durability problem, all exposed shrinkage-compensating concrete installed to date have contained entrained air. Several structures and highway installations after 3 years of exposure showed no damage due to freezing and thawing except in a few isolated cases where laboratory testing confirmed low air content or where deicing salts were applied before the concrete had cured and aged for 1 month.

Water-reducing, retarding, and water-reducing retarding mixtures: Some ASTM C-494 Types A, B, and D admixtures are not compatible with certain shrinkage-compensating cements. The effects of high-range water reducers on shrinkage compensating concrete have not been thoroughly investigated. Chemical formulations of these newer admixtures are being revised, modified, and improved by some of the suppliers, so the behavior experienced with an outdated formulation may not be the same as for a current formulation from the same supplier. It is recommended that such admixtures as well as the Type A, B, and D admixtures be tested before acceptance, using the particular cement and other materials selected for the job. Special attention should be given to their effect on slump, restrained expansion, and drying shrinkage. The use of certain retarding, water-reducing, or water-reducing and retarding admixtures with some shrinkage-compensating cements has resulted in excessive drying shrinkage. The same effects may be experienced for certain high range water-reducing admixtures.

Generally, admixtures which are acceptable may be used in the normal dosage recommended for Type I or Type O Portland cement concretes under moderate temperature conditions. During hot weather, larger than normal dosages of acceptable ASTM C-494 retarding Types B and D have on occasion been used successfully to delay initial setting time of some shrinkage-compensating concretes.

Expanding concretes exhibit more rapid setting characteristics than do corresponding plain concrete mixes. Polivka and Bertero report that the initial and final sets of expanding concrete were reached about 25% earlier than for Portland cement concrete. They also present test results with three types of set-retarding admixtures, all of which increased the time of set beyond the plain concrete values. Apparently, the use of set-retarding admixtures had no significant influence on the concrete expansion characteristics. According to Klein, Karby and Polivka, a retarding water-reducer appeared to affect the expansive characteristics of the concrete only through the resulting reduction of water-cement ratio, which improved the strength and to some degree the expansion.

Air-entraining admixtures - Air entraining admixtures which comply with ASTM C 260 may be used for the same purpose with shrinkage-compensating concrete as with other types of Portland cement concretes. Generally, the same amount of a given air-entraining admixture will produce a comparable percentage of entrained air, all other conditions being equal.

An interesting observation was made on an experimental pavement section at Antelope Valley Freeway, in California. Half of the units in the test section had air-entrainment and half did not. The average observed distance between cracks for the air-entrained units was 69% of the distance for the non-air-entrained units. Apparently, under the conditions existing for this test pavement, the air-entrained concrete may have had substantially greater drying shrinkage.

Accelerators - Calcium chloride is generally not recommended for use in expansive cement concrete due to its effect on reducing expansion and increasing subsequent drying shrinkage. Some expansive cement producers, however, permit its use under certain conditions, but limit the content to 1 percent by weight of cement. It is recommended that the cement producer be consulted before calcium chloride or ASTM C-494 Types C and E admixtures are added in solution in accordance with accepted practice for Types I and II cement concretes.

2.5.4 CONSTRUCTION TECHNIQUES

Consistency: Good results can be obtained using slumps at time of placement within the maximum range specified by ACI 211.1 for the work involved when concrete temperatures do not exceed 75 °F (24 °C). At higher concrete temperatures, the maximum slumps at point of placement shown in Table 2.4 are recommended.

With ready-mixed concrete operations, the delivery time of concrete between the batch plant and placement may be as short as 20 min or as long as 1-1/2 hr.

Ettringite will begin to form during this period in some shrinkage-compensating concretes, resulting in a premature stiffening and slump loss of 2 to 3 in. (50-80 mm). It is therefore essential that sufficient slump within maximum allowable water limits be provided at the batch plant to insure that the specified or desired slump is obtained at the jobsite.

Table 2.4. Recommended Slumps at Placement [9]	
Type of Constructions	Slump (in.)
Reinforced foundation walls and footings	5
Plain footings, caissons, and substructure walls	4
Slabs, beams, reinforced walls	6
Building columns	6
Pavements	4
Heavy mass construction	4

It must be understood that if, because of ambient conditions at the time of placement, acceptable extended mixing times, etc, additional water is added to the mixture at the jobsite to increase the slump to the point that it approaches the maximum limit specified, close control must be kept over the amount of added mix water so that the total water in the mixture does not exceed the limit established by the mixture proportions. To do so could reduce strengths and offset the effectiveness of the expansive cement in controlling shrinkage cracks. The importance of taking this

slump loss into account in selecting proportions for shrinkage-compensating concretes cannot be over-emphasized. It becomes even more important during hot weather when concrete temperatures are relatively high and reactions are accelerated. While normal delivery time of ready-mixed Portland cement concrete under adverse hot weather conditions results in a significant slump loss, some shrinkage-compensating concretes develop an even greater slump loss under the same hot weather conditions. Slump loss which are successful for portland cement concrete are equally effective for shrinkage-compensating concrete. Because of the possibly greater slump loss, more liberal use and stricter enforcement of these controls is recommended when expansive cements are used. Recommended controls include using chilled mix water, using evaporated cooling on coarse aggregate stockpiles, reducing the speed of truck scheduling to an absolute minimum. When job locations require extended travel time, dry beached truck delivery with jobsite mixing is effective so long as the cement is charged on top of the aggregates without turning the drum so that moisture in the aggregate does not effectively contribute to the start of the hydration process and production of ettringite in the bulk quantity of cement. This batching procedure mandates the use of mixers in excellent condition.

Placement: The plastic characteristics of all three types of shrinkage-compensating concrete are sufficiently similar to concretes made with Type I or II Portland cement so that no special equipment or techniques are required for satisfactory placement. The recommendations set forth in ACI 304 shall be followed where applicable. Successful placements have been made by wheelbarrow, mixer truck, bucket,

conv

some

this

com

has a

in pe

shin

cons

expe

com

form

dy

wate

the e

quite

a bal

shou

stuc

conveyor, pump, and shotcrete. In general, shrinkage-compensating concrete has more cohesiveness or "fat" than Portland cement concrete and less tendency to segregate. For this reason, it is especially adaptable to pumping; a large percentage of shrinkage-compensating concrete has been placed by this method. Shrinkage-compensating concrete has also been used without difficulty in the manufacture of pipe in precast operations and in paving machines. [32]

Placing recommendations for Portland cement concrete are equally important for shrinkage-compensating concrete. However, the characteristics of shrinkage-compensating concrete require that good concreting practices be followed so as to insure adequate expansions and satisfactory results. If adequate expansions are not achieved, compensation for drying shrinkage is not available and the results may be seen in the form of cracking.

Where the plastic concrete will be in contact with an absorptive material such as dry soil or previously placed dry concrete, the base or subgrade shall be thoroughly wetted. Sprinkling lightly is not sufficient. Recommended practice is to soak the base the evening before placement and to sprinkle ahead of the placement as necessary. A quick check is to obtain a handful of subgrade and/or base material and squeeze it into a ball. The retention of its volume indicates adequate moisture content, and dampening should not be necessary. It is also good practice to wet the forms and reinforcement for structural concrete, particularly in hot weather.

In hot, dry, and windy placing conditions, all concretes tend to lose moisture unevenly and may develop plastic shrinkage cracks. Experience has shown that with shrinkage-compensating concrete, plastic shrinkage cracking is more prevalent because of water required for the early formation of ettringite. Finishing difficulties may be increased because of nonuniform moisture loss between top and bottom surfaces during the drying period, particularly when the concrete is placed directly over a vapor barrier. Where a vapor barrier is required, it is recommended that it be covered with a 1 to 3 in. layer of sand and properly dampened before placing shrinkage-compensating concrete. This practice results in more even moisture loss of the shrinkage-compensating concrete, less plastic shrinkage cracking, and protection of the vapor barrier during placement. Care shall be taken during placement of concrete to maintain a uniform layer of sand. In protected environments where a vapor barrier is required, the sand layer may be eliminated. When either a sand layer or a vapor barrier is used, consideration shall be given to larger than normal joint openings due to the reduction of the subgrade friction factor.

Should plastic shrinkage cracking occur, sheeting, monomolecular films, either sprayed or rolled on, as well as very fine fog sprays of water, have been used quite successfully in arresting this tendency. The purpose of fog spraying is to replace necessary surface water that has been lost due to an excessive rate of evaporation. Consequently, very fine sprays are recommended and heavy applications of water shall be avoided until concrete has taken its final set.

Care must be taken to maintain the reinforcement in its proper position during placement and consolidation to assure that it provides the required restraint. A quick check on the actual positioning of the steel can be made by inserting a wooden, plastic, or metal gage into the plastic concrete. The reinforcement will prevent further imbedment of the gage and depth of cover should be indicated and measured by the wet concrete on the gage. At the same time, the concrete shall be properly consolidated to insure good bond with the still.

Special precautions shall be taken to avoid placing delays at the jobsite when using ready-mixed concrete. A substantial increase in mixing time over that assumed when selecting mixture proportions increases the slump loss. Any water added at the jobsite, after slump loss, to maintain consistency not only decreases the strength but may also reduce the expansion to unacceptable levels. [52, 53]

Concrete temperature and time in the mixer (from intermingling cement and damp aggregate) are important factors because of their effect on expansion. It is recommended that the temperature of the shrinkage-compensating concrete at the time of placement not exceed 90 °F (32 °C) and the mixing time for shrinkage-compensating concrete at temperatures above 85 °F (30 °C) be limited to 1 hr. For shrinkage-compensating concrete below 85 °F (30 °C), the mixing time shall be a maximum of 1 1/2 hr. During cold weather concreting the methods recommended are those described in ACI 306R to provide adequate strength gain and expansion. Placement sequence is important in developing the proper strain on the internal reinforcement shrinkage-compensating concrete.

Although the successful combination of shrinkage compensating concrete and vacuum dewatering have been reported, it is generally not recommended unless proven by expansion and shrinkage of prisms to note net strain.

Finishing: The cohesiveness or "fat" inherent in expansive cements provide excellent finishing qualities. Its behavior is similar to air-entrained concrete but this usually presents no problems. Similarly, there is little or no bleeding even though a relatively high slump may be used. Due the to lack of bleed water, however, there is a tendency for finishers to start too soon. On the other hand, in warm weather, shrinkage-compensating concrete will typically set faster than Type I or Type II Portland cement concretes and finishing may start somewhat sooner than normal.

Curing: Shrinkage-compensating concrete, as with all Portland cement concretes, requires continuous curing at moderate temperatures for several days after final finishing operations to prevent early drying shrinkage and to develop strength, durability, and other desired properties. Any deficiencies in the method of curing may also reduce the amount of initial expansion which is needed to offset later drying shrinkage. When the effect of curing is considered, the properties of the mixture may be modified, i.e., increased cement factor may be used to compensate for the effects so as to insure adequate expansion. The usually-accepted methods of curing are satisfactory for shrinkage-compensating concrete; however, those that provide additional moisture to the concrete such as ponding, continuous sprinkling, and wet coverings are preferred to insure adequate water for ettringite formation and expansion. Other methods such as

moisture-proof covers and sprayed-on membranes have been successfully utilized, provided that coverage is complete so that it prevents loss of moisture from the entire concrete surface. Curing of shrinkage-compensating concrete shall be continued for a minimum of 7 days.

Curing of concrete flatwork shall commence immediately after final finishing. It may be necessary to fog spray or cover the surface of the concrete temporarily if other methods of curing are delayed, especially in hot, dry, or windy weather. If a liquid curing membrane is used, it is recommended that it conform to ASTM C-309 and be applied in two directions, at a coverage rate suggested by the manufacturer, immediately following the final finishing as it progresses. To accomplish this, power spray equipment capable of covering large areas more rapidly shall be used rather than small, portable spray tanks.

For architectural or structural concrete, the normally accepted practice of curing with the formwork in place is adequate for shrinkage-compensating concrete. All uncovered surfaces shall receive additional water; soaker hoses or water sprays shall be used to supplement the protection of the in-place form-work. If the forms must be removed prior to 7 days, one of the other accepted methods of curing shall then be employed for the balance of the curing period.

Shrinkage-compensating concrete shall be protected during the initial curing period against extreme temperatures during either cold or hot weather periods. The methods recommended are those described in ACI 305R and ACI 306R. There are

benefit

establish

importance

hydrate

humidity

condition

are different

is very much

consequence

increase

Some of

and the

2.6

2.6

comp

At the

consequence

expansion

beneficial effects of internal curing associated with the use of light aggregate.

The significance of curing through supply of water for hydration, is well established in Portland cement concretes. In expanding concrete it is of even greater importance. Here we are dealing with tobermorite and of the expansion-causing trisulfate hydrate, and these are affected differently by curing conditions, temperature and relative humidity. The trisulfate hydrate formation is the more rapid of the two and curing conditions can be used as a means of control in the "strength-expansion" relation. There are different rates of expansion at different temperatures. With a high temperature, there is very rapid early expansion would take place, while the concrete is still plastic, and consequently less total maximum expansion. The compressive strength probably increases with the curing temperature, being inversely proportional to late expansion. Some optimum temperature probably exists with respect to the magnitude of self-stress and the compressive strength. [52, 53]

2.6 DESIGN WITH SHRINKAGE COMPENSATING CONCRETE

2.6.1 INTRODUCTION

The design of reinforced concrete structural elements using shrinkage-compensating concrete shall conform to the requirements of applicable ACI standards. At the same time, adequate concrete expansion should be provided to compensate for subsequent drying shrinkage to minimize cracking. Since the final net result of expansion and shrinkage is essentially zero, no structural consideration need normally be

given to the stresses developed in the concrete during this process. Provision for dead and live loads required by building codes and specifications will result in at least the same structural integrity with shrinkage compensation concrete as with Portland cement concretes. However, provisions shall be made for initial expansive movements.

Restraint is vital for expanding concrete, first to limit the disruptive expansion and, secondly, to utilize the expansion and induce prestress. The amount of restraint is a crucial design parameter. It determines the stress induced and the effective strength of concrete. The type of restraint, uniaxial, biaxial or triaxial is also most important. Most of the investigations to date used uniaxial or biaxial restraint (mostly in slabs). A number of structural elements, pipes, beams with stirrups and columns, have been designed with triaxial restraint. [3, 4]

An investigation of uniaxial restraint has been reported by Bertero and Polivka (1966). The amount of restraining steel ranged from 0.22 to 6.47 percent and the conclusion was reached that there exists an optimum percentage of longitudinal restraint as far as the mechanical properties of the concrete are concerned. This optimum was estimated to be between 1 and 2 percent. Also, it was concluded that for successful use of expanding concrete in some structural elements, triaxial restraint is necessary.

This conclusion is now generally shared. Bertero (1967) demonstrated the damaging effects of lateral expansion and the large deterioration in strength that can occur with age. Anisotropy, non-homogeneity and detrimental lateral expansion increases with the size of specimen. Except for two dimensional elements, like slabs and

stalls.

elimination

expansion

difficult

restored

the in

the me

size of

expect

in large

would

of the

shells, where biaxial restraint might be satisfactory, triaxial restraint will be required to eliminate or minimize the above detrimental features.

There is probably an optimum triaxial restraint in the complex relation of expansion-strength-restraint and this should be investigated further. There is a certain difficulty, requiring care, in the determination of mechanical properties of triaxially restrained expanding concrete. To determine the compressive strength of the material, the triaxial restraint has to be removed before testing and the danger exists of damaging the material in the process.

Size Effect--Since curing normally proceeds from the surface of a specimen, the size of the specimen, which determines the ratio of surface area to volume, would be expected to influence the history of expansion and therefore the mechanical properties. In large specimens a moisture gradient will be set up and therefore an expansion gradient would exist, which may produce internal stresses detrimental to the mechanical properties of the concrete. [26, 29]

Bertero (1966) confirmed experimentally on uniaxially restrained specimens that:

- (1) Size affects the history but not the ultimate magnitude of longitudinal expansion, but it affects both the ultimate magnitude and history of lateral expansion;
- (2) An increase in size:
 - * decrease initial expansion and increases later lateral expansion;
 - * increases the time needed for expansion stabilization;

- * increases the anisotropy and non-homogeneity of the material.

Monfore also found a decrease, at an early age, of the expansion of mortar prisms with increase in size. Most of the detrimental effects mentioned above should be eliminated with triaxial restraint and internal curing provided by a porous aggregate.

Types of restraint: A resilient type of restraint, such as that provided by internal reinforcement, shall be provided to develop shrinkage compensation. Other types of restraint, such as adjacent structural elements and subgrade friction are largely indeterminate and may provide either too much or too little. Subgrade frictional coefficients in the range of 1 to 2 1/2 have been found satisfactory. High restraint will induce a high compressive stress in the concrete, but provide little shrinkage compensation. Wherever possible, the design shall, therefore, specify the reinforcement recommended. The design shall be performed using the procedures for predicting expansion.

Minimum reinforcement and location: Established engineering design practices for structural elements will normally provide a sufficient amount of steel. In some non-load-bearing members, slabs on grade and lightly-reinforced structural members, the usual amount of steel may be less than the minimum amount necessary for shrinkage-compensation concretes. For such designs, a minimum ratio of reinforcement area to gross concrete area of 0.0015 shall be used in each direction that shrinkage compensation is desired. This minimum is approximately that recommended by ACI 318 for temperature and shrinkage stresses. However, when procedures outlined for

predic

1221

requir

partic

correc

in cou

is not

reap

portion

third

ratio

the th

denom

know

and

the

the

the

the

predicting expansion are followed, a reinforcement ratio less than the above minimum may be used.

In structural members, the reinforcement location will be determined from design requirements. This may result in an over-concentration of reinforcement in one section - particularly in flat plate construction. Experience has shown that warping caused by concentrated reinforcement is not a problem in structural slabs because dead weight tends to counteract the warping deflection. However, when the location of the reinforcement is not determined by structural considerations, it shall be positioned to minimize warping. For example, in slabs on grade where most of the drying occurs in the top portion, the reinforcement should be placed in the upper half of the slab (preferably one-third of the depth from the top), while still allowing for adequate cover.

Prediction of expansions: When structural design considerations result in reinforcement ratio greater than the recommended minimum, or when it is desired to use less than the minimum reinforcement and location, the level of expansion in structural members should be estimated from Figure 2.2. This graph shows the relationship between member expansion, prism expansion, and percentage reinforcement when member and prism are made from the same concrete and are mixed and cured under identical conditions. The prism expansion test is defined in "Method of Test for Restrained Expansion of Shrinkage-Compensating Concrete" (ASTM C-878). The figure is based on published data by Russell et al, but modified to allow use of the ASTM C-878 test. Figure 2.2 indicates that for a given prism expansion, a higher amount of

reinforcement will reduce expansion.

Figure 2.2 may also be used to estimate the required expansion of control prisms to obtain a given expansion in a structural member. To provide satisfactory shrinkage compensation, the required expansion in the reinforced structural member is recommended to be greater than, or at least equal to, the anticipated shrinkage. [26, 31]

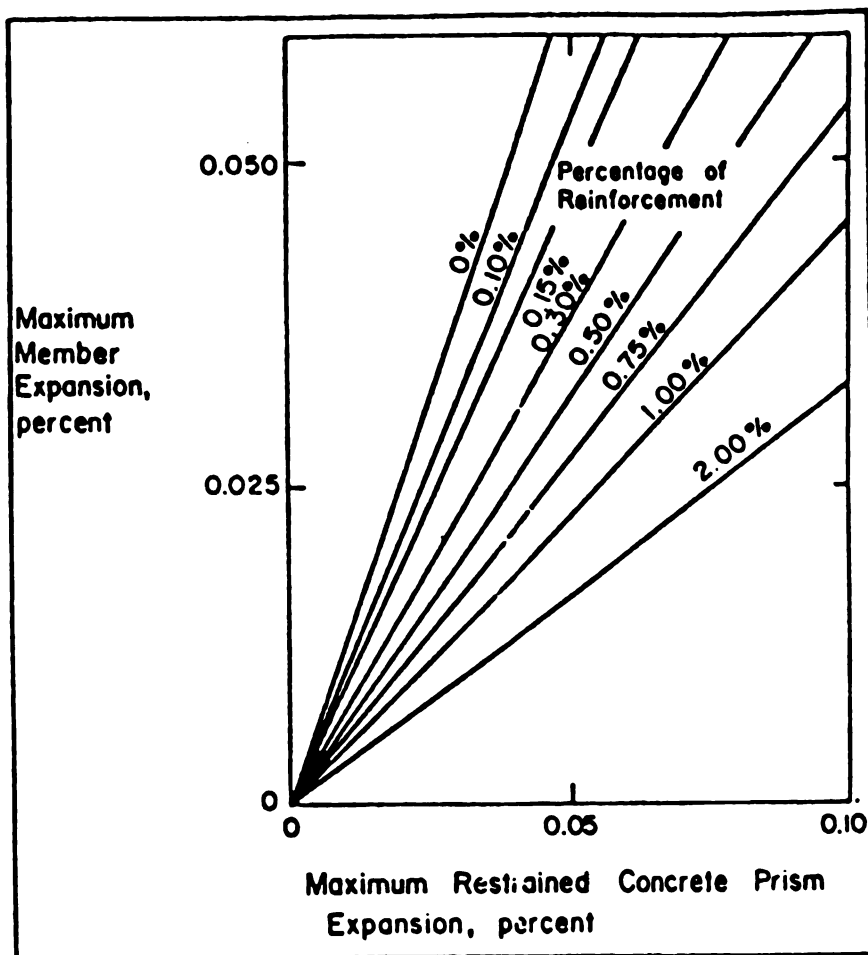


Figure 2.2. Prediction of member expansion from prism data [26]

Consider a slab in which the anticipated shrinkage is 0.025 percent. The required expansion for complete shrinkage compensation is also 0.025 percent. If the slab contains 0.5 percent reinforcement, then a restrained prism expansion of 0.04 percent is required for compensation. On the other hand, if the restrained prism expansion is 0.05 percent, then a reinforcement percentage up to 0.75 percent may be used and shrinkage compensation can still be achieved.

For a slab containing less than the minimum reinforcement specified in prediction of expansion, the same procedure may be used. Consider an anticipated shrinkage of 0.025 percent in a slab containing 0.1 percent reinforcement. Using Figure 2.2 the restrained prism expansion required to offset shrinkage is 0.025 percent. A slab expansion in excess of the shrinkage will increase the likelihood that complete shrinkage compensation will be obtained.

Slab expansion is reduced as the amount of reinforcement is increased. Shrinkage is also reduced, but to a lesser extent. Consequently, to achieve complete shrinkage compensation, the expansive potential should be higher for more heavily reinforced members. Increased expansion can be obtained with a higher cement content. However, expansion shall not be greater than 0.1 percent as measured using ASTM C-878.

When the amount of reinforcement in a member varies from area to area, an average expansion shall be overcompensated and the heavily reinforced areas undercompensated. In determining the anticipated shrinkage, the effects of member thickness and concrete materials on shrinkage should be considered. [9, 26, 31]

Reinforcing steel: Reinforcement should be either welded wire fabric or deformed bars meeting the requirements of ACI 318. Plain bar reinforcement shall not be used because adequate bond can not be developed. To ensure accurate positioning, deformed bars placed on chairs or tied to other rods, concrete supports, or portions of the structure should be used. Where wire fabric is used in lieu of deformed bars, it should be in flat sheets or mats rather than rolls. If rolled fabric is used, it should be unrolled on a hard flat surface to remove all curvature before being placed in final position. The fabric may be sandwiched between two layers of plastic concrete or supported on chairs or blocks. Hooking or pulling the fabric off the form or subgrade should not be permitted. Working the fabric in from the top may be permitted if it can be demonstrated that the reinforcement will be at the correct depth from the top surface throughout the slab. [43]

2.6.2 REINFORCED STRUCTURAL SLABS

Structural design: To provide proper safety factors, the design shall be based on the strength design provisions of ACI 318. This procedure will avoid consideration of the amount of stress in the reinforcement caused by the expansion of concrete; this is due to the fact that in strength analysis, the previous state of prestress does not influence the capacity of the section. In structural members, however, where it is anticipated that there will be high concrete expansion combined with loading at an early age, it is desirable to check that the net steel stresses caused by the expansion and loading conditions do not exceed permissible values. [26]

The magnitude of concrete stresses induced by tension in the reinforcement may be determined as follows:

Consider a reinforced concrete member which expands an amount e_c ,

If the areas of concrete and steel are A_c and A_s , respectively, then

Tensile force in steel = $e_c E_s A_s$

Compressive Force in Concrete = $e_c E_s A_s$

Stress in concrete = $e_c E_s A_s / A_c$

where:

E_s = modulus of elasticity of steel

Example: A concrete member containing 0.15 percent steel which expands 0.10 percent has an induced compress stress of 43.5 psi (300 kPa), whereas a member which only expands 0.02 percent but contains 2 percent steel has a compressive stress of 116 psi (800 kPa) (providing that the expansive potential of the concrete is not exceeded).

Hence, the induced compressive stress is a function of the amount of reinforcement as well as the expansion of the concrete. The induced compressive stress causes an elastic shortening of the concrete which for practical reinforcement ratios (up to 1/2%) is small compared to the errors in predicting the shrinkage. As the amount of reinforcement in a member increases, the expansive strains of a highly reinforced member are usually low, requiring only a small amount of shrinkage for the member to return to its original length and then to develop negative strains and concrete tension. Strain in concrete and steel is the most important element to consider when attempt is

made to counteract shrinkage. Concrete strain should stay positive since negative strains indicate the concrete is going into tension.

Deflection: The deflection analysis to satisfy load performance criteria shall be made in the same manner as for Portland cement concrete. Any residual compressive stress caused by expansion will improve the service load performance since a higher moment is required to produce first cracking. Residual compressive stress, however, shall not be taken into account when calculating deflections.

Crack spacing: In two independent investigations, in which shrinkage-compensating concrete was compared with portland cement concrete, it was observed that the number of cracks were less in the shrinkage-compensating concrete. This occurred for reinforced concrete specimens loaded in flexure and in direct tension. Since the comparisons in each case were made on specimens with the same length, the data can be interpreted to mean that fewer cracks will occur in shrinkage-compensating reinforced concrete members. This has been confirmed in field observations by Randall and Rosenlund.

Cracking moment: While the use of shrinkage-compensating concrete does not affect the flexural strength of reinforced concrete members, it does influence the moment at which flexural cracking occurs. Tests have shown that members made with shrinkage-compensating concrete crack at a 15 to 59 percent higher bending moment than corresponding members made with Portland cement concrete. The increase occurs because shrinkage-compensating concrete members have an induced concrete compressive

stress caused by the expansion. This is equivalent to a mild prestressing. Therefore, a higher tensile stress caused by the applied moment is needed to overcome the tensile strength of the concrete and the induced compressive stress. [15, 16, 17]

2.6.3 REINFORCED SLABS ON GRADE

Because shrinkage-compensating concrete expands shortly after setting, reinforced slabs on grade with shrinkage-compensating concrete will initially behave differently than Portland cement concrete slabs. Certain differentiating items are discussed below.

Tensile strains: Portland cement concrete is generally assumed to possess limited tensile strain capacity. Therefore, reinforcement in slabs on grade is primarily used to restrict crack widths caused by bending and drying shrinkage.

Research has shown that Portland cement concrete has approximately 200 millionths tensile strain capacity before cracking. Shrinkage-compensating concrete has a higher tensile strain capacity than comparable reinforced Portland cement concrete when the former is allowed to expand and elongate the reinforcement.

Comparative net strain values from drying with various percentages of restraining steel and different amounts of expansion have been reported for shrinkage-compensating concrete by Kesler, Russell, Spellman, and Pfeiffer. Values of strains due to expansion and subsequent shrinkage in a 50 percent relative humidity environment are given in Tables 2.5 and 2.7. Various levels of internal restraint are given for normal-weight and light-weight concrete. The data in Table 2.7 have been selected to make a

simple comparison between Type I and Type K cements. The data in Tables 2.6 and 2.7 indicate that there are two types of strain for shrinkage-compensating concretes: (a) positive expansive strain (length increase); and (b) negative shrinkage strain which may produce a net negative strain (net shortening) on the concrete. With Portland cement concrete, only a negative strain condition develops.

Warping: Because of the subgrade restraint and the internal top steel restraint against the expansion of the concrete, differential expansive strains between top and bottom can be expected. Upon drying shrinkage, expansive strains are relieved more quickly at the top drying surface than at the subgrade. Research has shown that the net effect indicates residual restrained expansive strains to be greater at the top surface than at the bottom, so reversed curling conditions develop. Warping stresses tend to be counterbalanced by the dead weight of the slab itself. [31, 32]

Table 2.5. Typical Tensile Strain Crack Capacity for Normal Weight Concrete.		
Type of cement	I	K
Force on concrete, lb-f	18,360	22,640
Area of concrete, in. ²	84	84
Stress, psi	219	270
Difference, psi	-	+51
Strain, millionths	730	900
Difference, millionths	-	+170

Metric equivalents: 1 lb = 4,448 N; 1 in.² = 645 mm.²; 1 psi = 6,894 kPa [9].

Table 2.6. Typical Comparative Residual Strains for Reinforced Normal Weight Concrete.

A _r /A _c percent	Strain, (Millionths)			
	0.15		0.75	
Cement Type	I	K	K	K
Expansion	60	660	290	-
Shrinkage	-450	-530	-340	-320
Net Strain	-390	130	-50	-130
Differential: Type K	-	+520	-	-

Kesler, et. al., after 120 days of drying @ 50 percent relative humidity.

Expansion is positive, shortening is negative.

Differential = net strain of Type K concrete minus net strain of Type I concrete.

Table 2.7. Typical Comparative Residual Strains for Reinforced Light-Weight Concrete.

A _r /A _c percent	Strain, (millionths)					
	0.28		0.46		1.72	
Cement type	I	K	I	K	I	K
Expansion	0	273	0	239	0	109
Shrinkage	-344	-283	-360	-266	-296	-238
Net Strain	-344	-10	-360	-27	-296	-129
Differential	-	+344	-	+333	-	+167

Russel, after 250 days of drying at 50 percent relative humidity. The reference also includes data for Types Sand M cements and position of reinforcement/expansion is positive, shortening is negative.

Differential = net strain of Type K concrete minus net strain of Type I concrete.

Isolation joints: Joints used to accommodate vertical movement or horizontal movement shall be provided at junctions with walls, columns, machine bases, footings, or the points of external restraint, e.g., pipe, stairways, etc. In addition to their normal action, these joints shall be used to accommodate the initial expansion of the concrete to allow the expansive strain to elongate the internal reinforcement.

Rigid exterior restraint shall not be used since it prevents expansion of the concrete and the least amount of shrinkage later will result in negative strains and tensile stresses in the concrete. In addition, large forces will be imposed on the restraining members. Laboratory tests have shown that rigid restraints result in stresses as high as 170 psi (1.2 MPa).

Stresses of this magnitude could produce sufficient force to damage the restraining structure. Footings, pits, walls, drains, and similar items should, therefore, be protected by isolation joints to prevent damage during the expansion stage of shrinkage-compensating concrete and allow the necessary expansive strains to develop. Compressible filler strips or joint materials shall be used for this purpose.

Isolation joints shall be composed of materials which are compressible enough to deform under the expansive action of the concrete. If too stiff, some rigid asphaltic isolation materials may act as external restraint and restrict the expansion of the concrete. A material with a maximum compression of 25 psi (170 kPa) at 50 percent deformation according to ASTM D-1621 or D-3575 should be used. Joint materials meeting ASTM D-994, D-1751, and D-1752 may be too stiff to allow adequate expansion.

Column box-outs may be reduced or eliminated if a compressible material is provided. A compressive bond-beaker wrapped around the column or compressible cardboard forms brought to floor level have been satisfactory in permitting vertical movement. At the same time, the reinforcement or mesh should be increased locally in the column area where high stresses are likely to develop. This will restrict width of any cracks that occur.

Construction joints: With the use of shrinkage-compensating concrete, slab placement patterns of approximately 20 to 30 ft or 6 to 9 m used with Portland cement concrete may be enlarged. Slabs located inside enclosed structures, or where temperature changes are small, may be placed in areas as large as 16,000 square ft or 1500 m² without joints. For areas where slabs are not under enclosed structures, slab placements are normally reduced to 7000 to 12,000 square ft or 650 to 1100 m². It is recommended that the area shall not be larger than a work crew can place and finish in a day.

Building slab sections should be placed as square as possible. the length-to-width ratio should not exceed 3:1. for pavements, which are thicker and more heavily reinforced than building slabs, successful installations have been made with higher length-to-width ratio.

Note that construction joints typically should be designed to accommodate temperature movements, allowing the opportunity for the joint to open, relieving the tensile stress acting on the slab. When load transfer is required, slip dowels at the joint should be used rather than deformed dowels. Tongue and grooved joints may be used

when I

require

joint m

The te

the per

in slat

stink

concre

a max

is pro

to 20

way a

stink

speci

port.

term

Howe

place

when large temperature contractions are not present and high load transfer is not required.

Bonded joints with deformed reinforcement (bars or mesh) passing through the joint may be used provided that only two slabs are locked together in each direction. The temperature, expansion, and shrinkage movement strains must be accommodated at the perimeter edges of the two slabs.

Contraction (control) joints: These joints are sawed, formed, or otherwise placed in slabs between other joints. Their primary purpose is to induce controlled drying shrinkage cracking along the weakened planes (joints). With shrinkage-compensating concrete, larger distances may be used between contraction joints. For exposed areas, a maximum spacing of 100 ft (30.5 m) between joints is recommended. Where the area is protected from extreme fluctuations in temperature and moisture, joint spacing of 150 to 200 ft (45.7 to 61 m) have been used. Contraction joints may be made in the same way as for Portland cement concrete. Normally, contraction joints are eliminated with shrinkage-compensating concrete except in high stress areas. However, the larger joint spacing with a shrinkage-compensating concrete will produce larger movement at the joint. This shall be taken into account when considering load transfer and joint sealing.

Expansion joints: The location and design of expansion joints for control of thermal movements are not changed with the use of shrinkage-compensating concrete. However, joints for thermal movements shall ensure that adequate expansion can take place during the expansion phase of the shrinkage-compensating concrete. An expansion

of 0.06

transfe

223.16

expar.

when

brace

ation

comp

comp

with

to A.

D-17

of 0.06 percent is equivalent to a 100° F temperature change. In the event of high load transfer, dowel bars should be provided. (See ACI Manual of concrete practice, page 223-16 Figure 3.4 b for more details).

Placing sequence: For slabs on grade, the placement sequence shall allow the expansive strains to occur against a free and unrestrained edge. The opposite end of slab when cast against a rigid element shall be free to move. A formed edge should have the brace stakes or pins loosened after the concrete is set to accommodate the expansive action. For this reason, checkerboarded placements should not be used unless a compressible joint material is placed between the slabs prior to concrete placement. The compressible joint material shall accommodate the anticipated movements. A material with a maximum compression of 25 psi (172 kPa) at 50 percent deformation according to ASTM D-1621 or D-3575 should be used. Joint materials meeting ASTM D-944, D-1751, and D-1752 may be too stiff to allow adequate expansion. [9, 26, 31, 52]

2.7 APPLICATIONS

Expansive cements have been used in a wide variety of concrete structures. One of the more frequent uses in the United States has been in parking structures, to prevent water leaks that can cause damage to cars. The largest such structure is the parking building at O'Hare International Airport in Chicago, which used 120,000 yd³ (90,000 m³) of shrinkage-compensating concrete.

In concrete pavements using expansive cement, the shrinkage control joints can be combined with thermal expansion control joints and other construction joints; the elimination of shrinkage control joints is an attractive advantage in the laying of pavements. The largest paving to date using this material is probably at the Love Field Airport at Dallas-Fort Worth, where more the 150,000 yd³ (1150,000 m³) of shrinkage-compensating concrete was used in taxiway. Use of shrinkage-compensating concrete in bridge deck overlays can help minimize cracking and control the corrosion of steel in bridge decks. In patching of concrete pavements, where shrinkage can cause separation, shrinkage compensating concrete has found broad applications.

Expansive cement can also be used in concrete structures where water-tightness is an important requirement, such as water storage tanks, swimming pools, and ice rinks. Shrinkage-compensating concrete has also been used in tilt-up construction, where residual prestress helps elements withstand the stresses imposed during lifting, and minimizes the separations between elements.

CHAPTER III

DEVELOPMENT OF EXPANSIVE CEMENT WITH PAINT SLUDGE POWDER

3.1 INTRODUCTION

Expansive cements are used to chemically induce compressive stresses in steel reinforced concrete, and thereby reduce the size and amount of restrained shrinkage cracks that frequently occur in Portland cement concrete during drying.

Expansions caused by the paint sludge powder can contribute to the development of prestress in restrained Portland cement concrete only if the expansions are generated after the concrete has gained rigidity through the hydration of cement. Any expansion produced while the concrete is still plastic is of little value. Temperature and humidity generally play important roles in deciding the rates of expansion and hydration; the physical and chemical properties of cement and mix proportions of concrete are some other factors with potentially significant effects on the balance between the expansion and hydration rates.

The interference of paint sludge powder with the hydration process of cement may influence the setting time and also the water requirements, processing procedures and durability of Portland cement concrete. The environmental impacts of using paint sludge powder in concrete also need to be investigated.

This phase of research intends to determine if the above technical requirements can be satisfied through the addition of Paint sludge powder to conventional Portland cement concrete.

Immediately after completion of molding, the specimens are covered with polyethylene sheet. They are removed from the molds 6 hours after addition of water to the cement during mixing operation, and then, allowed to air cure for 30 minutes from the time it was demolded before the initial length measurement is performed. The specimens are then cured in lime-saturated water at 73 °F (22.78 °C) until they have reached the age of 7 days (including the period in molds). The specimens are subsequently removed from water one at a time, wiped with damp cloth, and immediately measured for length with the cap nuts in place. Upon completion of each measurement, the specimens are returned to lime-saturated water. Another length measurement should be made at the age of 28 days (including the period in molds). It is suggested by the author to that length measurement be continued on a daily basis until the 28-day age is reached, and subsequently as the individual goal warrants.

According to ASTM C-845, mortar should also be tested for slump (ASTM C-143), air content (ASTM C-231), time of set (ASTM C-403), and compressive strength (ASTM C-39). An expansive cement should satisfy the requirements in Table 3.3 in order to be suited for use in shrinkage-compensating concrete.

3.2 PAINT SLUDGE POWDER

A process recently adopted by automobile assembly plants converts the liquid waste stream from painting operation into powder. There are three major components of this drying system [1]:

- 1) A dewatering device
- 2) A thermal fluid dryer with an oil heater
- 3) A back-end equipment to handle the solids that result from the drying process.

The dewatering device helps boost the solids concentration and increase the efficiency of the drying process when water/paint mixture is pumped into it. A circular flow of the mixture in this system forces the light paint solids to the top where a foamy raft is formed with the water below it. That top foamy part is the paint sludge and the water under it is pumped away. At this point, the raft (paint sludge) is siphoned off and propelled across a barrel-shaped wheel of wire mesh that wicks away more of the water before sending the paint sludge down the line. After passing through the dewatering device, the sludge has a solids concentration of about 25%. From here on, it is sent to the hopper with special solids handling pump, which moves the sludge onward to the thermal fluid dryer.

In the thermal fluid dryer, in order to reduce the volume of the sludge while preventing the paint from sticking to the screws from screw dryer, a variational screw dryer with two hollow, auger like screw conveyors are used that run parallel to the floor and slowly propel the sludge from one end of the dryer to the other in a continuous

process. This screw conveyor contains oil that has been heated to 350 °F (177 °C), and comes into contact with the paint sludge as it passes through the dryer. In this process, water vapor and other liquids including the volatile organic compounds that were contained in the paint solvents are driven off and fried. Within an hour, the sludge is broken down into gases and a residual powder.

From the dryer, the off gases are siphoned into a cyclonic separator, where a rotating drum traps the water vapor for return to the spray booth flushing system and sends the organic compounds to an after burner. The freshly cured sludge powder, which has the consistency of dust or fine sand, is collected, (see Figure 3.1).

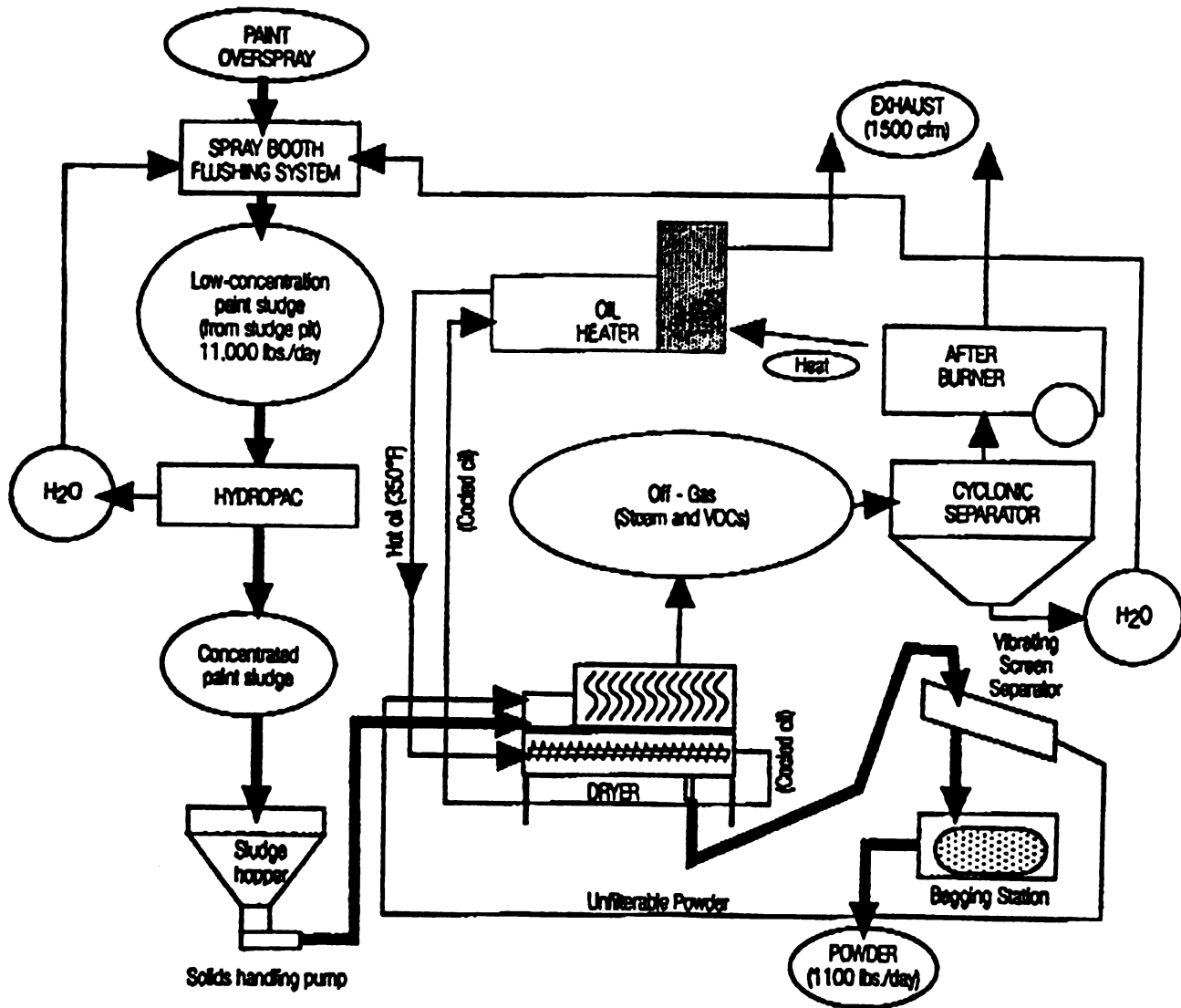


Figure 3.1 Paint Sludge Powder Production Flow Chart [57]

3.3 OVERALL APPROACH

3.3.1 SCREENING OF THE POWDERS

Four paint Sludge powders obtained from different paint systems were screened in order to select those suited for use in concrete as expansive admixtures. Six types of sludge powder obtained from the following paint systems were evaluated in this research:

- (1) PPG Solvent-Based
- (2) BASF Solvent-Based
- (3) PPG Water-Based
- (4) PPG Solvent/Water-Based

Some key aspects of the physical and chemical characteristics of these paint sludge powders were investigated using the ASTM C-311, X-Ray Diffraction and Inductively Coupled Plasma-Atomic Emission Spectroscopy procedures. Effects of the addition of different dosages of these powders to conventional concrete mixtures on the restrained expansion and some key aspects of fresh and hardened material properties were then investigated:

Test results on the physical and chemical characteristics of different paint sludge powders together with the observed effects of different powders on the restrained expansion and fresh and hardened concrete material properties provided the basis for the selection of two paint systems the sludge powders of which presented the highest promises for providing expansive effects in concrete without negatively influencing other short and long-term engineering properties of the material. These two sludge powders

were then considered through in all the remaining parts of the project. The effects of between-plant variations were also investigated through comparing the effects new shipments of PPG Solvent-based and BASF Solvent-based paint sludge powders from different plants (Dodge City and Jefferson, respectively).

3.4 EXPERIMENTAL PROGRAM

Effects of the addition of the Paint Sludge Powders to cement at different fractions (by weight of cement) on mortar and concrete mixtures were investigated following the ASTM C-878 and ASTM C-806 procedures. For comparison purposes, two control mixes were also considered; a type K expansive cement and a type I Portland cements were used in the control concrete and mortar mixtures.

The mix proportions used in this study after several repeated trials and errors are presented in Table 3.1. These mixes were selected to have reasonable workability and produce the best physical properties of the resulting concrete. Concrete and mortar mixtures were prepared in accordance with ASTM C-878 and ASTM C-806.

Table 3.1. Mix Designs of Experimental Program

Mix Designation	Aggregate/Cement	Coarse/Fine Aggregate	Water/Cement	Powder/Cement	Air Entraining Agent (by weight of Cement)
MORTAR					
Time of Set	2.75	Sand only	0.5	0.5, 1.0 & 1.5	0
Air Content	4.0	Sand only	0.92	0.5, 1.0 & 1.5	0
Flow	2.75	Sand only	0.5	0.5, 1.0 & 1.5	0
Restrained Expansion	2.75	Sand only	0.5	0.5, 1.0 & 1.5	0
Compressive Strength	2.75	Sand only	0.5	0.5, 1.0 & 1.5	0
CONCRETE					
All Tests	4.0 & 4.5	1.0	0.45 & 0.55	0.5, 1.0 & 1.5	0.05 & 0.10

The mortar and concrete tests were conducted following the ASTM C-806 and ASTM C-878 procedures, respectively. A summary of these test methods is presented below. While ASTM C-878 just presents the test methods to be employed on concrete specimens, ASTM C-806 is accompanied with limits on measured properties, presented in ASTM C-845 (see Table 3.2).

Table 3.2. ASTM C-845 Physical Requirements for Expansive Cement Mortar	
Time of Setting, Min., Minutes	75
Air Content, Max., Vol %	12.0
Restrained expansion of mortar: 7-day expansion: Min., % Max., % 28-day percentage of 7-day expansion, maximum.	0.04 0.10 115
Compressive Strength, min. 7-day, psi (MPa) 28-day, psi (Mpa)	2100 (14.7) 3500 (24.5)

3.5 RESTRAINED EXPANSION TEST PROCEDURES

3.5.1 MORTAR TESTS

The restrained expansion of mortar test specimens (ASTM C-806) are 2 x 2 x 10 in. (50.8 x 50.8 x 254 mm) mortar bars. Each bar is reinforced with 1/4 in. (6.35 mm) 20-threaded rod (Figure 3.2) placed along the longitudinal axis of the prism. The mortar is mixed mechanically following ASTM C-305 procedures with the proportions of one part of cement to 2.75 parts of graded standard sand by weight.

Immediately after completion of molding, the specimens are covered with polyethylene sheet. They are removed from the molds 6 hours after addition of water to the cement during mixing operation, and then, allowed to air cure for 30 minutes from the time it was demolded before the initial length measurement is performed. The specimens are then cured in lime-saturated water at 73 °F (22.78 °C) until they have reached the age of 7 days (including the period in molds). The specimens are subsequently removed from water one at a time, wiped with damp cloth, and immediately measured for length with the cap nuts in place. Upon completion of each measurement, the specimens are returned to lime-saturated water. Another length measurement should be made at the age of 28 days (including the period in molds). It is suggested by the author to that length measurement be continued on a daily basis until the 28-day age is reached, and subsequently as the individual goal warrants.

According to ASTM C-845, mortar should also be tested for slump (ASTM C-143), air content (ASTM C-231), time of set (ASTM C-403), and compressive strength (ASTM C-39). An expansive cement should satisfy the requirements in Table 3.3 in order to be suited for use in shrinkage-compensating concrete.

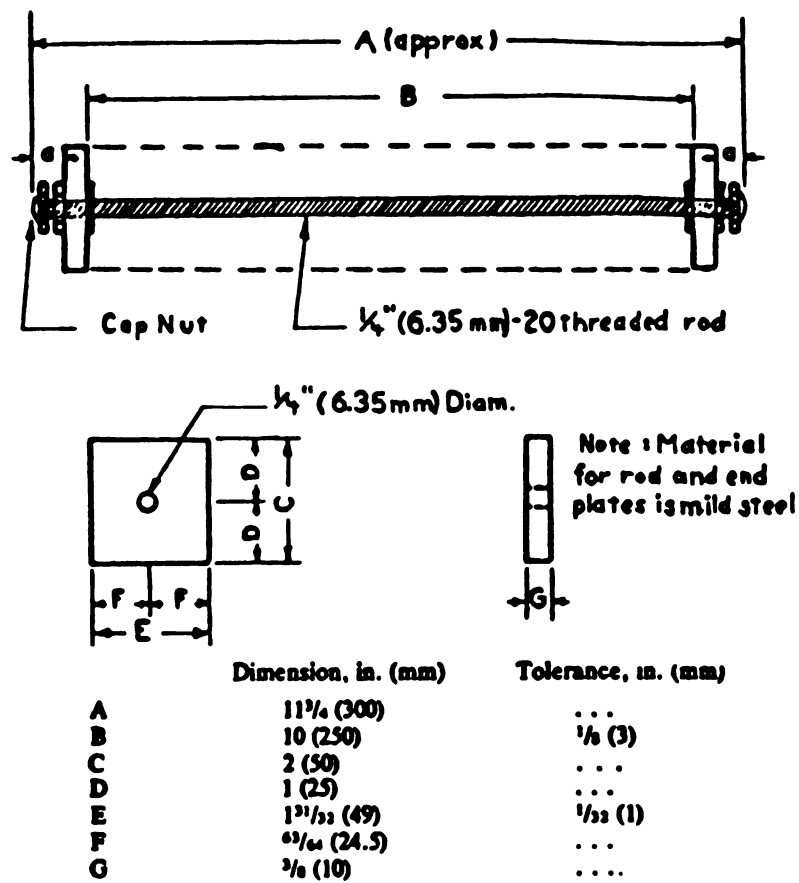


Figure 3.2. Restraining Mortar Cage Mold (ASTM C-806).

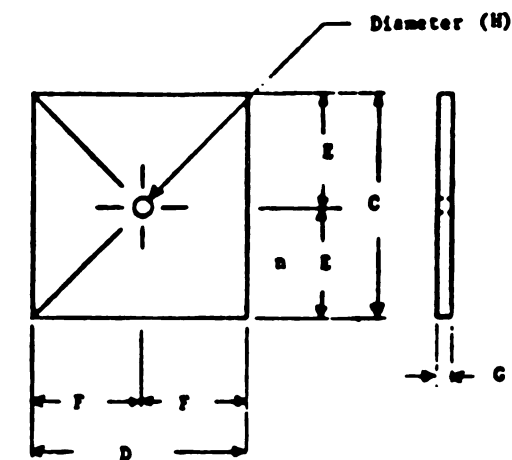
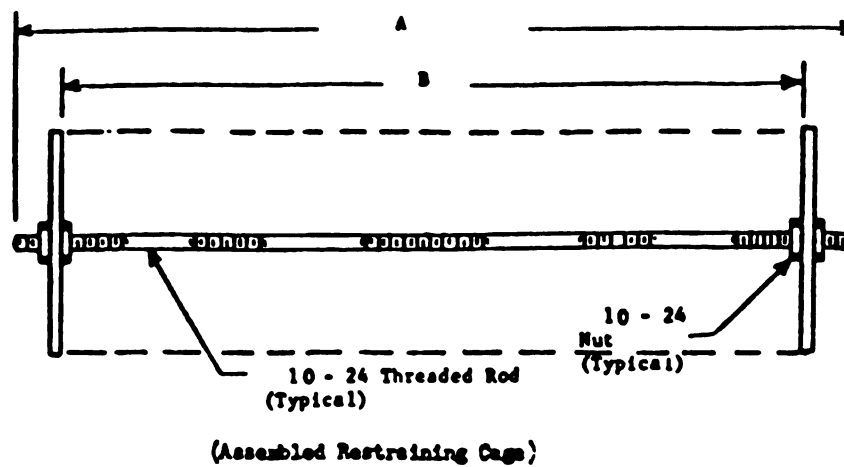
3.5.2 CONCRETE TESTS

The concrete test specimens (ASTM C-878) are 3 x 10 in. (76.2 x 254 mm) concrete. Each bar is reinforced with a 10-24 threaded rod (Figure 3.3). The rod is placed along the longitudinal axis of the prism. Concrete is mixed mechanically in accordance with ASTM C-305.

Immediately after completion of molding, the specimens are covered with polyethylene sheet. The specimens are then removed from the molds one hour after the mortar reaches its final set, as determined following the ASTM C-403 test method. Initial length measurement should be made 30 minutes after demolding.

The specimens are then cured in lime saturated water at 73 °F (22.78 ° C) until they have reached the age of 7 days, including the period in molds. The specimens are then removed from water one at a time, and their length is immediately measured with the cap nuts in place. No additional length measurement is required beyond the 7-day period, unless these measurements are technically desired.

There are no limits specified in ASTM C-878 for the restrained expansion of concrete.



Dimensions		
	in.	mm
A	$11\frac{1}{2} \pm \frac{1}{8}$	293 ± 1.5
B	10	254 (page length)
C	3	76
D	$2\frac{1}{2} \pm \frac{1}{8}$	75 ± 1
E	1 $\frac{1}{2}$	38
F	$1\frac{1}{4}$	37.5
G	$\frac{1}{2}$	10
H	$\frac{1}{8}$	3

Note—All material is mild steel.

Figure 3.3 Restraining Concrete Cage Mold (ASTM C-878)

3.6 EXPERIMENTAL RESULTS.

This section presents the powder properties, restrained expansion test results for mortar and concrete specimens, and fresh mix and hardened material test results on mortar specimens for conditions with different powder types and contents.

3.6.1 POWDER PROPERTIES

The properties of the powders and the two control cement materials used in the investigation are presented in Table 3.3.

Table 3.3. Properties of the Paint Sludge Powders and Type K Cement				
Properties	Type I Portland Cement	Type K Expansive Cement	PPG Solvent Based	PPG Water Based
Silicon Dioxide (SiO_2)	21	21.6	6.5	11.7
Aluminum Oxide (Al_2O_3)	6	4.8	9.2	6.8
Ferric Oxide (Fe_2O_3)	3	2.8	0.92	1.8
Calcium Oxide (CaO)	63	62.4	7.9	17.4
Magnesium Oxide (MgO)	2	0.7	0.5	0.78
Total Alkalies (Na_2O)	0.43	0.18	0.29	0.31
Specific Gravity	3.15	3.07	0.59	0.59

3.6.2 RESTRAINED EXPANSION TEST RESULTS

3.6.2.1 MORTAR TEST

The restrained expansion test results mortar specimens prepared with the four paint sludge powders (at 0.5%, 1% and 1.5% by weight of cement), as well as the control Type K expansive cement and regular Type I Portland cement, are presented in Figures 3.4 (a, b and c) and 3.5 (a, b and c) for 7-day and 28-Day tests respectively. These results suggest that the PPG-SB (PPG Solvent-Based) and PPG-WB (PPG Water-Based) at 1% by weight of cement satisfy the minimum 7-day and the maximum 28-day limits on the restrained expansion of mortar as specified in ASTM C-845. However, since the 7-day measured expansions are rather close to the minimum limits, the increase in powder dosage from 1% to 1.5% give a comfortable space in satisfying the ASTM C-845 limits.

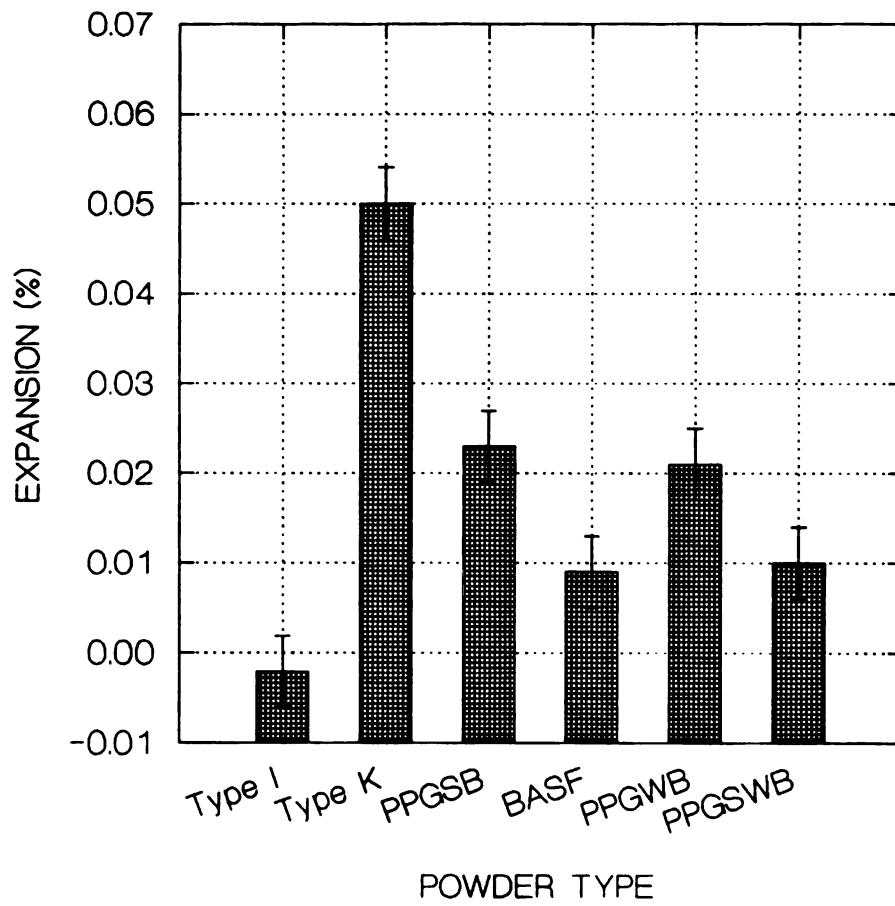


Figure 3.4 (a). 7-Day Restrained Expansion Test Results for Mortar Mixtures (0.5% Powder).

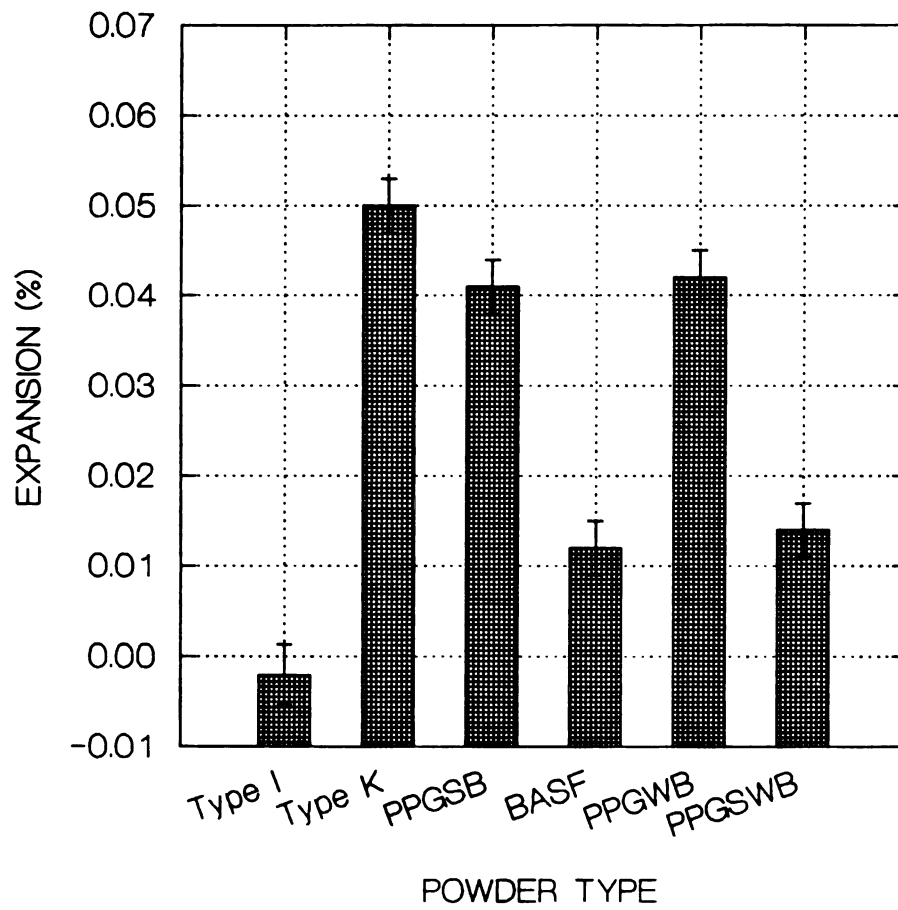


Figure 3.4 (b). 7-Day Restrained Expansion Test Results for Mortar Mixtures (1% Powder).

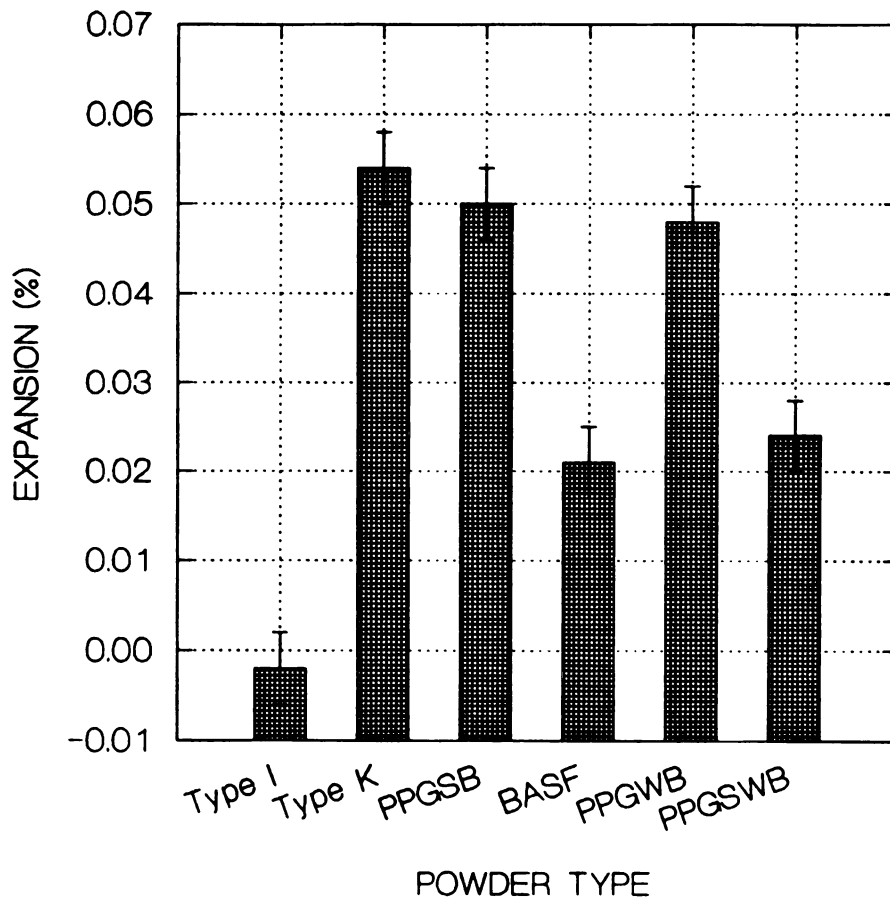


Figure 3.4 (c). 7-Day Restrained Expansion Test Results for Mortar Mixtures (1.5% Powder).

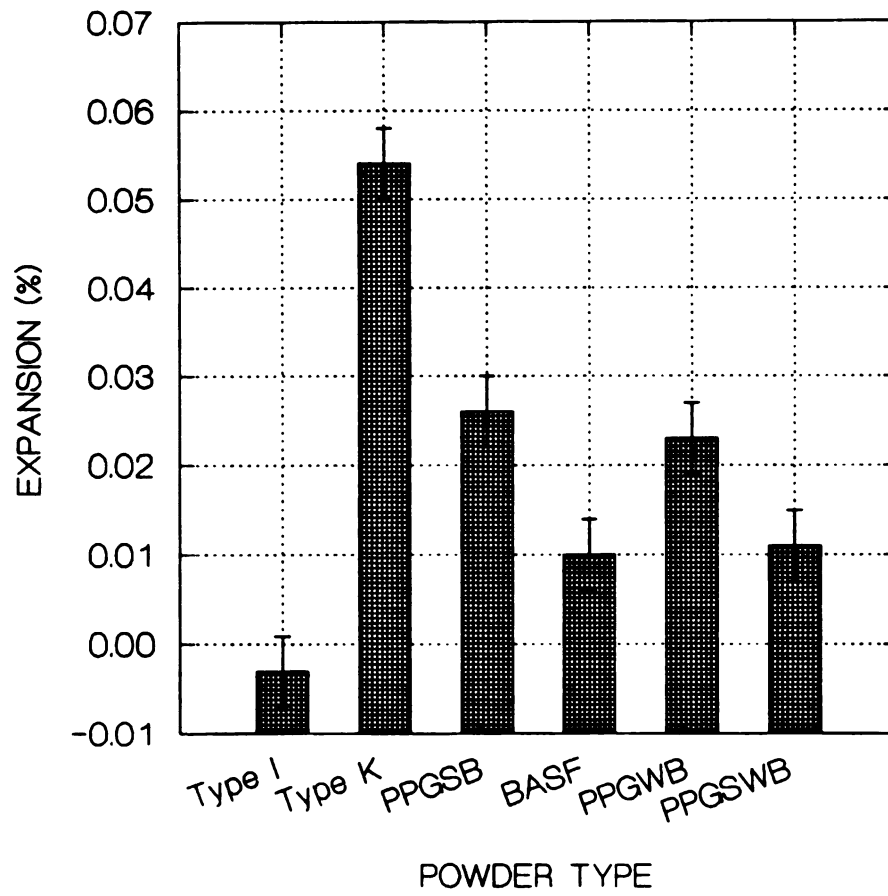


Figure 3.5 (a). 28-Day Restrained Expansion Test Results for Mortar Mixtures (0.5% Powder).

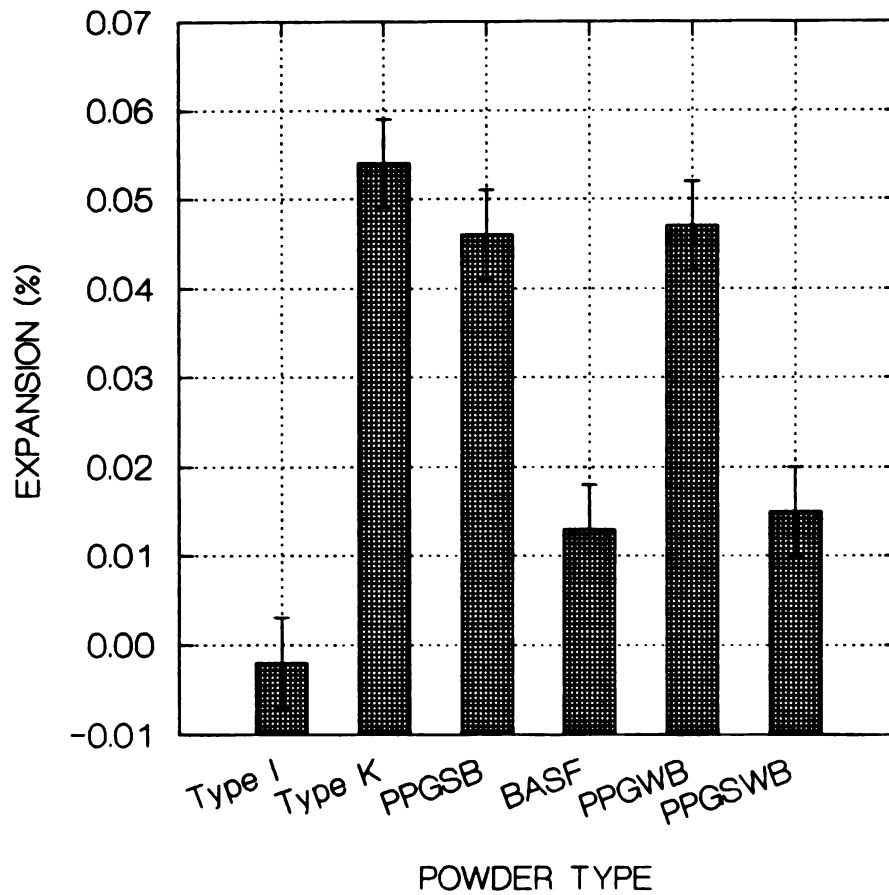


Figure 3.5 (b). 28-Day Restrained Expansion Test Results for Mortar Mixtures (1% Powder).

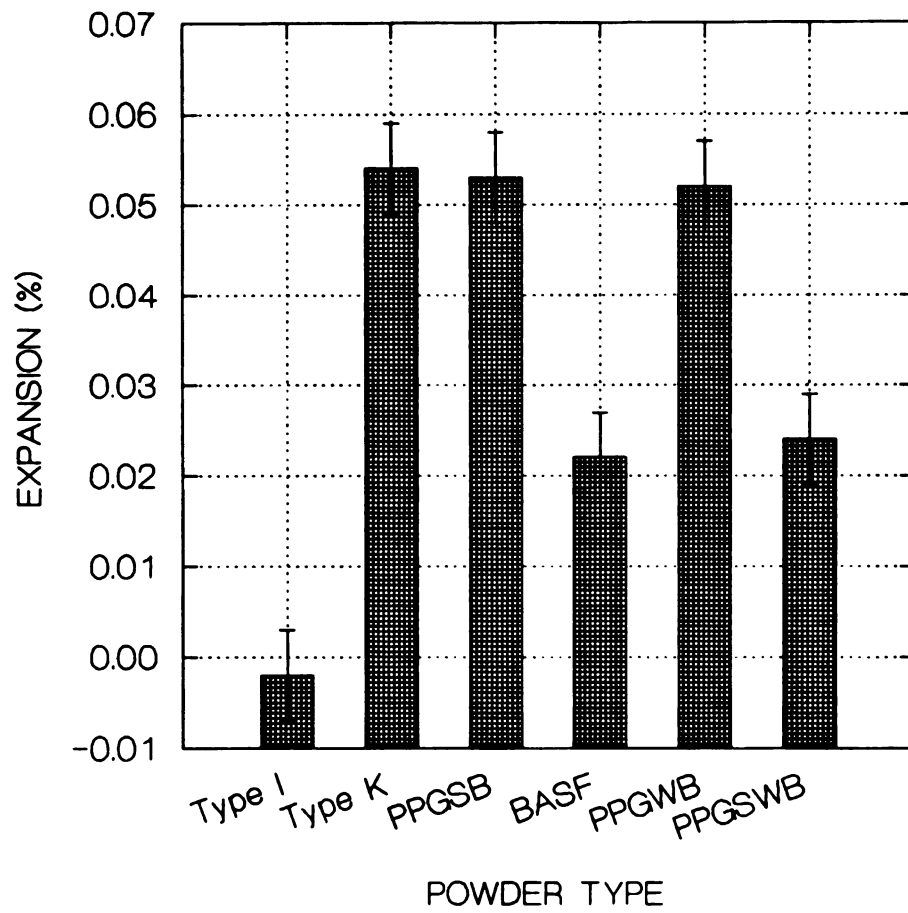


Figure 3.5 (c). 28-Day Restrained Expansion Test Results for Mortar Mixtures (1.5% Powder).

3.6.2.2 CONCRETE TEST

The 7-day and 28-day restrained expansion test results for concrete mixtures incorporating paint Sludge powders at 0.5%, 1% and 1.5% by weight of cement are presented in Figures 3.6 (a,b and c) and Figures 3.7 (a, b and c).

ASTM C-878 does not provide any limits on the restrained expansion of concrete mixtures. A comparison of the performance of different paint sludge powders in concrete with that of type K cement confirms that the PPG-SB and PPG-WB, which both performed well in mortar, have adequate potentials and can produce (1.5% by weight of cement) restrained expansions in concrete comparable to those of Type K expansive cement concrete.

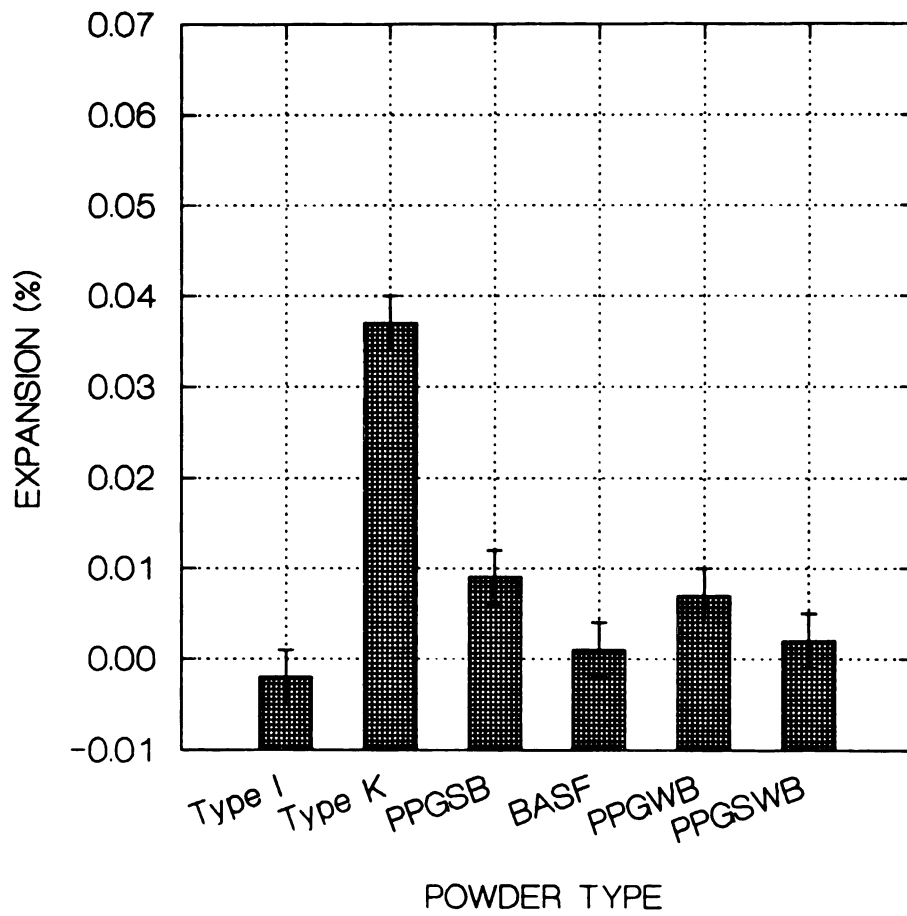


Figure 3.6 (a). 7-Day Restrained Expansion Test Results for Concrete Mixtures (0.5% Powder).

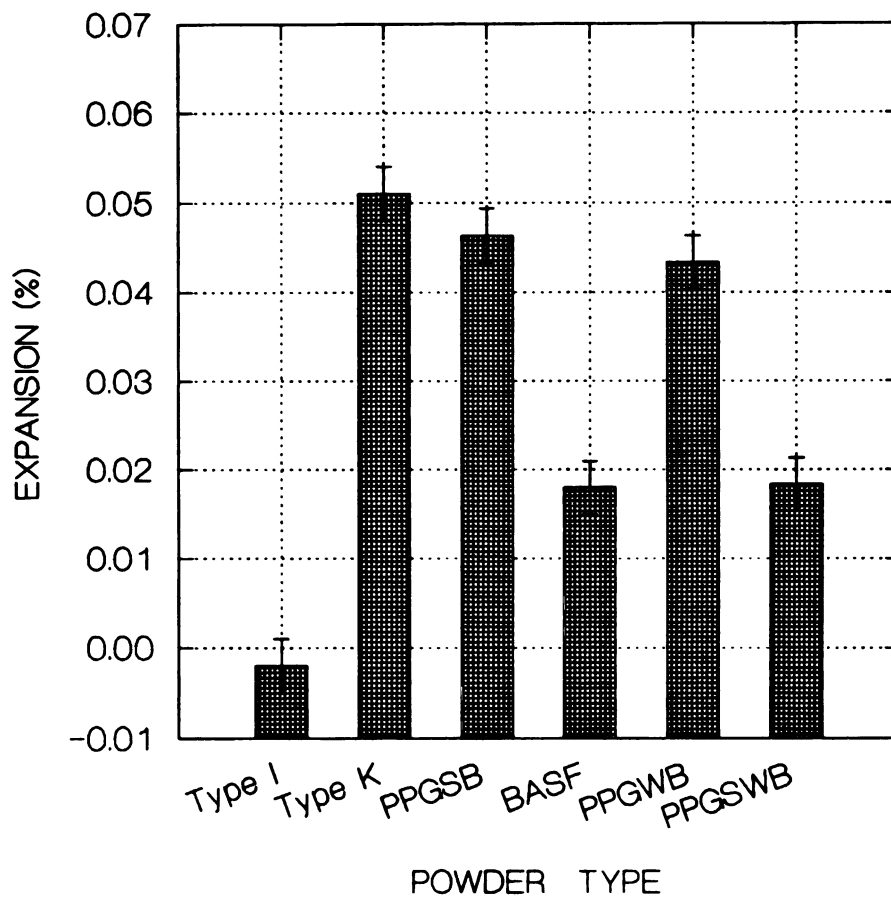


Figure 3.6 (b). 7-Day Restrained Expansion Test Results for Concrete Mixtures (1% Powder).

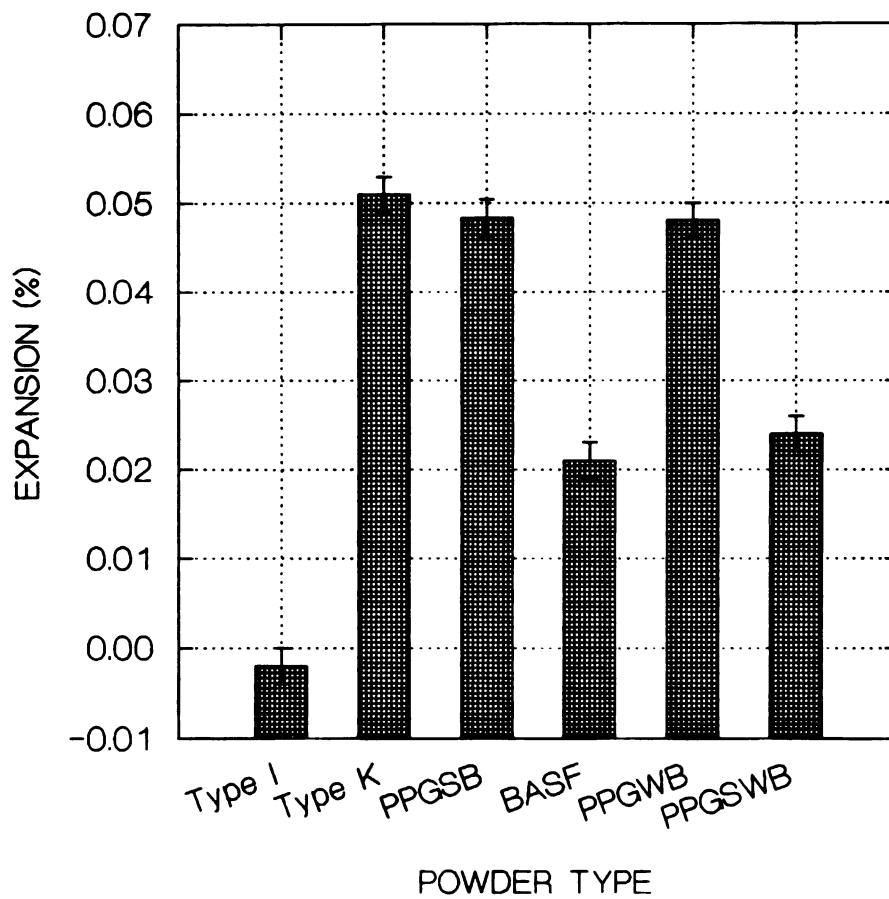


Figure 3.6 (c). 7-Day Restrained Expansion Test Results for Concrete Mixtures (1.5% Powder).

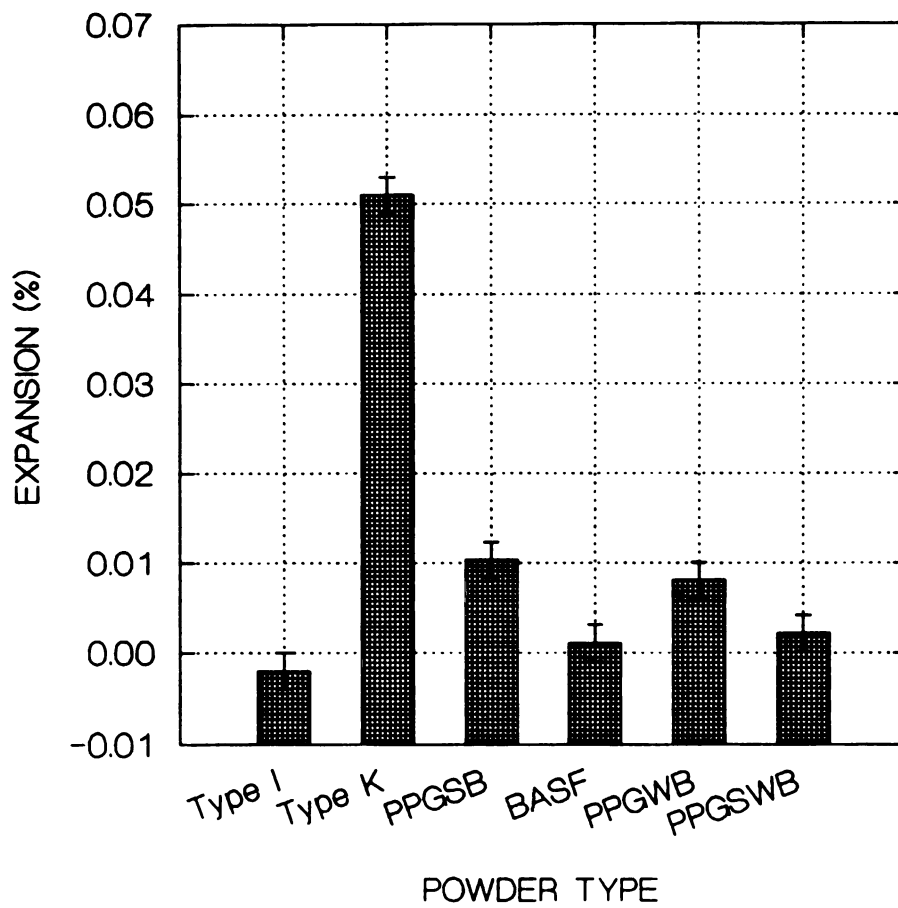


Figure 3.7 (a). 28-Day Restrained Expansion Test Results for Concrete Mixtures (0.5% Powder).

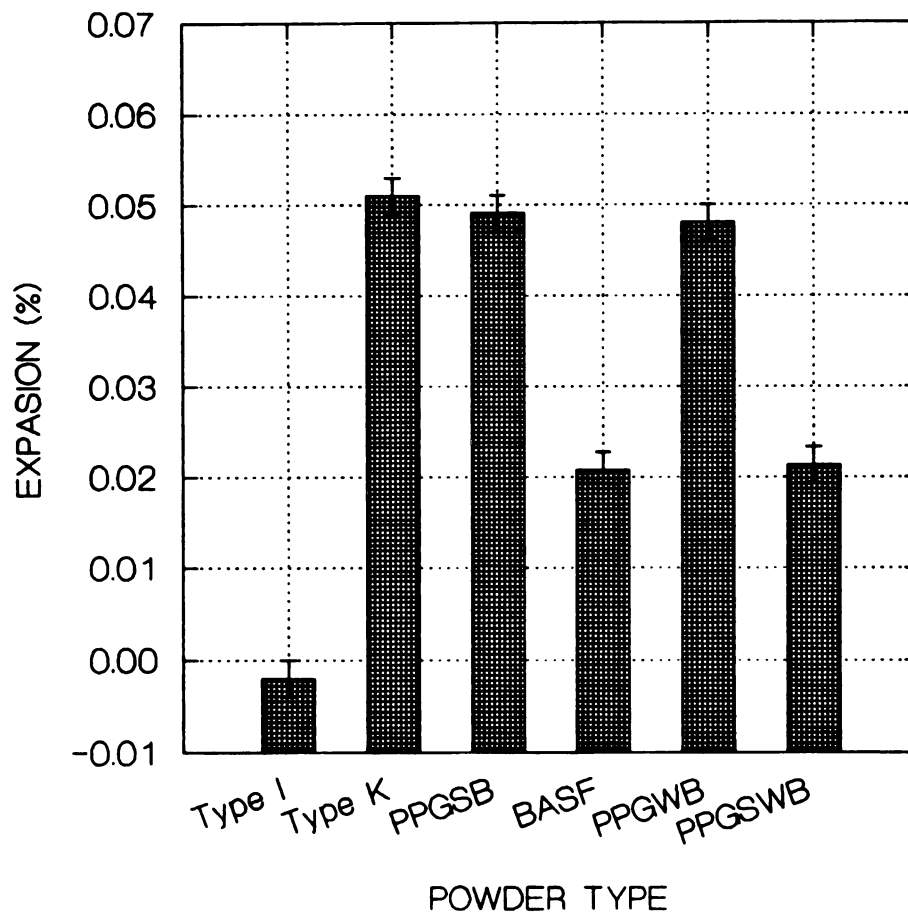


Figure 3.7 (b). 28-Day Restrained Expansion Test Results for Concrete Mixtures (1% Powder).

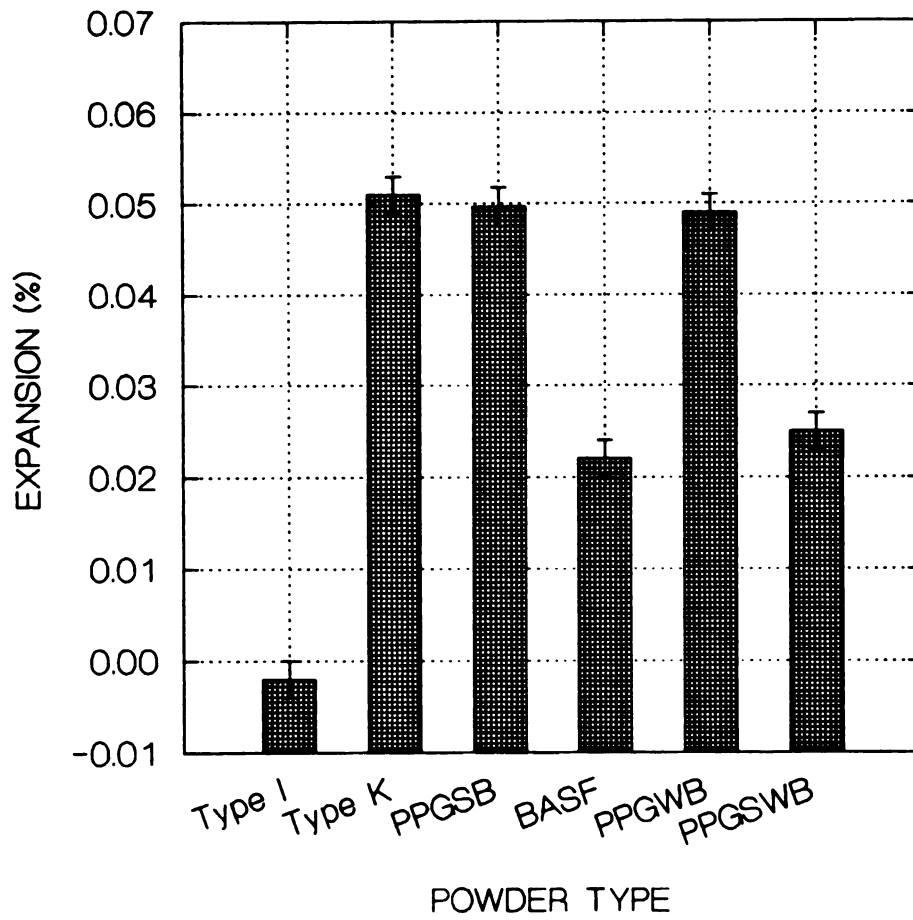


Figure 3.7 (c). 28-Day Restrained Expansion Test Results for Concrete Mixtures (1.5% Powder).

3.6.3 SET TIME

The paint sludge powders produced mortars satisfying the ASTM C-845 minimum requirement on mortar time of set as shown in Figure 3.8 (a, b and c), for different percentages of the powders by the weight of cement. The same is also true for concrete, though there is no ASTM requirements for concrete time of set. It has also been found statistically at 95% level of confidence that there is no significant difference in time of set between different powders.

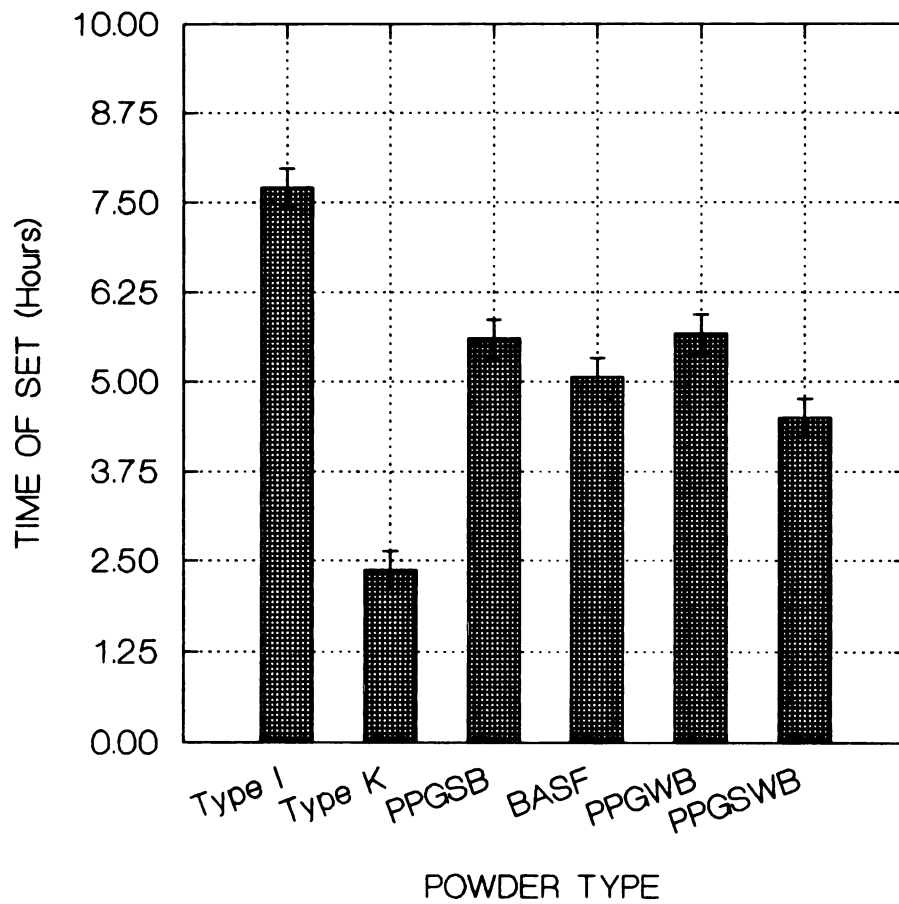


Figure 3.8 (a). Final Time of Set Test Results (0.5% Powder)

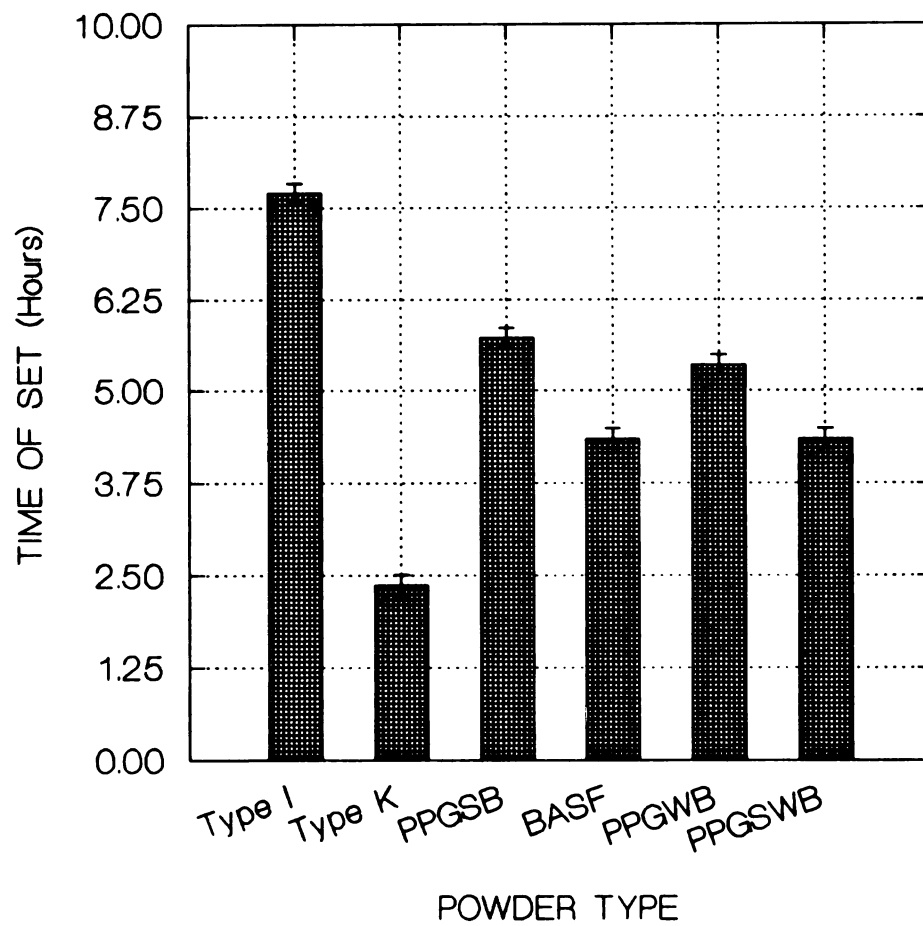


Figure 3.8 (b). Final Time of Set Test Results (1% Powder)

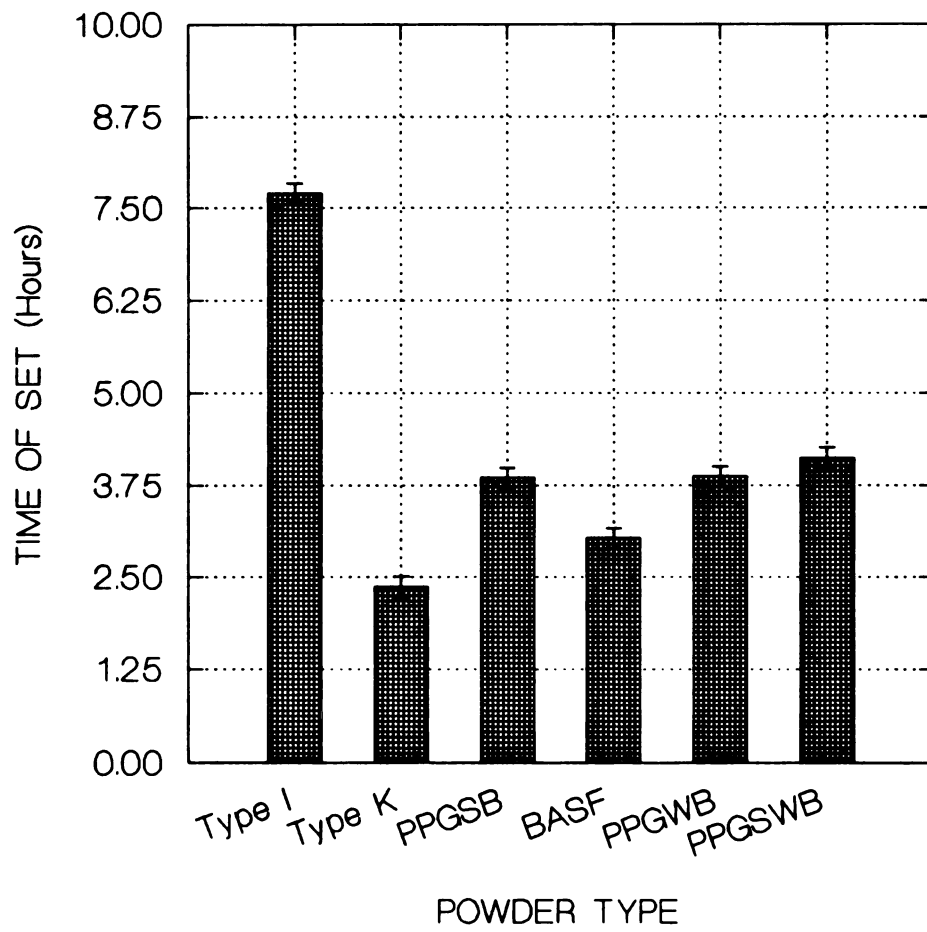


Figure 3.8 (c). Final Time of Set Test Results (1.5% Powder)

3.6.4 FLOWABILITY

Although some conventional expansive cements require about 10% more water to obtain the same amount of workability with conventional cement concrete, the paint sludge powders used here presented no workability problems. They all compared well with type K expansive cement in flowability obtained following ASTM C-230 test procedure. There is no statistical difference at 95% level of confidence between the workability of powders as shown in Figure 3.9.

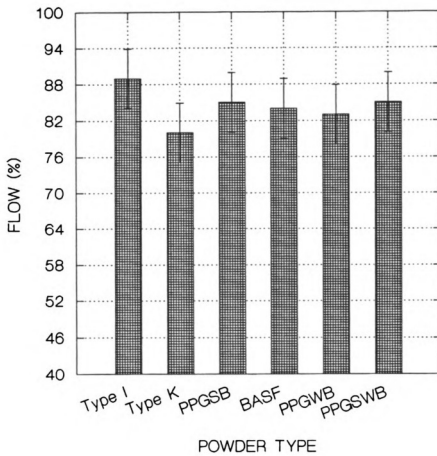


Figure 3.9. Effect of Paint Sludge Powder on Fresh Mix Flowability.

3.6.5 SLUMP

Workability of fresh concrete determines the ease with which a concrete mixture can be handled without harmful segregation. In all likelihood, a concrete mixture that is difficult to place and consolidate will not only increase the cost of handling but will also have poor strength, durability, and appearance. Similarly, mixtures that are prone to segregation and bleeding are more expensive to finish and will yield less durable concrete. Thus, workability can affect both the cost and the quality of concrete mixtures.

The slump test may be considered to be a measure of the shear resistance of the concrete flowing under its own weight.

None of the paint sludge powders used caused an adverse effects on workability of the mixtures used in this investigation. There is no statistical difference at 95% level of confidence between the slump of concretes made with different powders (at the three dosage levels used) as shown in Figure 3.10 (a, b and c).

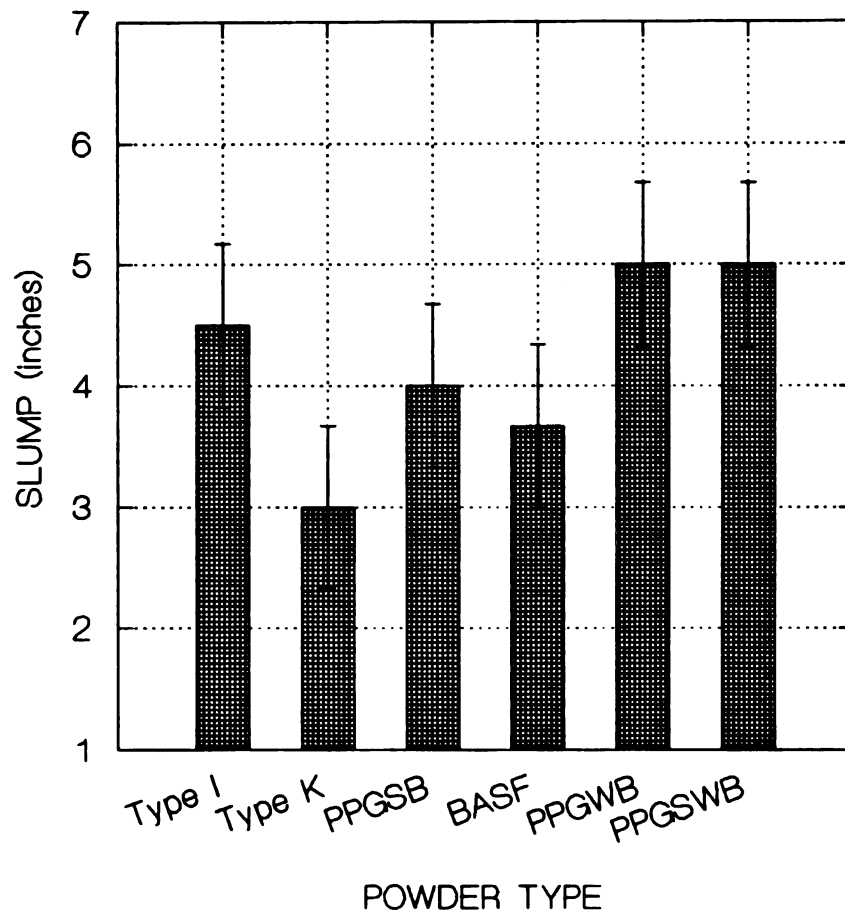


Figure 3.10 (a). Slump Test Results (0.5% Powder)

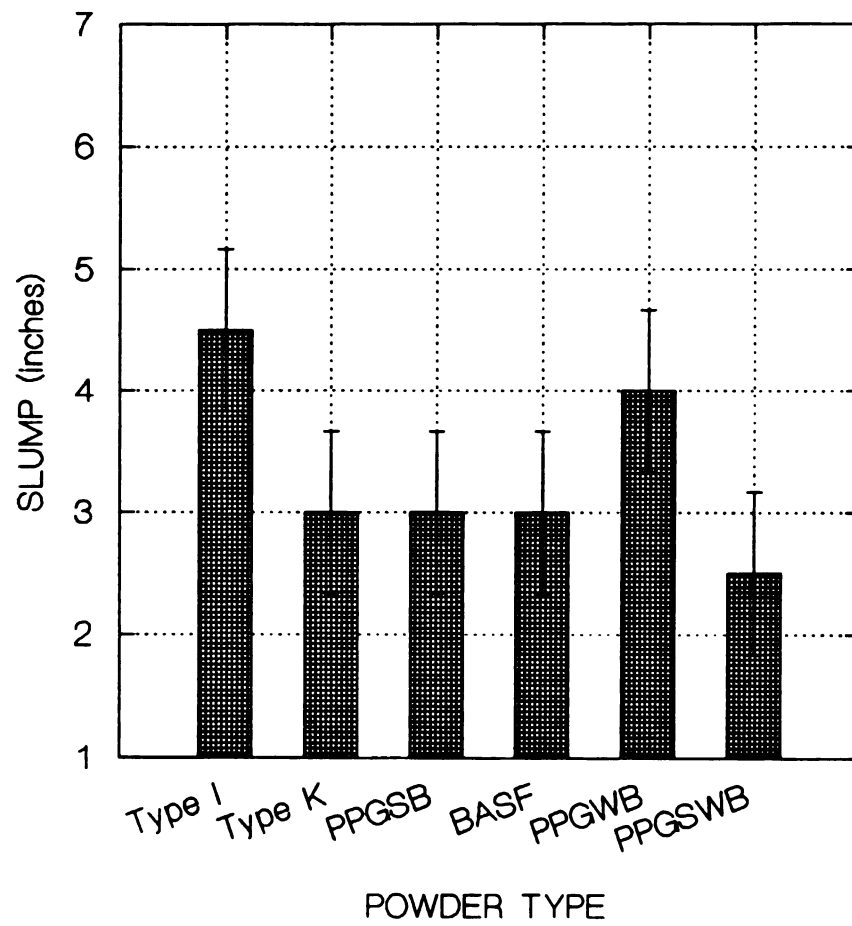


Figure 3.10 (b). Slump Test Results (1% Powder)

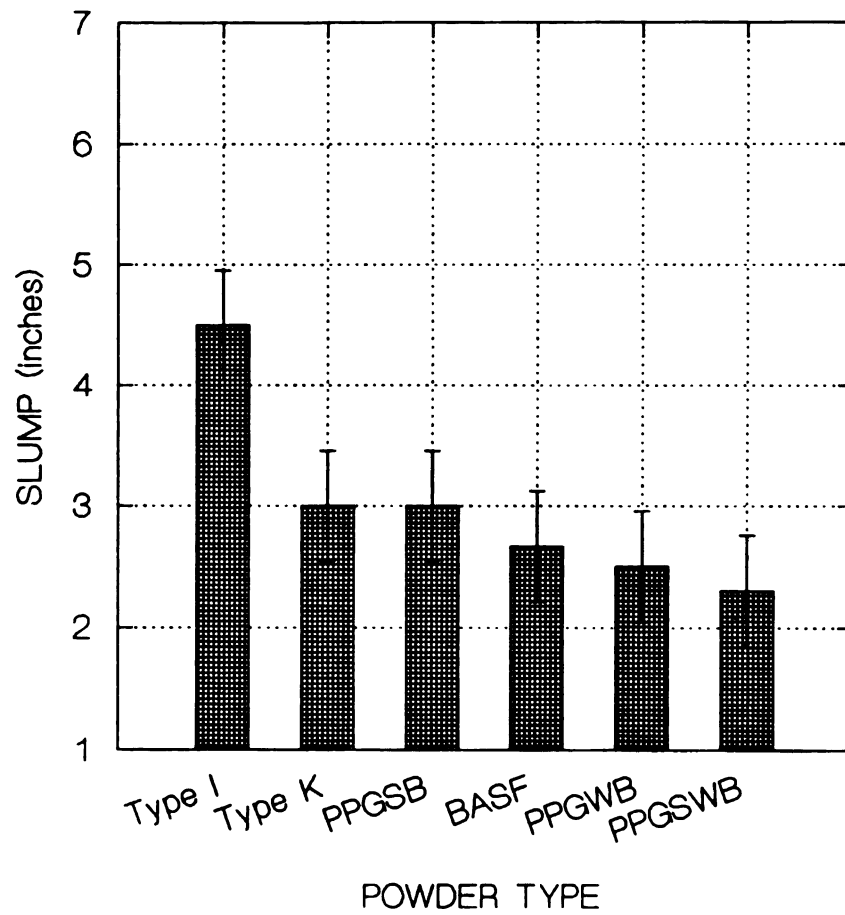


Figure 3.10 (c). Slump Test Results (1.5% Powder)

3.6.6 AIR CONTENT

Although entrained air for improving the frost resistance of concrete is present only in the cement paste, it is usual to specify and measure the air content as a percentage of the volume of the concrete. Table 3.4 presents the required air contents for freeze-thaw durability.

Table 3.4 Recommended air content of concretes containing aggregates of different maximum size, according to ACI 201.2R-77, (BS 8110: Part 1:1985). [9]				
Maximum Size of Aggregate		Recommended total air content of concrete for different levels of exposure (percent)		
		ACI		BS
mm	in. (approx.)	Moderate	Sever	Wet Conditions and De-icing salts
10	3/8	6.0	7.5	7.0
12.5	1/2	5.5	7.0	-
14	1/2	-	-	6.0
20	3/4	5.0	6.0	5.0
25	1	4.5	6.0	-
40	1-1/2	4.5	5.5	4.0
50	2	4.0	5.0	-
70	3	3.5	4.5	-
150	6	3.0	4.0	-

There are three methods of measuring the *total* air content of fresh concrete: *gravimetric* (ASTM C-138); *volumetric* (ASTM C-173); and *pressure* (ASTM C-231). Since the entrained air cannot be distinguished in these tests from the large bubbles of accidental air, it is important that the concrete be fully compacted. The most dependable and accurate method is the pressure method, which is based on the relation between the volume of air and the applied pressure (at a constant temperature) given by Boyle's law. The mix proportions or the material properties need not be known and the percentage of air is obtained directly. However, at high altitudes, the pressure meter must be re-calibrated, and the method is not suitable for use with porous aggregates.

A typical air meter is shown in Figure 3.11. The procedure consists essentially of observing the decrease in volume of a sample of compacted concrete when subjected to a known pressure as applied by a small pump. When the pressure gauge shows the required value, the fall in the level of the water in the calibrated tube above the concrete gives the decrease in volume of air in the concrete, i.e. the percentage of air content.

The paint sludge powders did not affect the air content in mortar as shown in Figure 3.12 (a, b and c), for different percentages of the powders by the weight of cement. The same is also true for concrete. It was confirmed statistically, at 95% level of confidence, that there is no significant difference in air content between different powders. It should be noted that all the mixtures had a dosage of 0.05% (by weight of cement) air entraining agent.

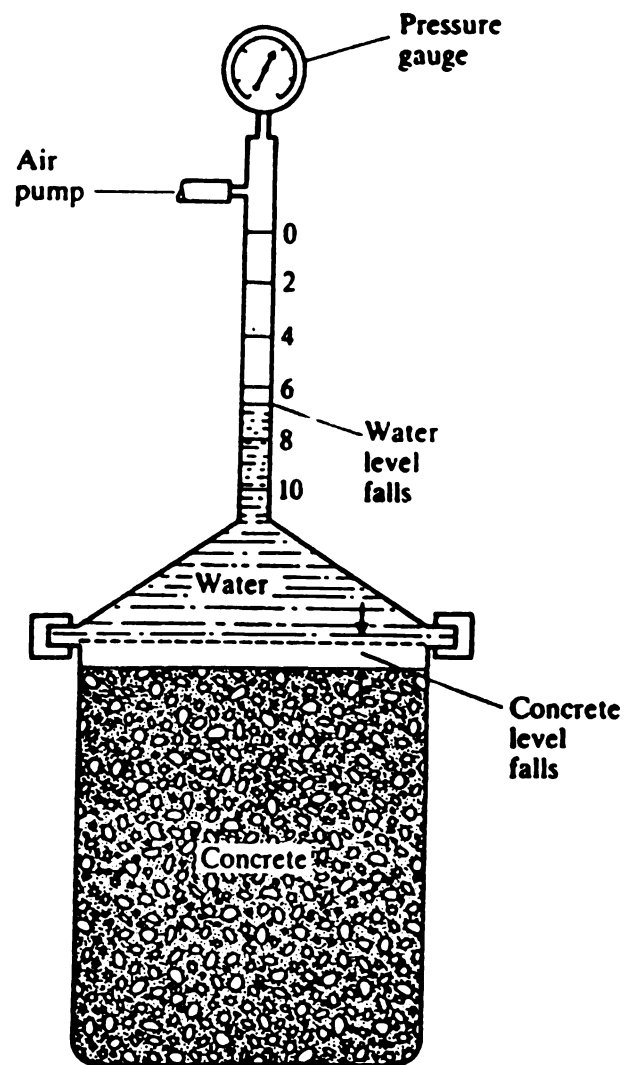


Figure 3.11. Pressure Type Air Meter [13]

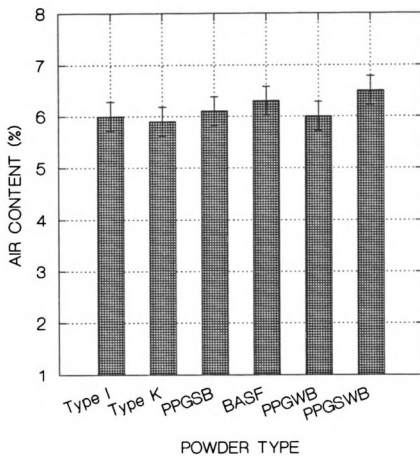


Figure 3.12 (a). Air Content Test Results, (0.5%)

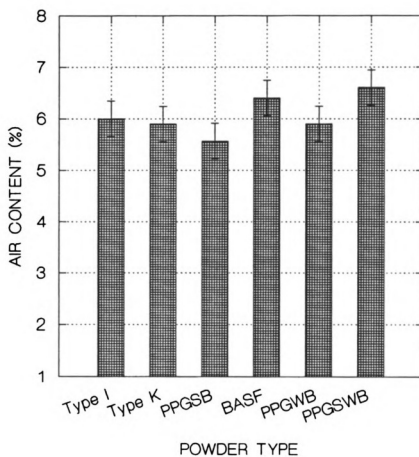


Figure 3.12 (b). Air Content Test Results, (1%)

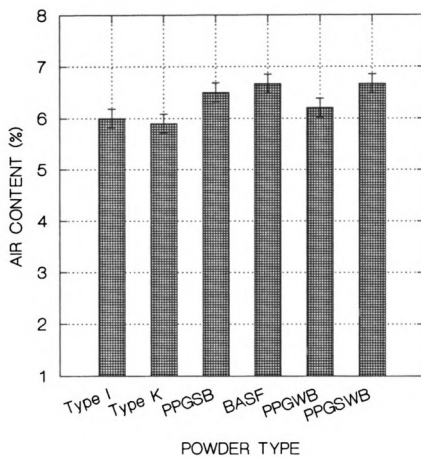


Figure 3.12 (c). Air Content Test Results, (1.5%)

3.6.7 UNIT WEIGHT AND YIELD

The density (unit weight) of the fresh concrete can be determined by weighing a known volume of concrete (ASTM C-138). The presence of entrained air affects the unit weight. The unit weight of normal-weight concrete can be used as an approximate indication of air content for concretes made with the same materials.

The unit weights of the paint sludge powders are given Figures 3.13 (a, b and c). Statistical analysis of results indicated that the powder type has no significant effect at 95% level of confidence on unit weight. It was further concluded that the dosage of powder (from 0.5% to 1.5% used in this investigation) has no statistically significant effect at 95% level of confidence.

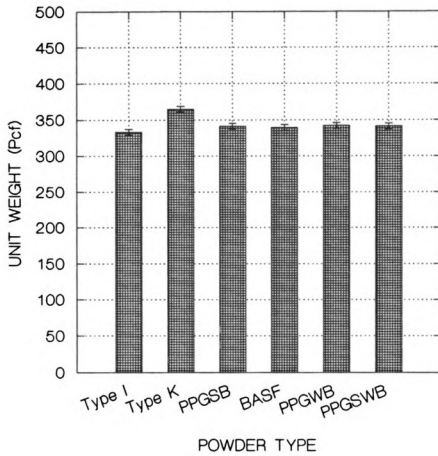


Figure 3.13 (a). Unit Weight Result (0.5% Powder)

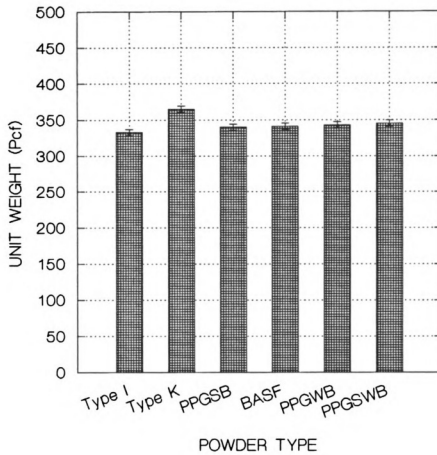


Figure 3.13 (b). Unit Weight Result (1% Powder)

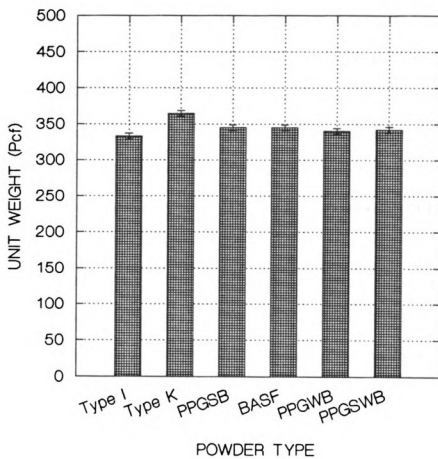


Figure 3.13 (c). Unit Weight Result (1.5% Powder)

3.6.8 STRENGTH

The strength of a material is defined as the ability to resist stress without failure. Failure is sometimes identified with the appearance of cracks. However, it should be noted that unlike most structural materials, concrete contains fine cracks even before it is subjected to external stresses. In concrete, therefore, strength is related to the stress required to cause fracture and is synonymous with the degree of failure at which the applied stress reaches its maximum value. In tension tests, fracture of the test usually signifies failure; in compression the specimen is considered to have failed when no sign of external fracture is visible, yet internal cracking is so advanced that the specimen is unable to carry a higher load without fracture.

The compressive strength of the cylinders is determined according to ASTM C-39 at a constant loading rate of 0.15 to 0.34 MPa/s (20 to 50 psi/s) for hydraulically operated machines, or at a standard deformation rate of 1.3 mm/min. (0.05 in./min.) for mechanically operated machines. The maximum recorded load divided by the area of cross section of the specimen gives the compressive strength, which is reported to the nearest 10 psi (0.05 MPa).

ASTM C-845 has a minimum requirement for 7-day and 28-day compressive strengths achieved with expansive cement. The two paint sludge powders selected to be carried on to the end of this investigation (PPG-SB and PPG-WB) both meet minimum compressive strength requirements (see Figure 3.14).

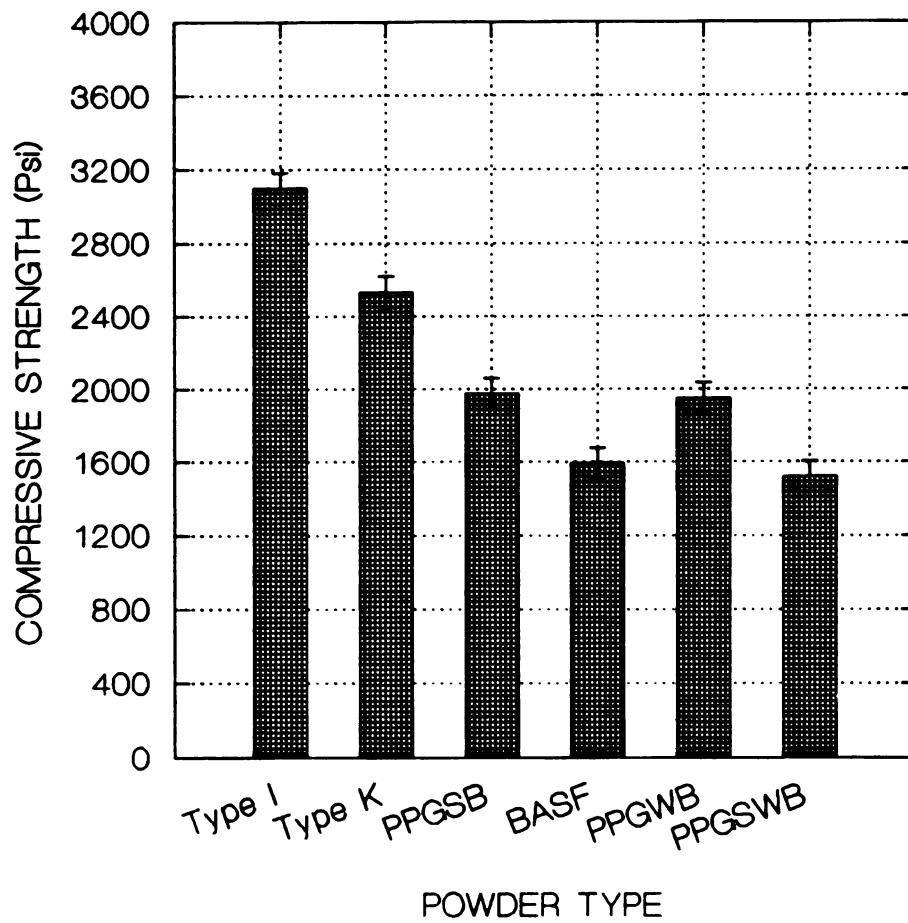


Figure 3.14 (a). Effects of Paint Sludge Powder on 7-Day Compressive Strength (0.5% Powder)

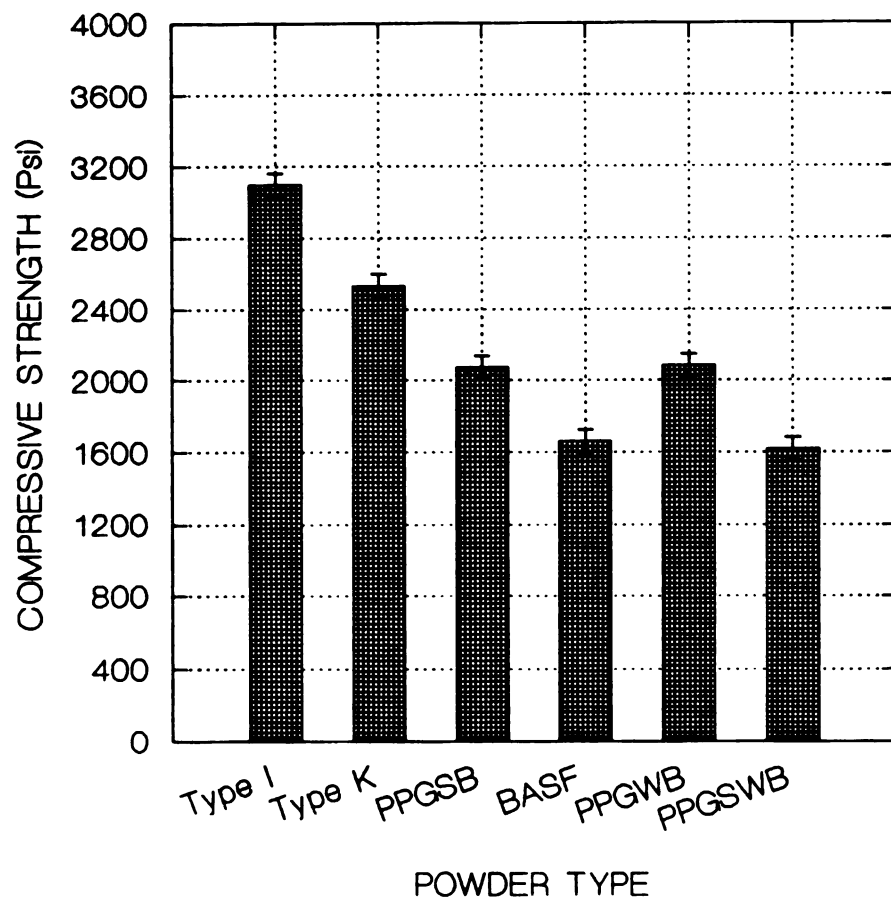


Figure 3.14 (b). Effects of Paint Sludge Powder on 7-Day Compressive Strength (1% Powder)

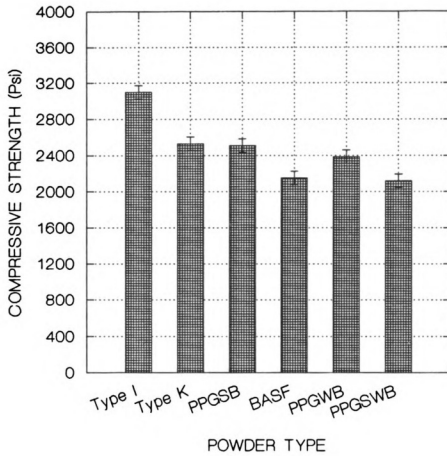


Figure 3.14 (c). Effects of Paint Sludge Powder on 7-Day Compressive Strength (1.5% Powder)

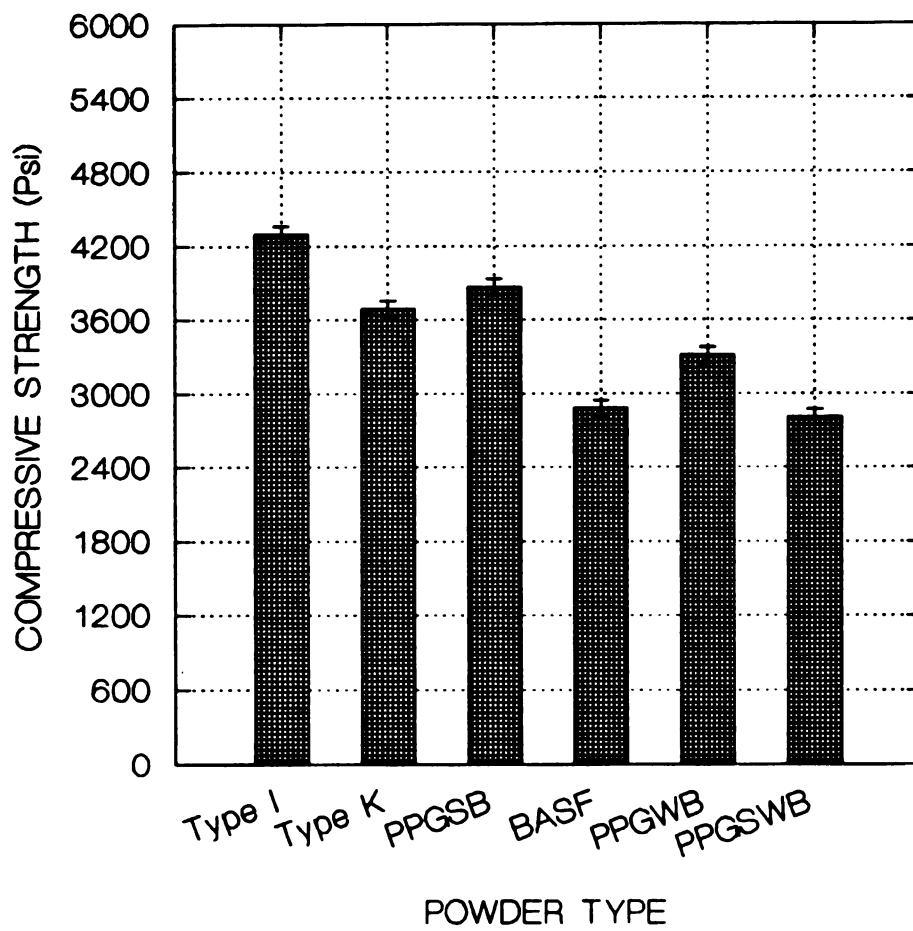


Figure 3.15 (a). Effects of Paint Sludge Powder on 28-Day Compressive Strength (0.5% Powder)

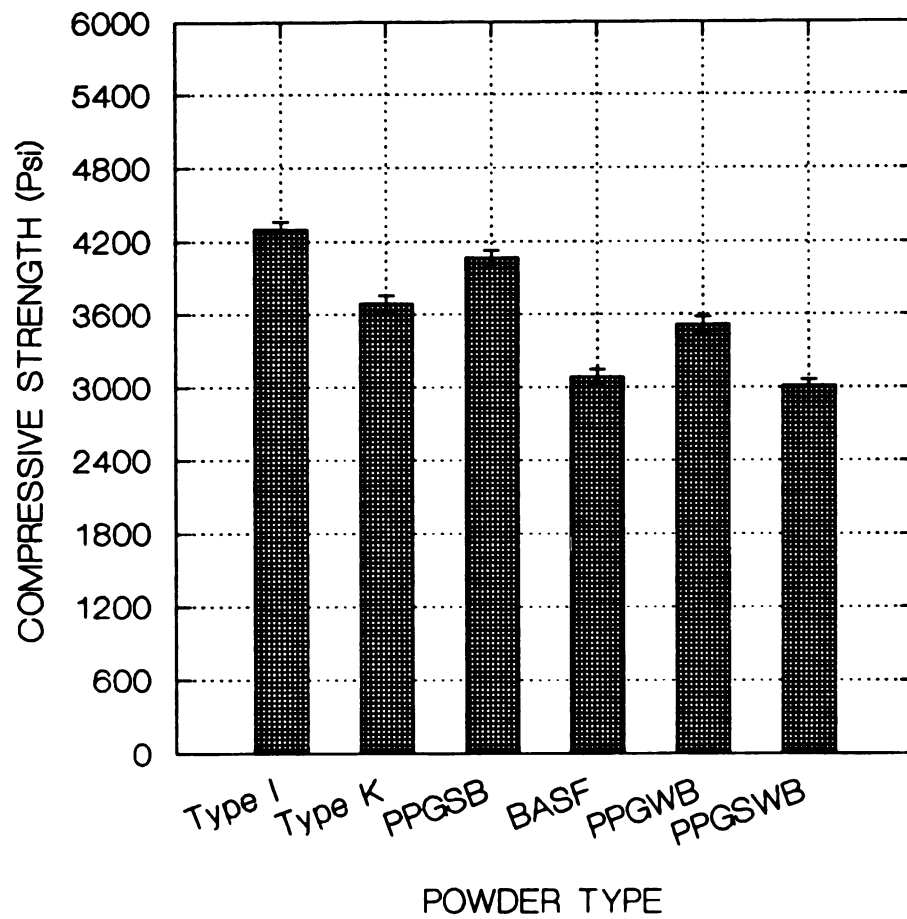


Figure 3.15 (b). Effects of Paint Sludge Powder on 28-Day Compressive Strength (1% Powder)

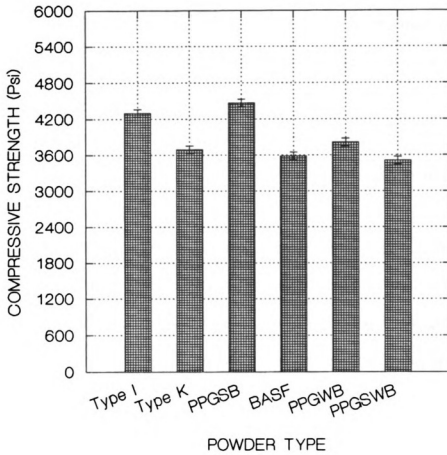


Figure 3.15 (c). Effects of Paint Sludge Powder on 28-Day Compressive Strength (1.5% Powder)

3.6.9 MODULUS OF ELASTICITY

The stress-strain curve for concrete is not linear. However, to calculate the stiffness or expected deflection of structural members, it is necessary that some estimation of the modulus of elasticity, E , be obtained. Although it is possible to define E in a variety of ways for a material with a nonlinear stress-strain curve, the most common way for concrete is to measure the chord modulus of elasticity, E_c , following the method outlined in ASTM C-469.

The observed results of the paint sludge powder Modulus of elasticity compares well with expansive cement type K. The ratio of modulus of elasticity verses compressive strength is shown in Figure 3.17.

The modulus of elasticity test results presented in Figure 3.16 were analyzed statistically and no significant effects of the powders could be detected at 95% level of confidence. The ratio of modulus of elasticity to compressive strength (Figure 3.16) further confirm that the addition of powders does not significantly change the mechanical properties of concrete.

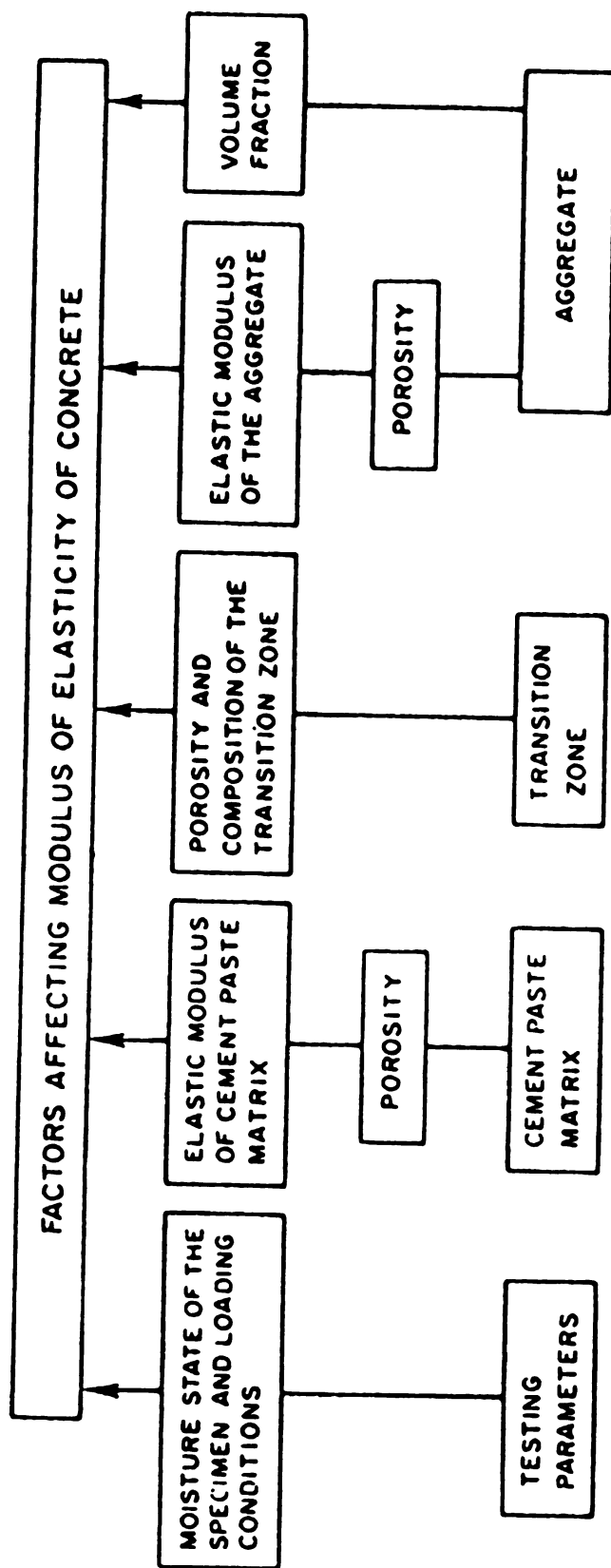


Figure 3.16. Various parameters that influence the modulus of elasticity of concrete.

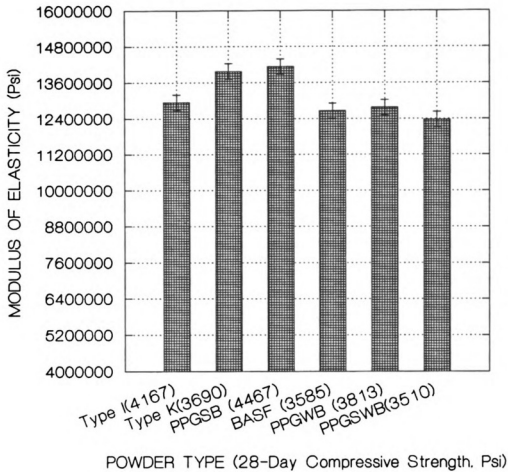


Figure 3.17. Modulus of Elasticity Versus Compressive Strength of Paint Sludge Powder.

3.6.10 FLEXURAL STRENGTH

One way of estimating the tensile strength of concrete is by flexural test (ASTM C-78). The specimen is cast in a beam of size 152 x 152 x 508 mm (6 x 6 x 20 in.) and the loading rate is between 0.0143 and 0.020 Mpa/sec (2.1-2.9 psi/sec). The beams are cured in the standard manner and are then tested in flexure by third-point loading, as shown in Figure 3.18.

In the flexural test, the theoretical maximum tensile stress reached in the bottom fibre of a test beam is known as the *modulus of rupture* (f'_b), which is relevant to the design of slabs ongrade, and highway and airfield pavements. The value of the modulus of rupture depends on the dimensions of the beam and, above all, on the arrangement of loading. Nowadays, symmetrical two-point loading (at third points of the span) is used both in the UK and the US. This produces a constant bending moment between the load points so that one third of the span is subjected to the maximum stress, and therefore it is there that cracking is likely to take place.

If fracture occurs within the middle one-third of the beam, the modulus of rupture (f_b) is calculated, to the nearest 0.1 Mpa (15 psi), on the basis of ordinary elastic theory.

$$f_b = (Pl)/(bd^3)$$

where P = maximum total load,

l = span,

d = depth of the beam, and

b = width of the beam;

ASTM C-78 allows for failure outside the load points, say, at an average distance from the nearest support, by the equation:

$$f_b = (3Pa)/(bd^3)$$

If, however, failure occurs at a section such that $(l/3 - a) > 0.051$, then the result should be discarded.

The flexural strength test results are presented in Figure 3.19. The paint sludge powder improved the flexural performance of concrete when compared to conventional expansive cement. Different powders performed similarly at 95 % level of confidence.

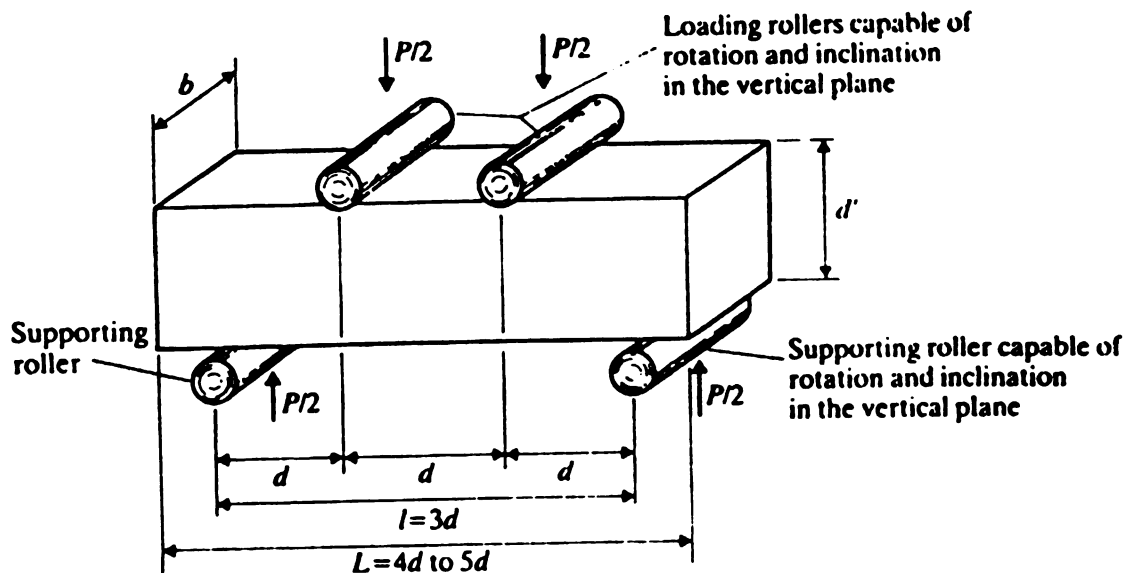


Figure 3.18. Arrangement of the Modulus of Rupture Test. [13]

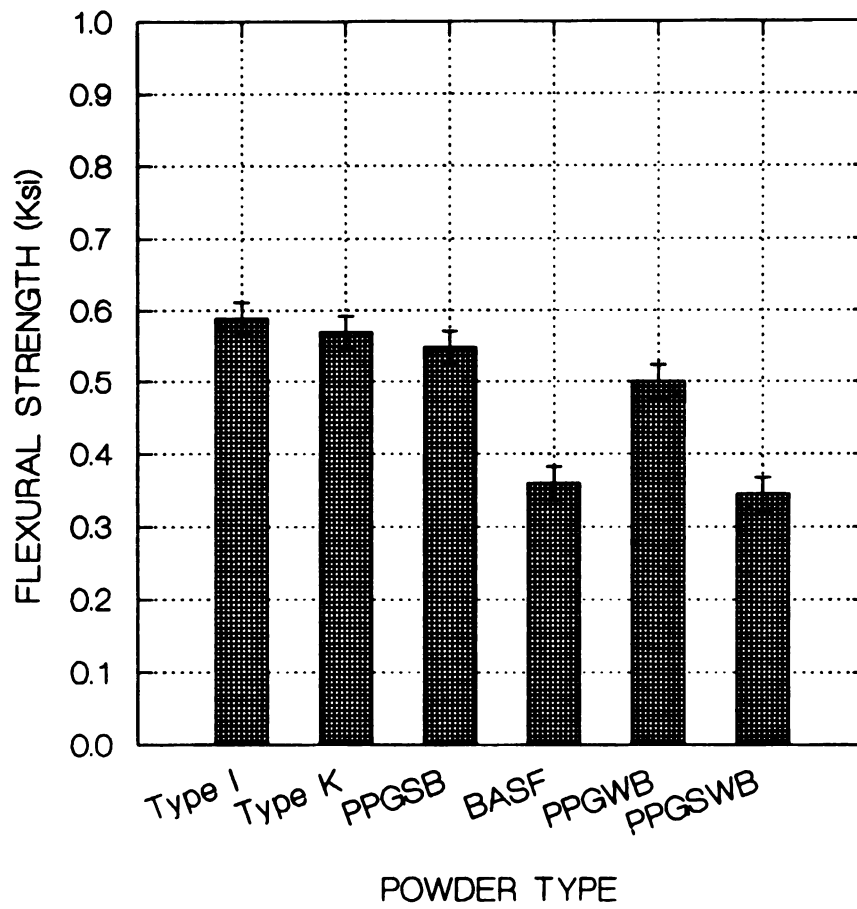


Figure 3.19 (a). Flexural Strength of Paint Sludge Powder Concrete (0.5% Powder).

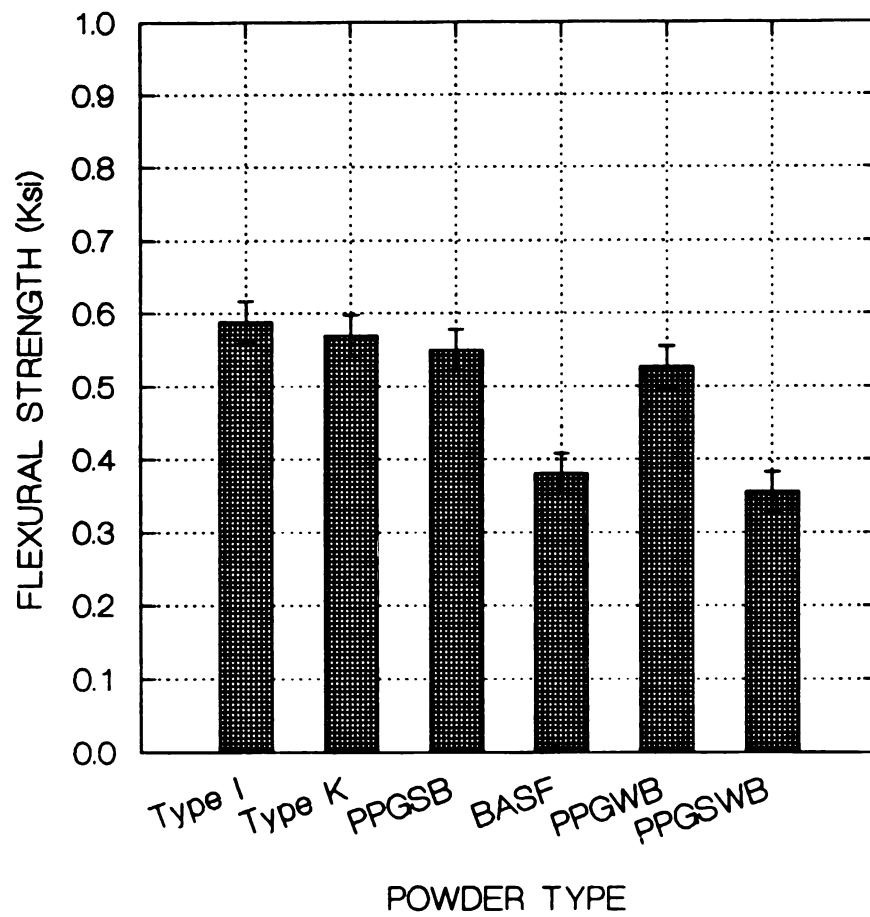


Figure 3.19 (b). Flexural Strength of Paint Sludge Powder Concrete (1% Powder).

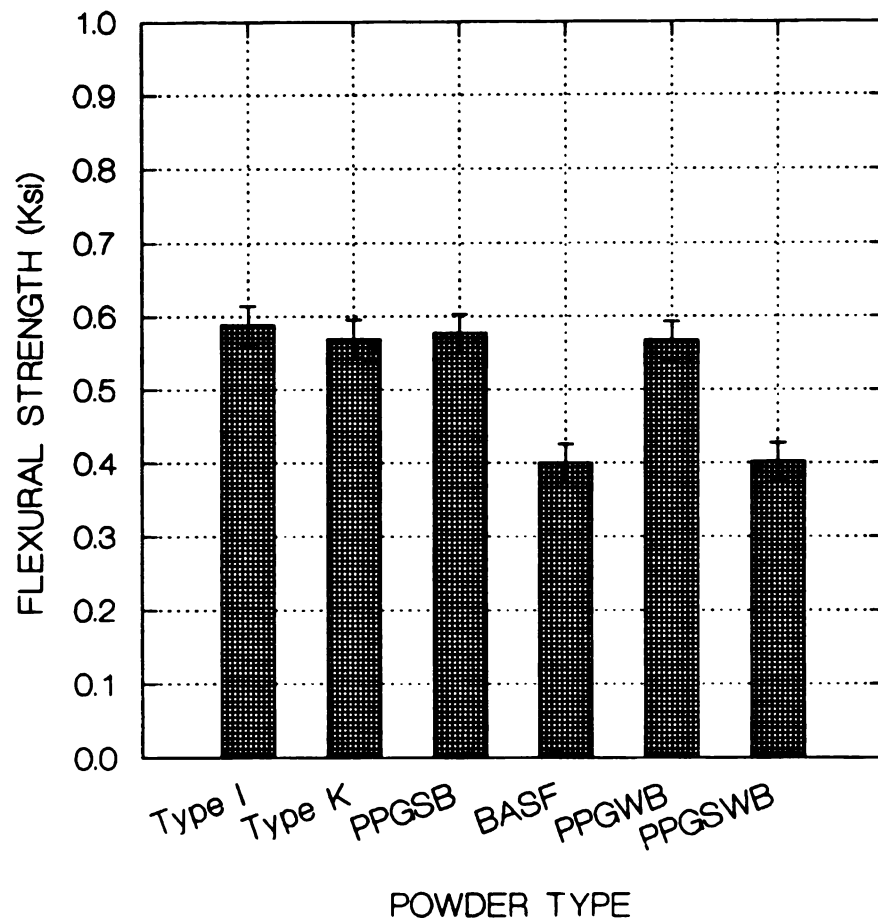


Figure 3.19 (c). Flexural Strength of Paint Sludge Powder Concrete (1.5% Powder).

3.7 VARIATIONS BETWEEN SHIPMENTS

Two different shipments of PPG Solvent-Based and BASF Solvent-Based paint powders were examined.

The two shipments were compared in all aspects of performance. No significant difference was observed between the qualities of powders incorporating 1.5% of different shipments of powders derived from similar paint systems (see Figure 3.20). The consistency in the performance of different shipments is particularly significant, noting that the two shipments of BASF Solvent-Based powders were obtained from two different plants using the same paint system. In these Figures, [PPGSB-1 = Shipment 1, PPGSB-2 = Shipment 2, BASF-1 = Shipment 1 and BASF-2 = Shipment 2]

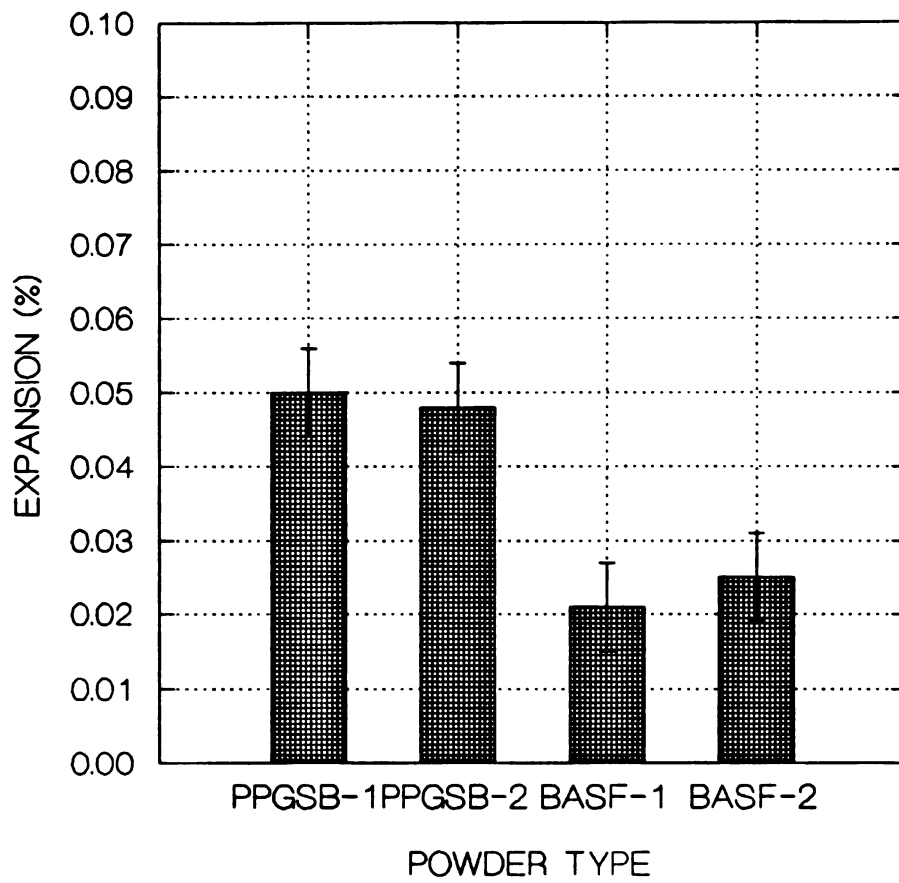


Figure 3.20 (a). Between Shipment Variations in 7-Day Expansion

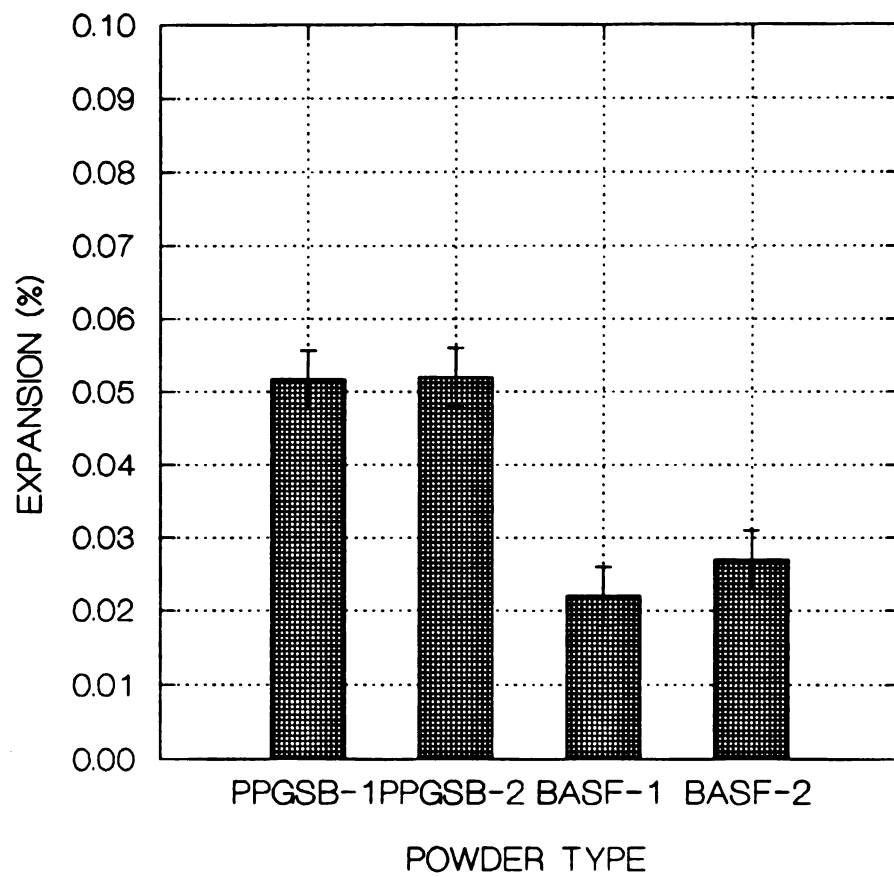


Figure 3.20 (b). Between Shipment Variations in 28-Day Expansion

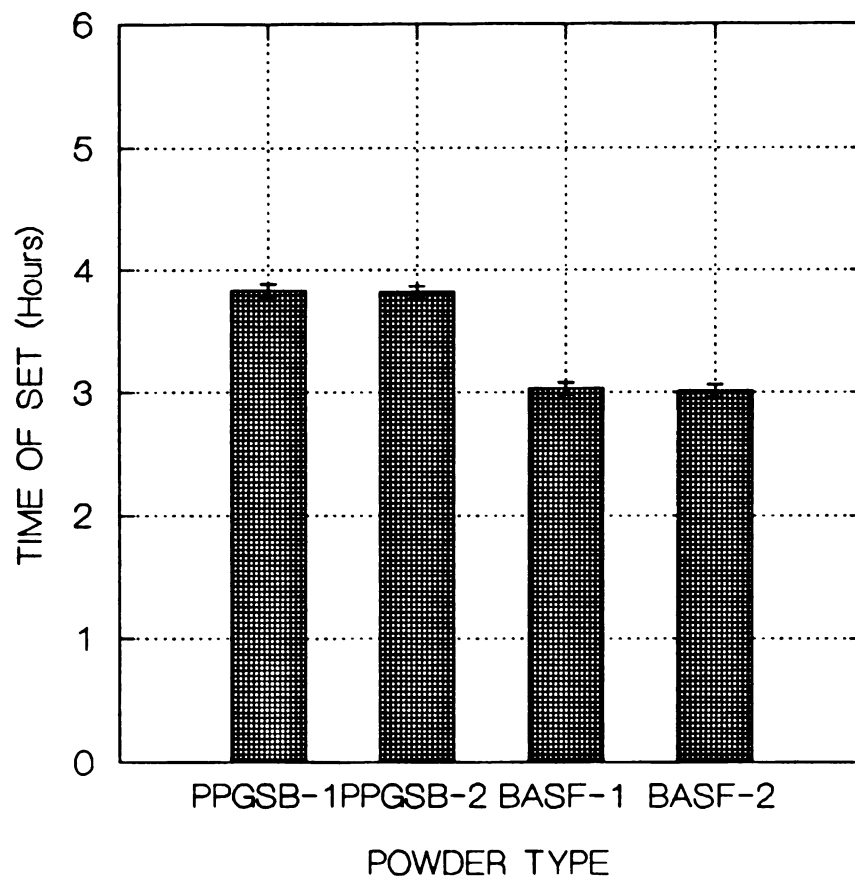


Figure 3.20 (c). Between Shipment Variations in Final Set

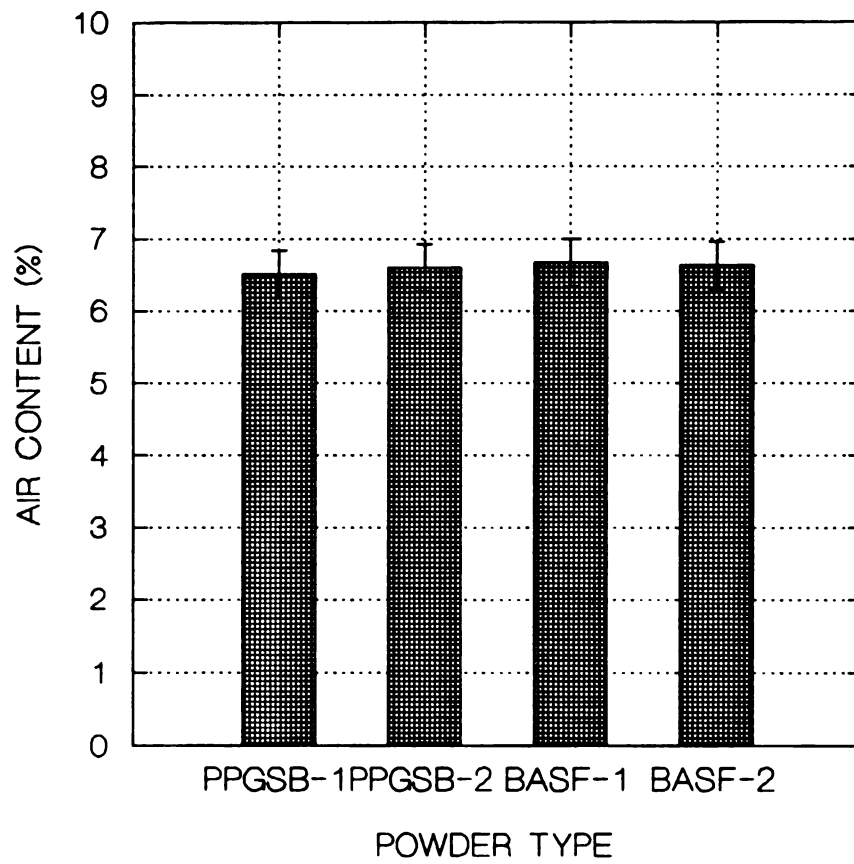


Figure 3.20 (d). Between Shipment Variations in Air Content

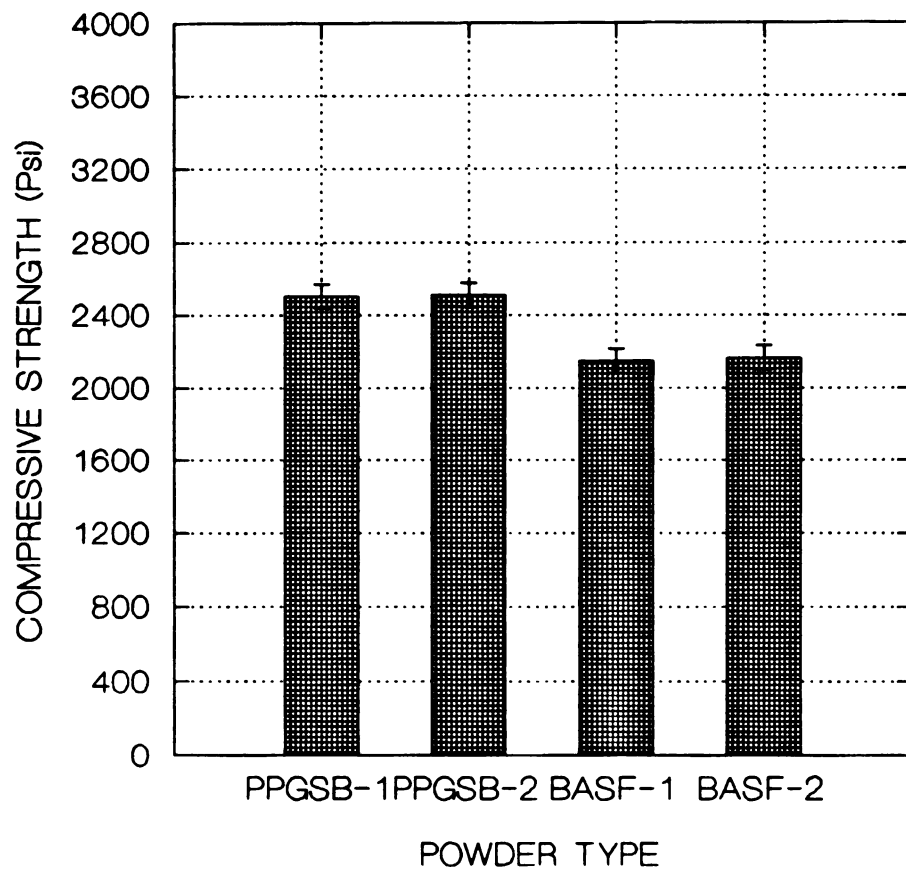


Figure 3.20 (e). Between Shipment Variations in 7-Day Compressive Strength

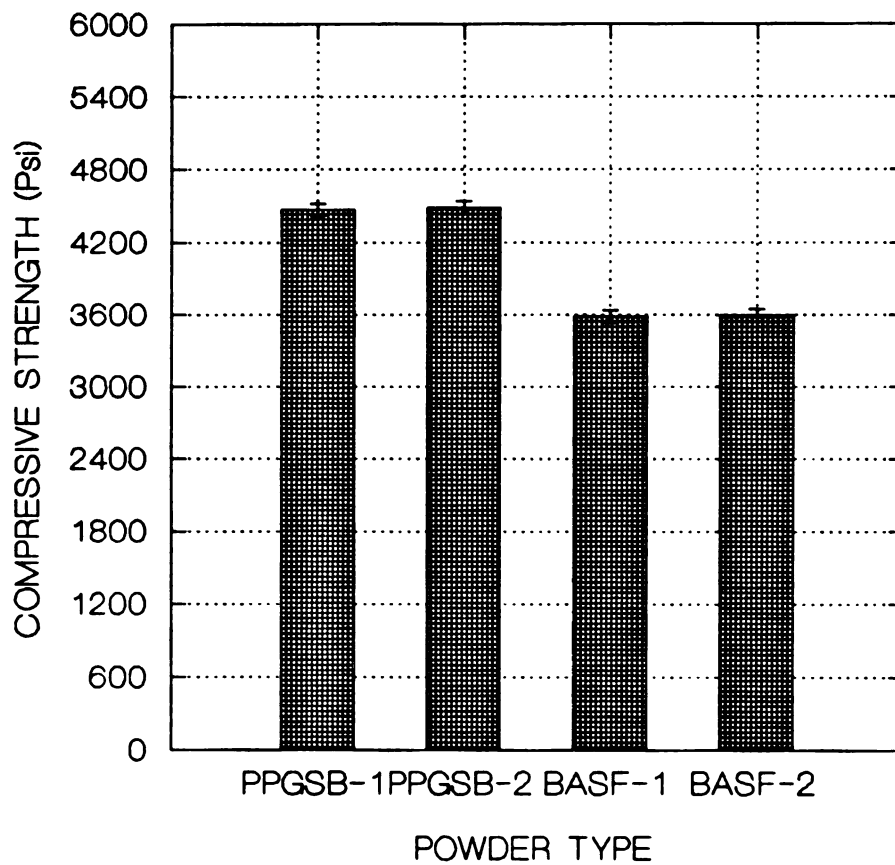


Figure 3.20 (f). Between Shipment Variations in 28-Day Compressive Strength

3.8 SUMMARY AND CONCLUSIONS

Axially restrained expansion tests were conducted on mortar (ASTM C-806) and concrete (ASTM C-878) materials incorporating 0.5%, 1% and 1.5% paint sludge Powders (by weight of cement), and also on expansive cement and conventional cement mortar and concrete materials.

The mortar test results suggest that the PPG Solvent-based (PPG-SB) and PPG Water-Based (PPG-WB) powders at 1% substitution level (by weight of cement) are capable of satisfying the ASTM requirements on the restrained expansion, set time and air content of expansive cement. The increase in dosage from 1% to 1.5% an added safety for satisfying the ASTM requirements on restrained expansion.

ASTM C-878 does not specify any limits on concrete test results. Comparison with the corresponding values for Type K expansive cement thus provided the basis for the evaluation of powders. The powders produced performance characteristic which were essentially similar to those of Type K expansive cement.

It is interesting to note that the restrained expansion test results could reveal differences in the performance of the paint sludge powders which could not be detected through free expansion test results. It seems that the rate of expansion (rather than the total amount of free expansion) is the key factor distinguishing between the effects of different powders in mortar and concrete.

Between-shipment variations in the end product qualities are negligible.

CHAPTER IV

MECHANISM OF EXPANSION ASSOCIATED WITH PAINT SLUDGE POWDER

4.1 INTRODUCTION:

Shrinkage upon drying is a fundamental property of the calcium silicate hydrate which is the principal hydration product of portland cements. Small and medium-size concrete elements are prone to undergo drying shrinkage, and they usually crack to relieve the induced stress when it exceeds the strength of the material. Current design and construction practices assume that concrete will crack, and try to get around the problem in many ways, such as selection and proportioning of concrete mixtures that will shrink less, provision of adequate joints in floor slabs or pavements, and reinforcement of concrete elements with steel. The advent of expansive cement has offered an alternative and cost-effective approach. As a result, since the first industrial use of the shrinkage-compensating concrete in the United States about 25 years ago, the material has found successful application in the construction of a variety of elements, such as floor slabs, pavements, roofs, water storage tanks, and sewage digesters.

The potential expansion associated with ettringite formation is controlled by the use of ordinary steel reinforcement. The steel restrains the overall bulk expansion of the cement, thereby converting expansion into a slight prestress within the concrete. By resisting expansive forces, the steel is placed into tension and the concrete into compression. About 170 to 700 kPa (25 to 100 lb/in.²) compressive stress is generated,

which is generally sufficient to ensure that drying shrinkage will not cause tensile cracking; the concrete either remains in compression or develops only small tensile stresses. Some restraint of expansion may also occur through subgrade friction in slabs or from formwork. The exact degree of prestress will depend on the amount of expansive components and the amount of reinforcement (Figure 4.1). Lack of sufficient restraint will provide little protection against shrinkage cracking, and may, in extreme cases cause the concrete to literally self-destruct; but beyond a certain point additional restraint becomes wasteful. Prestress is developed only in the direction and vicinity of the reinforcing steel. Thus, correct positioning of the steel is important to provide correct restraint, and misplaced reinforcement could lead to lack of adequate prestress or complications such as warping due to differential expansions.

The amount of expansion before drying depends on the amount of expansive component, which should be chosen to ensure that the concrete will remain under a slight compression even after extended drying. Some expansion should be allowed to occur in order to develop maximum residual prestress. It should be emphasized that the development of adequate prestress is the key to shrinkage compensation. Concretes made with expansive cements shrink as much, if not more, than ordinary portland cement concretes, but the net tensile stress generated is much less because of the initial compressive prestress.

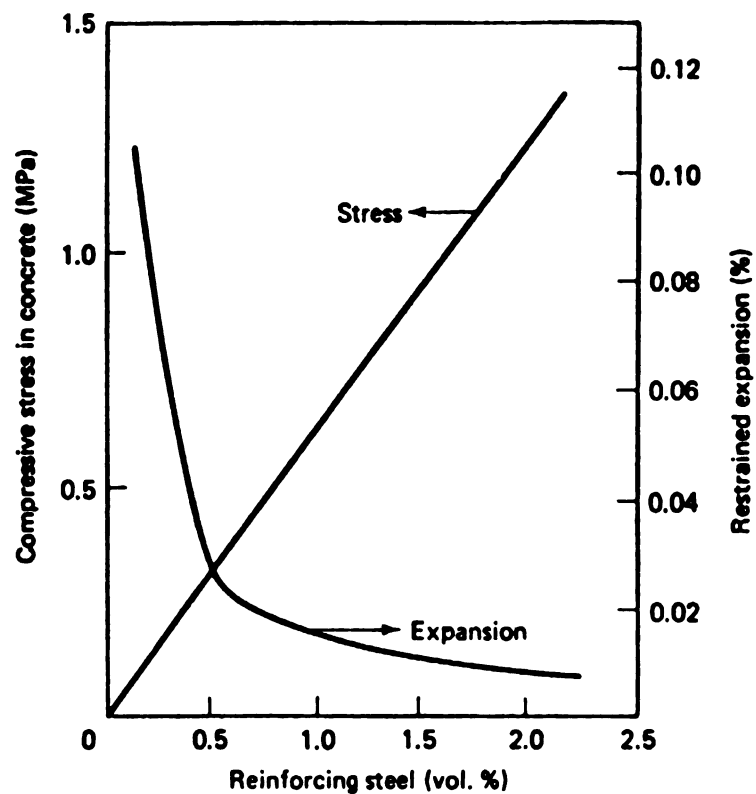


Figure 4.1. Effects of Reinforced Steel on Expansion of Concrete and Prestress Developed. [10]

4.2 CONTROL OF EXPANSION

Successful use of expansive cements depends upon proper control of the expansion of cement during hydration and is sensitive to a number of variables (Table 4.1).

The amount of ettringite formed is controlled by adjusting the cement content or varying the amount of expansive admixture (if one is being used). This assumes that adequate moist curing is provided during the time when ettringite is forming during the

first 7 days. Moist curing is even more critical than it is for ordinary Portland cement concrete and requires the supply of additional water for hydration during the curing period, because the formation of ettringite combines large amounts of water, which can not be completely provided by the mixing water. This is well illustrated in Figure 4.2 which compares the efficiencies of various standard curing procedures.

There are other factors influencing expansion that affect the amount of useful ettringite that is formed. Ettringite can only contribute to an overall volume expansion, and hence to prestress, when it is formed after the concrete has gained rigidity through hydration of C_3S . Any ettringite that forms while the concrete is still plastic is of little value.

At 38 °C (100.4 °F) hydration proceeds faster than at 21 °C (69.8 °F), and thus the rate of expansion is faster, but ultimate expansion is lower because more ettringite is formed when the concrete is still plastic. The reduction in expansion that occurs with an increase in cement fineness is also a result of the faster initial rate of hydration. An increase in water/cement (w/c) ratio decreases the amount of expansion. Admixtures may also interfere with the initial formation of ettringite and hence expansion. The effect of a particular admixture cannot be predicted; thus, tests should be run when admixtures are to be used.

Table 4.1. Variables Affecting Expansive Cements [32]	
General Category	Specific Variables
Reinforcement	Amount of steel; positioning of rebars.
Mix design	Cement Content; water/cement ratio; fineness of cement; admixtures.
Handling and curing	Time of mixing, handling, and placing; time of moist curing; temperature of curing.

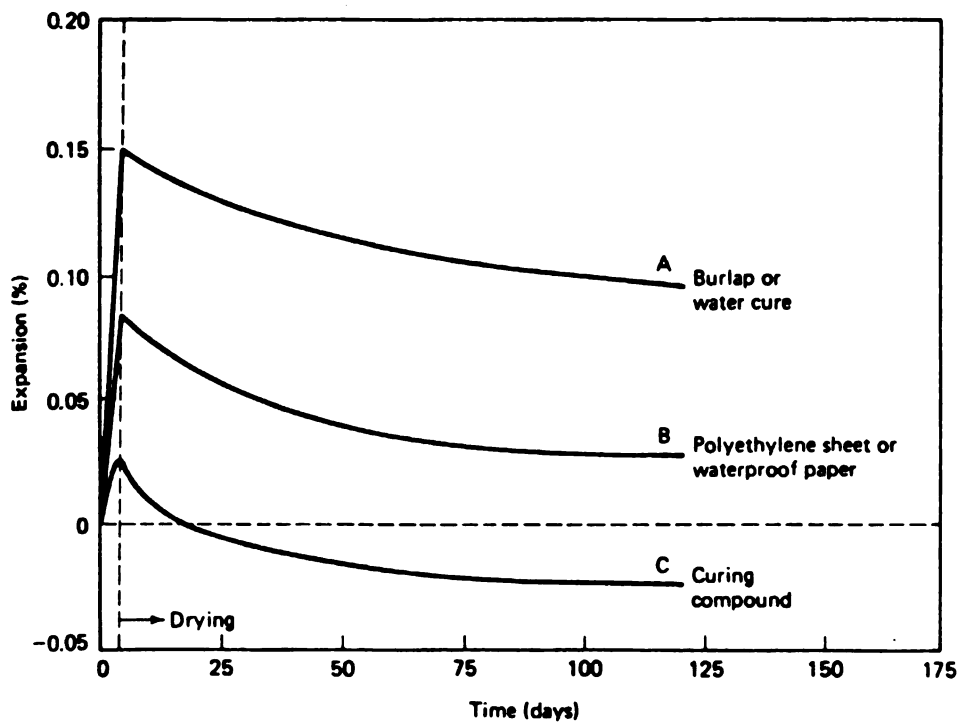


Figure 4.2. Effect of Different Curing Procedures on the Expansion of Type M Expansive Cement Concrete. [10]

4.3 POWDER CHEMISTRY

Inductively coupled plasma-atomic emission spectroscopy (ICAP) test procedure was used in this part to produce comprehensive information on the chemical constituents of the paint Sludge powders.

Table 4.3 presents the ICAP test results. The elements (e.g., Ca, Fe) shown in this Table may occur in the form of different compounds. A preliminary evaluation of Table 4.3 indicates that calcium (compounds) seems to dominate the chemistry of the powders, especially those producing satisfactory expansive qualities (i.e., PPG-WB and PPG-SB). This may indicate that the powder expansion mechanism differs from ettringite formation (generally used in conventional expansive cement) where, besides calcium, one needs sources of Aluminum and Sulfate compounds. In order to further understand the mechanism of expansion, correlations were drawn between restrained expansions and various chemical qualities of different powders.

The results of correlation analysis presented in Figure 4.3 indicates that a very strong positive correlation exists only between calcium content and 7-day restrained expansion. Increased sulfur and aluminum contents of powders do not cause any significant increase in restrained expansion. In Table 4.2, [PPGSB-1 = Shipment 1, PPGSB-2 = Shipment 2, BASF-1 = Shipment 1 and BASF-2 = Shipment 2].

Table 4.2. Chemical Analysis of the Chrysler Paint Sludge Powders

Chemical Contents	PPGSB		BASF		PPGWB	PPGSWB
	PPGSB-1	PPGSB-2	BASF-1	BASF-2		
B	92.7	<50.0	118	<50.0	77.0	<50.2
S	4530	1290	2040	4810	1520	1470
Co	<2.49	<5.0	32.5	<5.0	<2.49	13.6
Mo	<9.98	161	13.0	20.7	<9.96	<10.0
Sb	<25.0	<50.0	<25.0	<50.0	<24.9	<25.1
Hg	<25.0	<50.0	<25.0	<50.0	<24.9	<25.1
Na	2870	666	1080	1290	2910	<502
Ba	250	201	623	508	191	397
Fe	3860	3040	19400	4270	3850	11500
P	102	133	348	245	119	123
As	<25.0	<25.0	54.0	<25.0	<24.9	<25.1
Pb	<25.0	<25.0	<25.1	<25.0	<24.9	<25.1
K	192	494	4980	424	220	1520
Ca	44000	40800	5780	20100	61500	2350
Mg	2190	858	2150	2630	2710	718
Zn	157	260	49.5	148	231	19.2
Cr	9.82	<10.0	47.0	26.1	9.19	20.4
Se	<200	<200	<200	<200	<199	<201
Ni	29.7	23.7	53.9	55.2	26.7	24.8
Cu	778	619	88.7	486	688	33.0
Mn	<2.49	18.7	106	76.7	78.9	70.1
Al	34100	20300	21900	34200	30700	8110
Cd	<2.49	<5.00	<2.51	<5.00	<2.49	<2.51
Tl	<2.49	<125	<50.1	<125	<49.8	<50.2

** All values are in ppm **

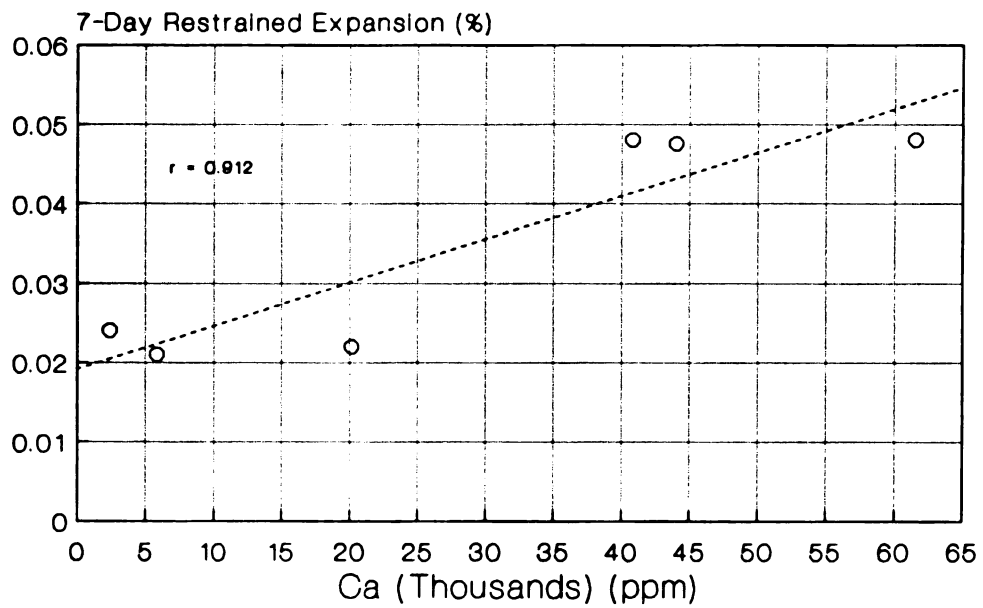


Figure 4.3 (a). Correlations Between Powder Chemistry (Calcium Content, Ca) and 7-Day Restraint Expansion.

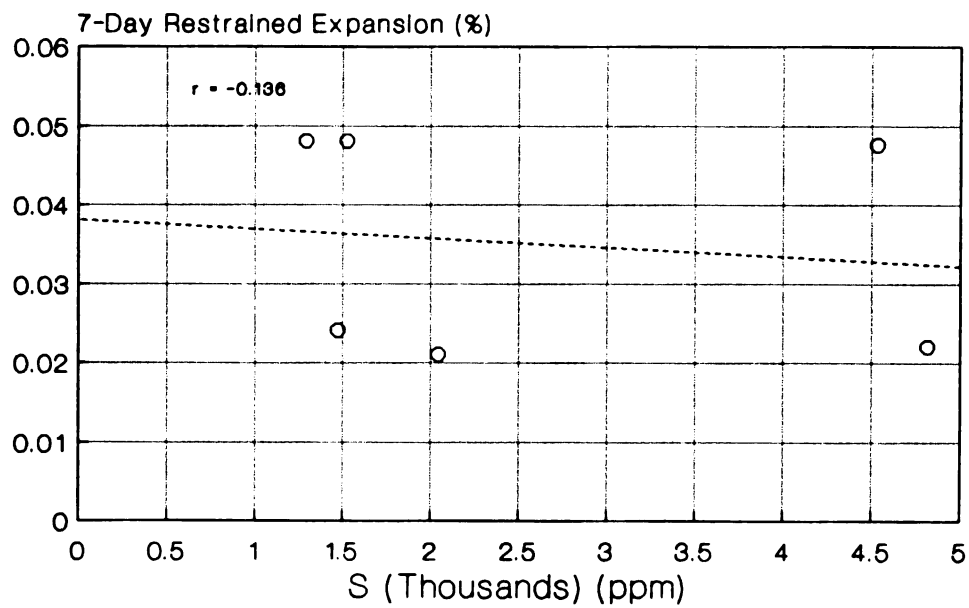


Figure 4.3 (b). Correlations Between Powder Chemistry (Sulfur Content, S) and 7-Day Restraint Expansion.

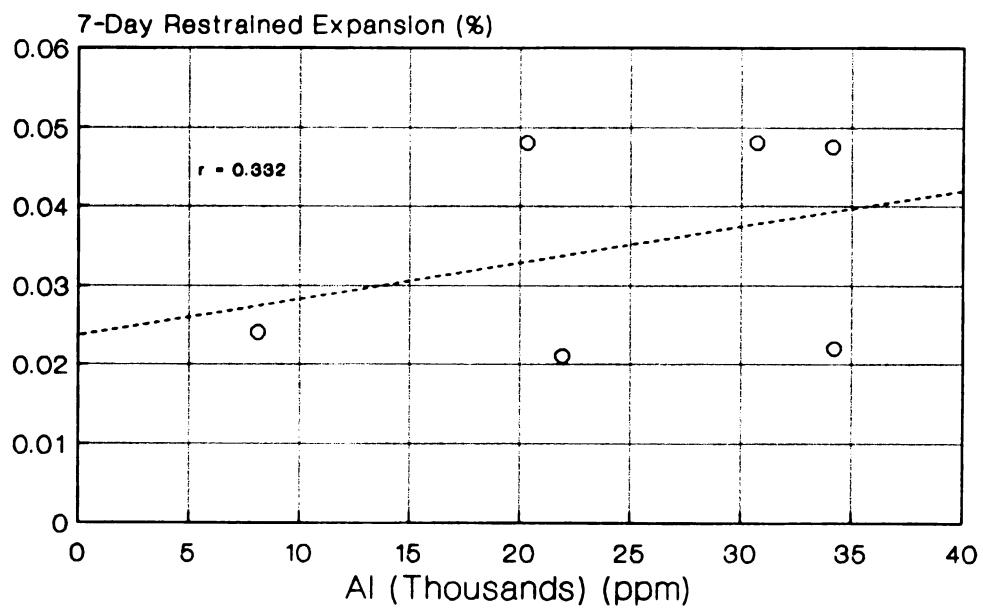


Figure 4.3 (c). Correlations Between Powder Chemistry (Aluminum Content, Al) and 7-Day Restraint Expansion.

The results presented in Figure 4.3 suggests that one may find the mechanism of expansion in calcium (compounds) present in the powders. There are two potential calcium-related expansion mechanisms in concrete; hydration of hard-burnt calcium oxide, and reactions of calcium carbonate with the alkalies. If calcium occurs in the form of calcium oxide, then hydration of hard-burnt CaO may cause expansion, while if calcium occurs in the form of calcium carbonate, then alkali-carbonate reaction may dominate the expansion process. In order to find out the source of calcium, mineralogical analysis of the powders were undertaken.

4.4 POWDER MINERALOGY

X-Ray Diffraction (XRD) techniques were used to determine the compounds, containing calcium in the sludge powders. Two sources of calcium were examined: Calcite (Calcium Carbonate) represented by Cc and Lime (CaO) represented by Lm. The XRD results presented in Figure 4.4 suggest that calcium occurs mainly in the form of calcite, and thus it may be hypothesized that alkali-carbonate reaction and not the hydration of hard-burnt CaO may produce the expansion qualities of powders. In these Figures, [Et=Ettringite, Gp=Gypsum, PL=Portlandite, Lm=Lime, Cc=Calcite, Ah=Anhydrite and Qz=Quartz].

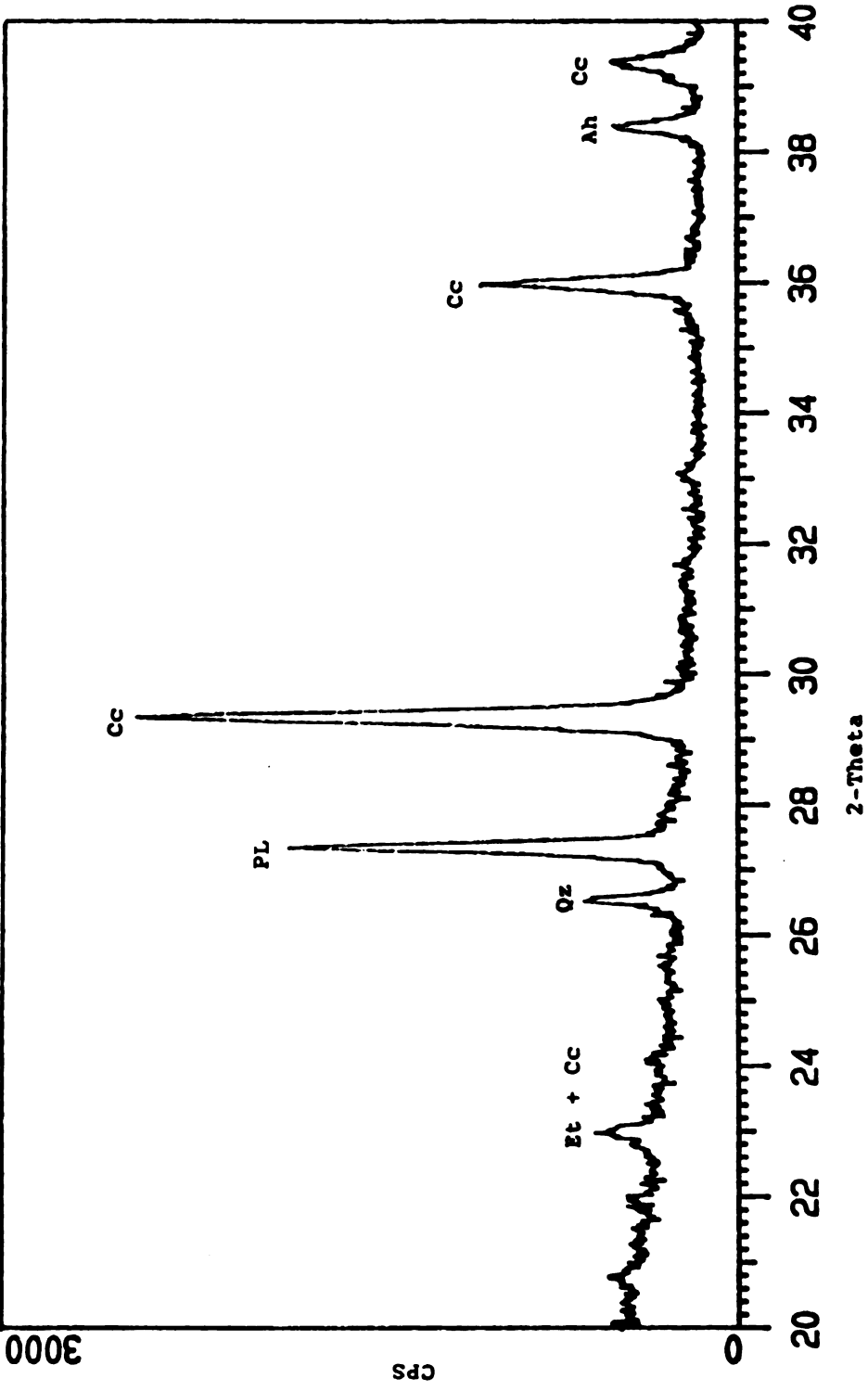


Figure 4.4 (a). Powder X-Ray Diffraction Results (PPG-SB)

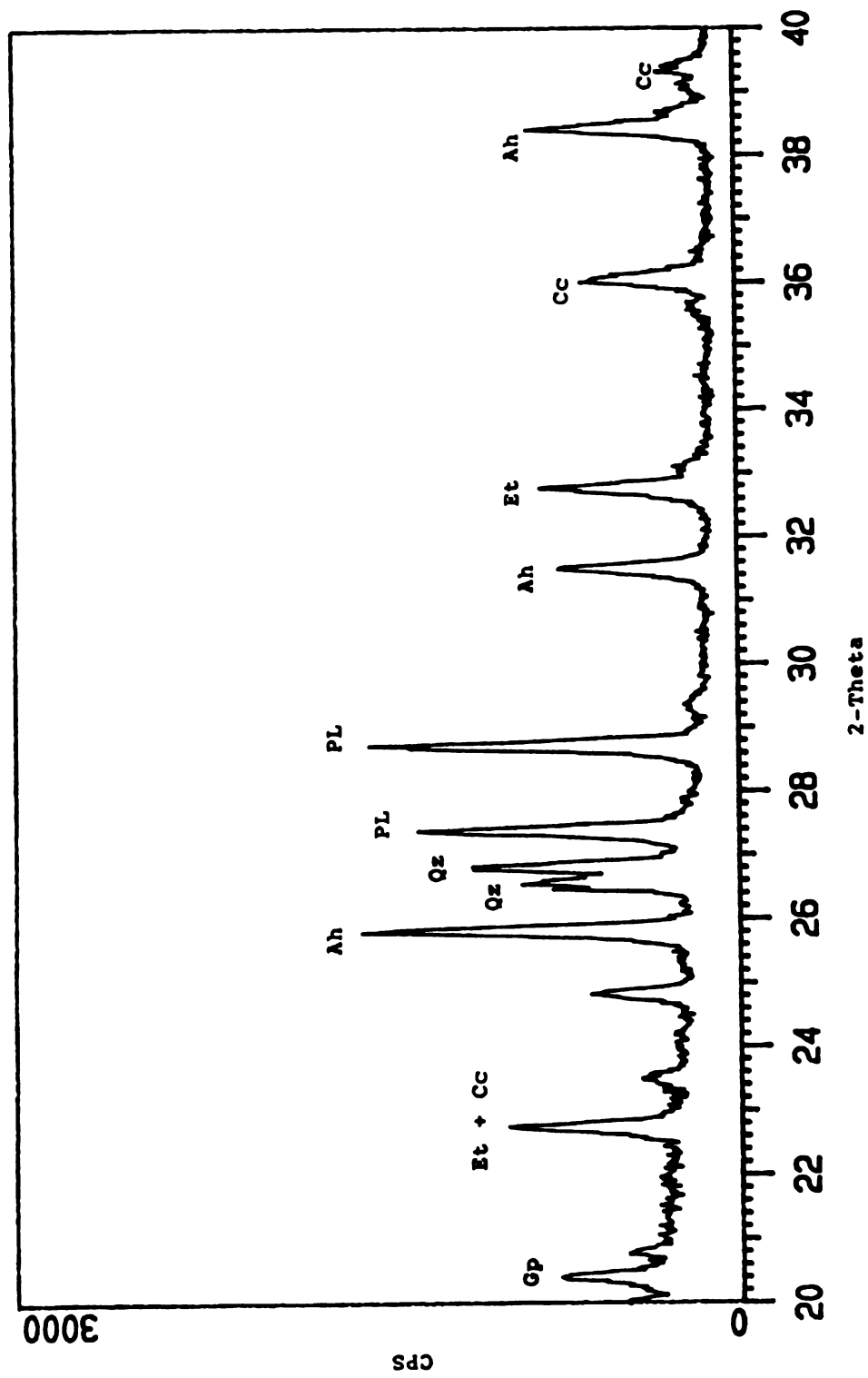


Figure 4.4 (b). Powder X-Ray Diffraction Results (BASF)

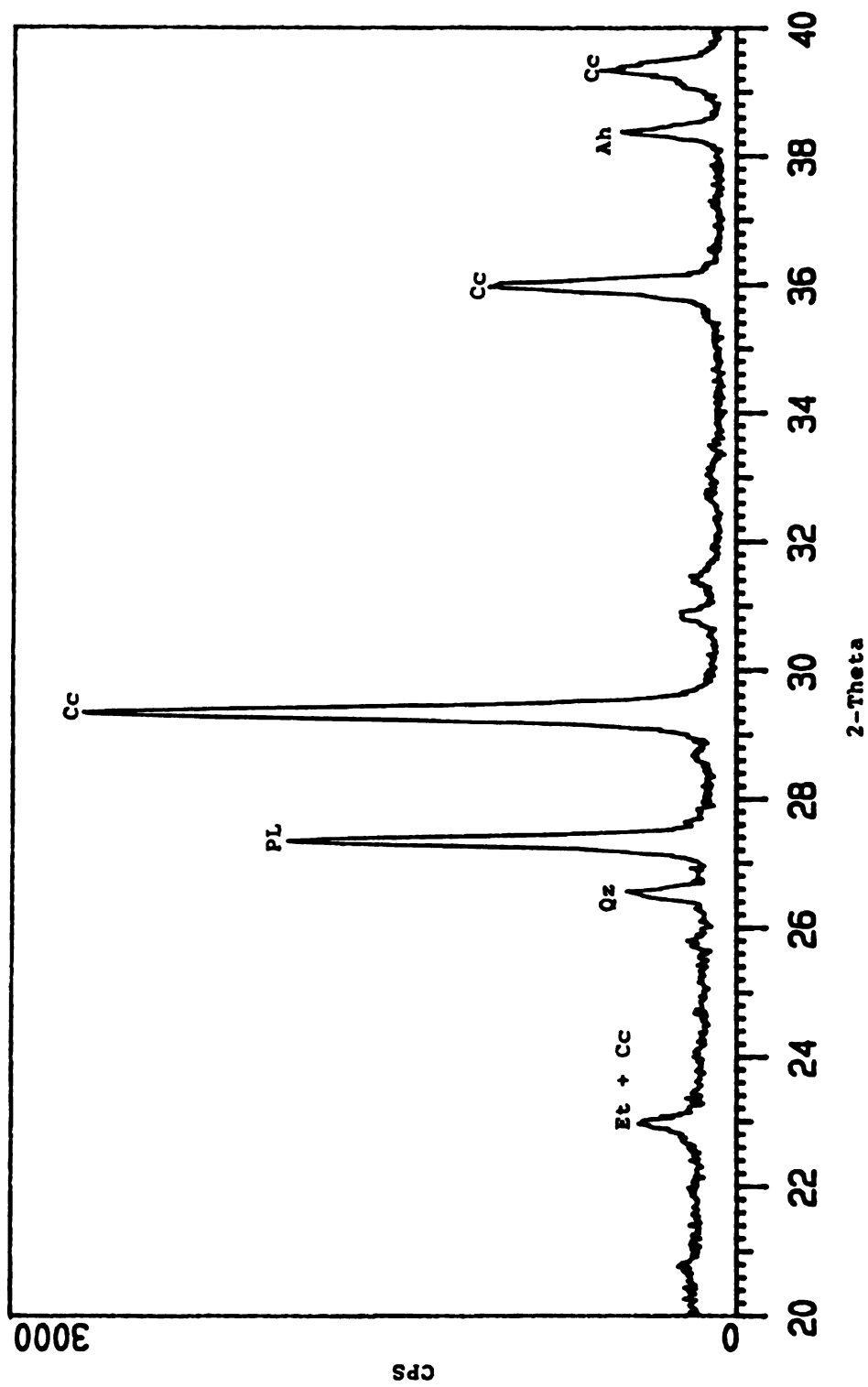


Figure 4.4 (c). Powder X-Ray Diffraction Results (PPG-WB)

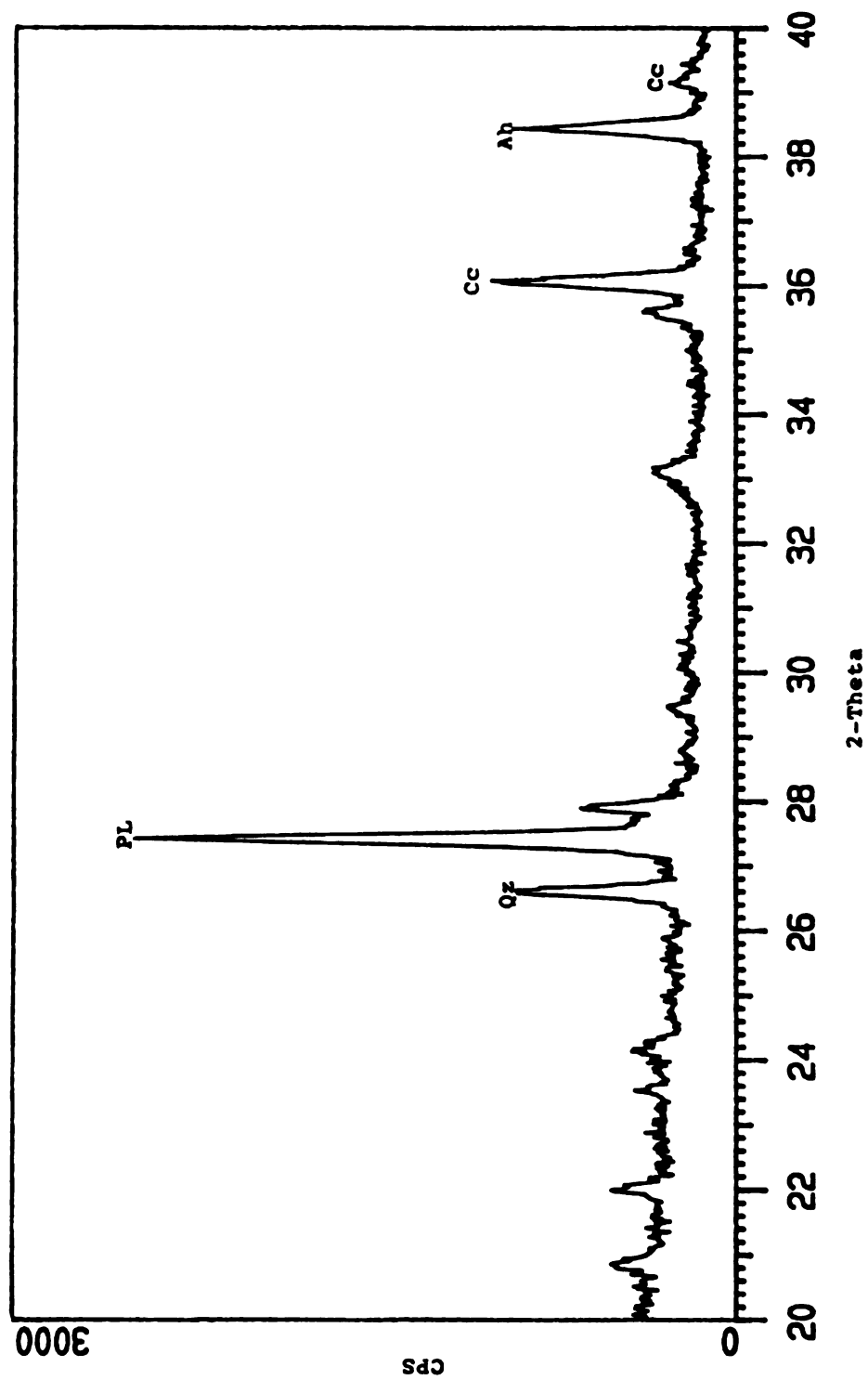


Figure 4.4 (d). Powder X-Ray Diffraction Results (PPG-SWB)

4.5 CONCRETE MINERALOGY

The concrete X-Ray Diffraction (XRD) test results presented in Figure 4.5 suggest that the powders producing satisfactory restrained expansion (PPG-SB and PPG-WB) also produced relatively high concentrations of Portlandite [$\text{Ca}(\text{OH})_2$] in hardened concrete. This observation complicates the picture because one may question whether the hydration of hard-burnt CaO is producing this excess calcium hydroxide. The process of alkali-carbonate reaction is not well understood; however, the Portlandite could also result from alkali-carbonate reaction. In these Figures, [Et=Ettringite, Gp=Gypsum, PL=Portlandite, Lm=Lime, Cc=Calcite, Ah=Anhydrite and Qz=Quartz].

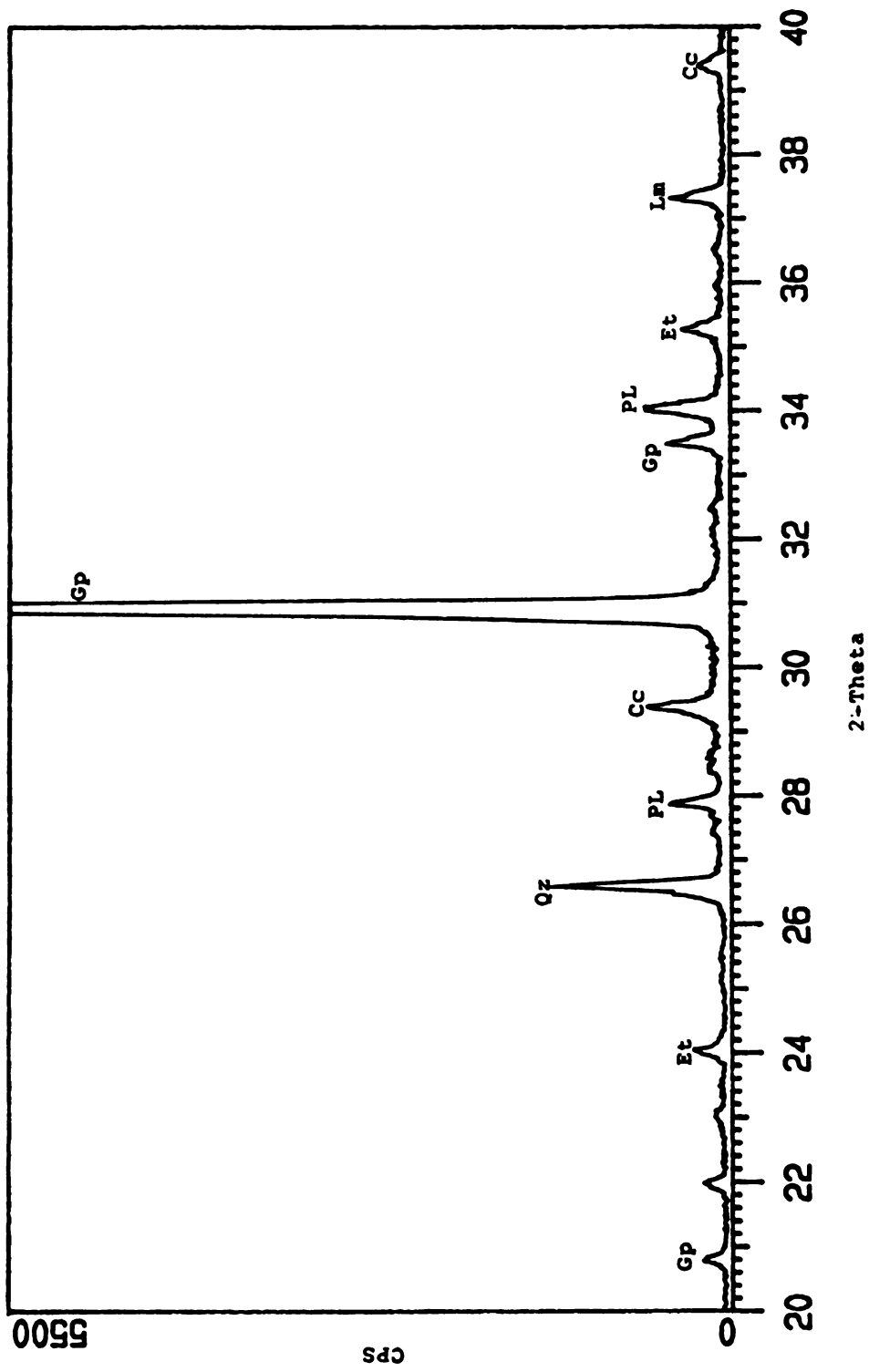


Figure 4.5 (a). Concrete X-Ray Diffraction Results (PPG-SB)

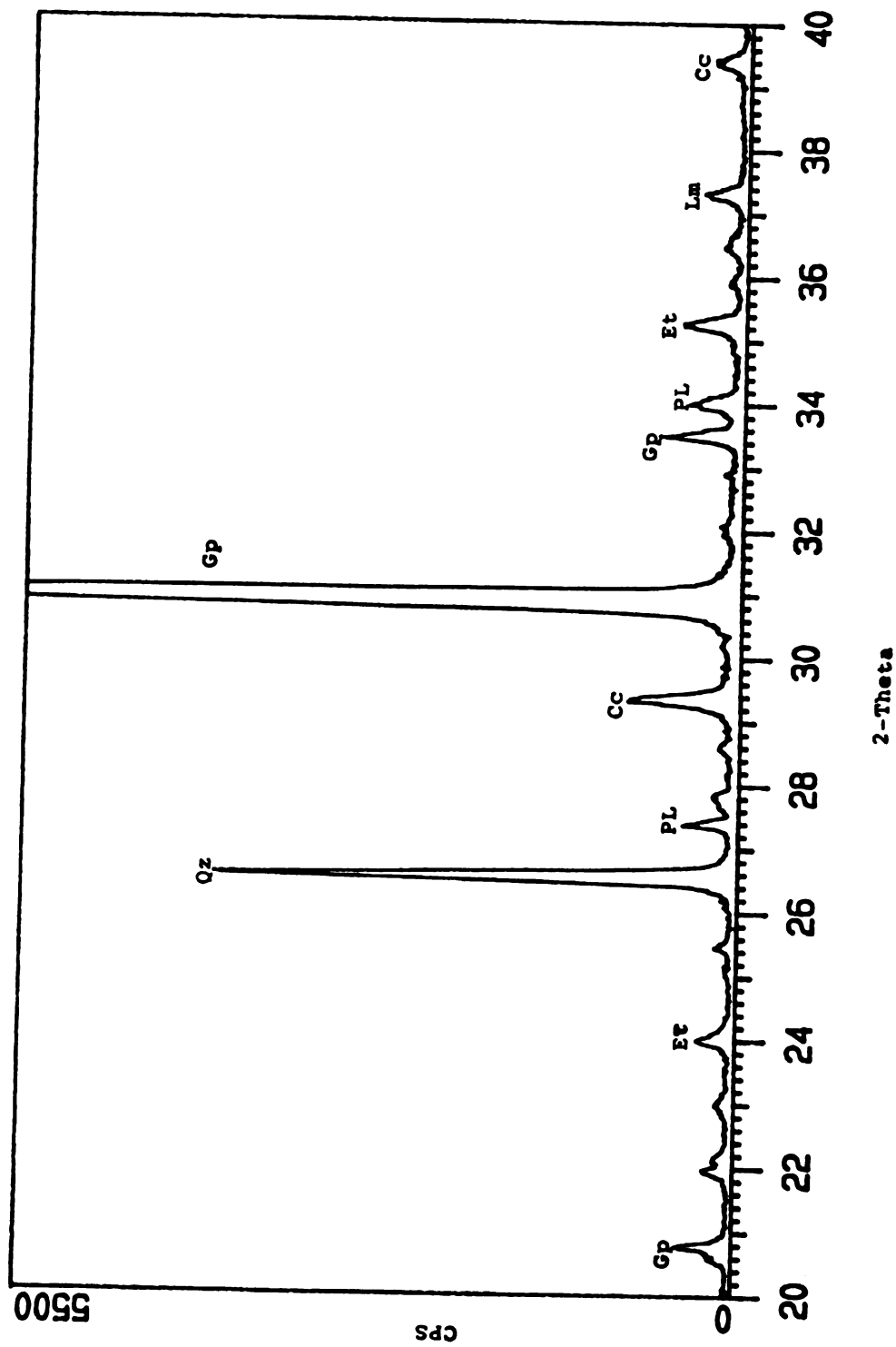


Figure 4.5 (b). Concrete X-Ray Diffraction Results (BASF)

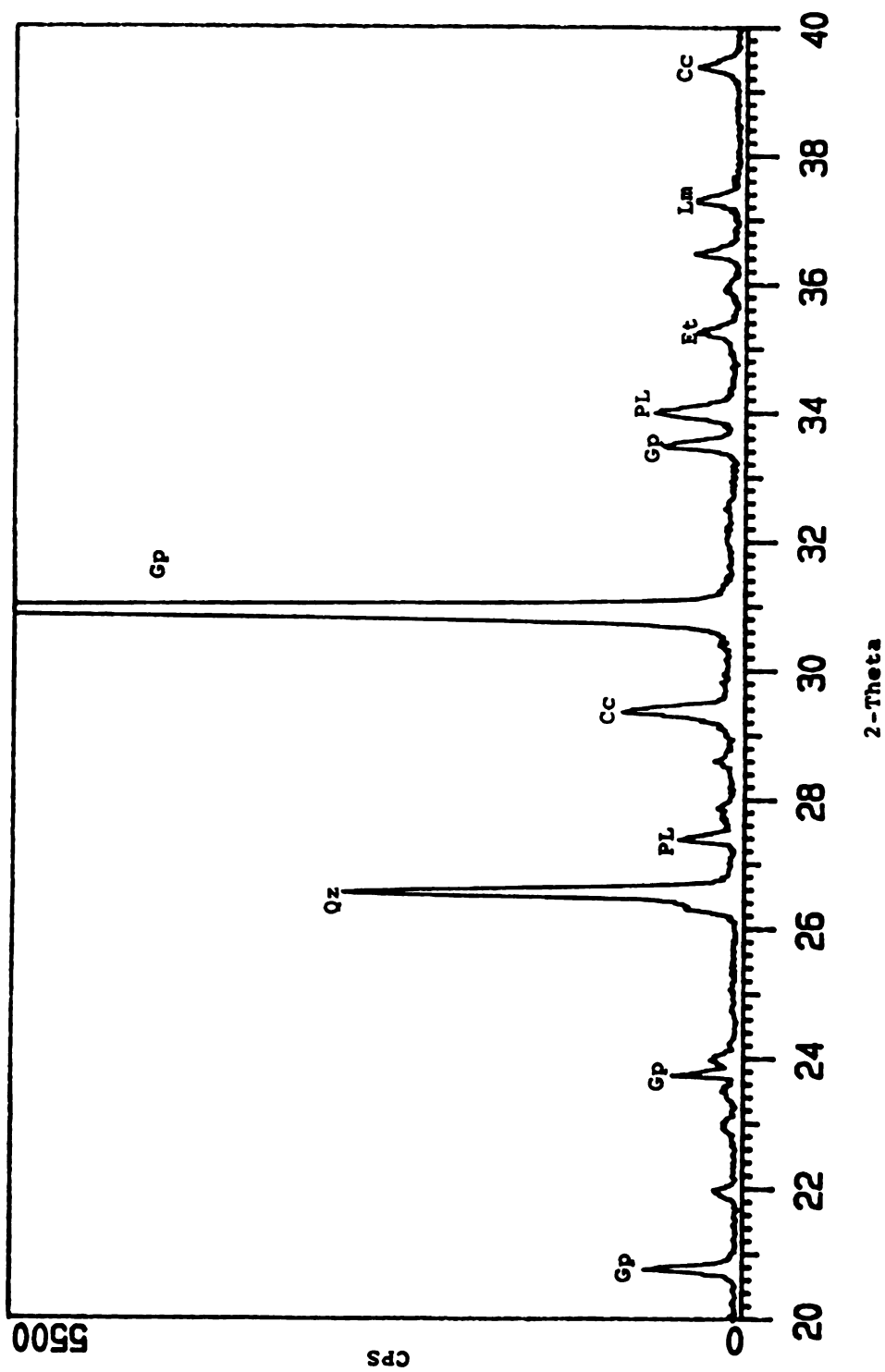


Figure 4.5 (c). Concrete X-Ray Diffraction Results (PPG-WB)

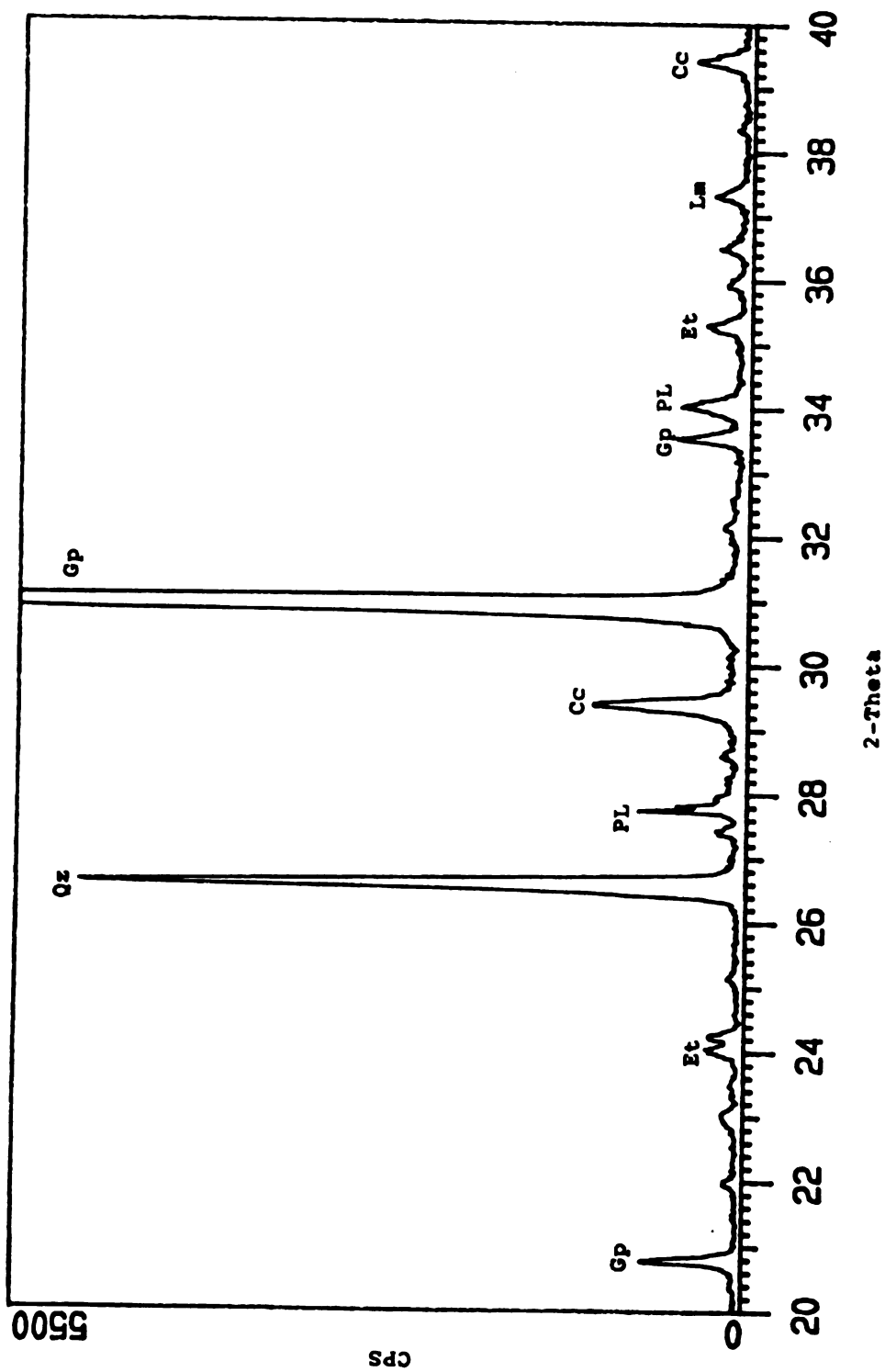


Figure 4.5 (d). Concrete X-Ray Diffraction Results (PPG-SWB)

4.6 ASSESSMENT OF CALCIUM CARBONATE REACTION WITH CEMENT ALKALIS AS THE CAUSE OF EXPANSION.

In order to determine if reaction of calcite with Cement alkalis is the cause of expansion, cements of low and high alkali contents were used in conjunction with the powders in restrained expansion tests. The low-alkali cement used had a total Na_2O equivalent alkali content of 0.53% and the one with high alkali content had a total Na_2O equivalent alkali content of 1.21%. The 7-day restrained expansion test results are presented in Figure 4.6.

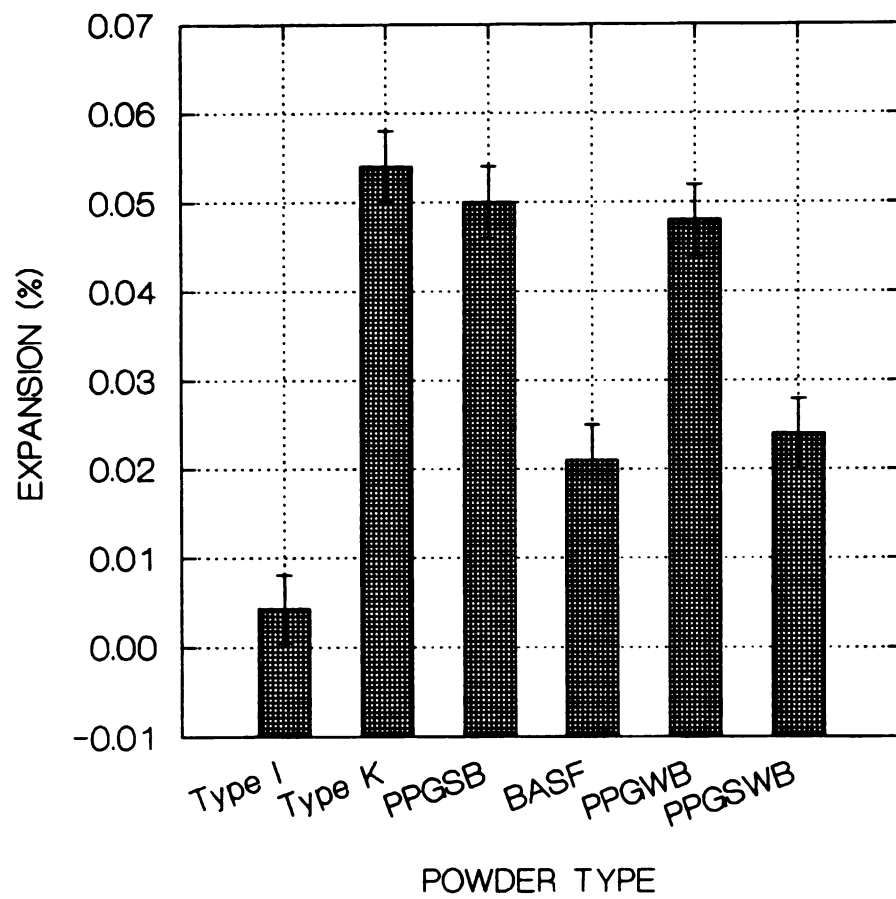


Figure 4.6 (a). Effects of Cement With Low Alkali Content on 7-Day Restrained Expansion.

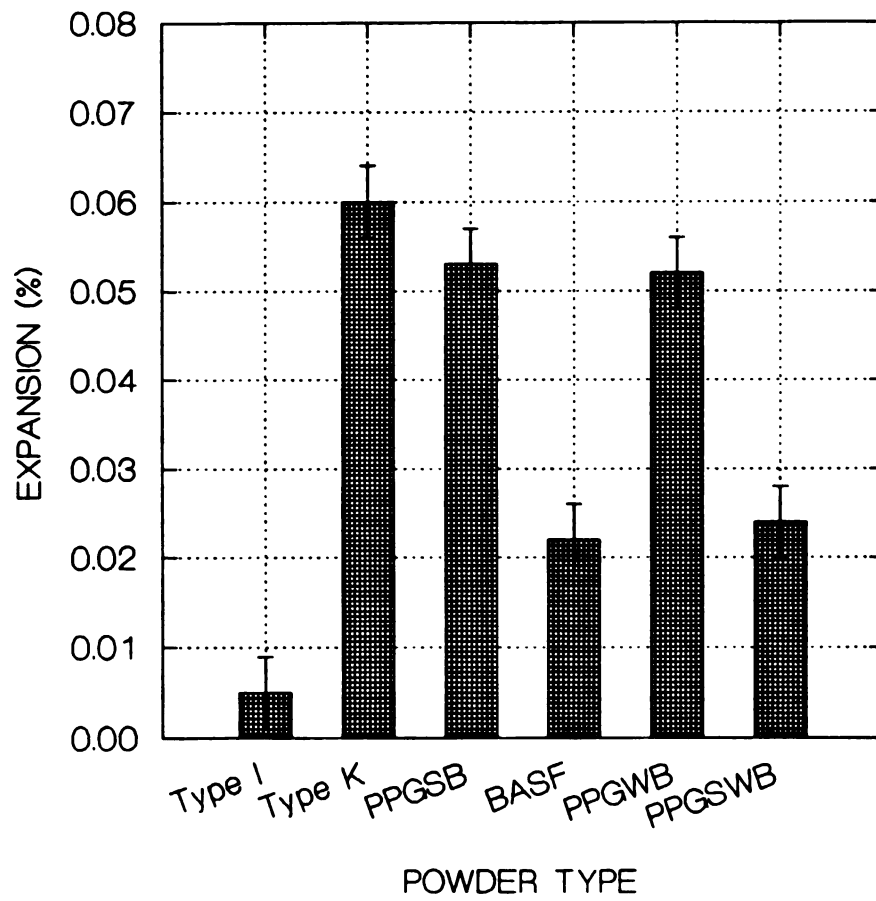


Figure 4.6 (b). Effects of Cement With High Alkali Content on 7-Day Restrained Expansion.

One may conclude from Figure 4.6 that the increase in cement alkali content leads to somewhat increased restrained expansion. This increase is relatively small but may be used to support the hypothesis that the reaction of cement alkalis with calcium carbonate may be the source of expansive qualities in paint sludge powders. It is also interesting to note that the powders, even with low-alkali cement, still produce restrained expansions satisfying the minimum ASTM requirements.

4.7 CHEMICAL AND PHYSICAL PROPERTIES OF THE LOW AND HIGH ALKALI CEMENTS

Table 4.3 Chemical and Physical Properties of the Alkali Cement		
Properties	Low-Alkali Cement	High-Alkali-Cement
Silicon Dioxide (SiO_2)	20.61	20.56
Aluminum Oxide (Al_2O_3)	4.77	5.88
Ferric Oxide (Fe_2O_3)	2.20	2.41
Calcium Oxide (CaO)	63.44	63.18
Magnesium Oxide (MgO)	4.19	3.16
Sulfure Trioxide (SO_3)	2.55	3.21
Total Alkalies (as Na_2O)	0.53	1.21
Fineness (% Retained on #325 Sieve)	11.0	10.8
Specific Gravity	3.15	3.15
Loss on Ignition (LOI)	1.18	1.63

4.8 CONCLUSION

Chemical and mineralogical analysis of the powders resulting concretes were undertaken in order to determine the sources of expansive qualities in powders. Chemical analysis indicated that calcium compounds dominate the chemistry of powders, and those powders with higher calcium compound contents produced larger restrained expansions. Calcium may produce expansion through hydration of hard-burnt calcium oxide (when occurring as CaO) or reaction with cement alkalis (when occurring as CaCO_3). Mineralogical analyses indicated that calcium in powders occurs dominantly in the form of CaCO_3 , suggesting that reaction of calcium carbonate with cement alkalis could be the cause of expansion; the fact that higher-alkali cements produced somewhat higher expansions with these powders further supported this hypothesis. Mineralogical analysis of concrete suggested that powders with higher restrained expansive qualities also produced higher calcium hydroxide contents in hardened concrete.

CHAPTER V

EFFECTS OF MIX VARIABLES

5.1 INTRODUCTION

A new concrete admixture should be ideally applicable to concrete mixtures with different constituents and proportions. The main objective of this phase of research was to determine if any limits exist as far as the concrete mix constituents and proportions are concerned for the paint sludge powder to be effective as an expansive admixture.

5.2 EXPERIMENTAL PROGRAM

The effects of the following variables on the restrained expansion and other qualities of concrete incorporating paint sludge powder were determined.

- * Powder type (PPG solvent-based vs. PPG water-based)
- * Powder content (1% vs. 1.5% powder-cement ratio)
- * Cement type (St. Mary's Type I vs. Lafarge Type I)
- * Aggregate content (4.0 vs. 4.5 aggregate-cement ratio)
- * Water-cement ratio (0.45 vs. 0.55)
- * Air entraining agent content (0.05% vs. 0.1% ratio of air entraining agent to cement).

A $1/2$ fractional factorial design of experiments was conducted in order to determine the effects and the first-level interaction of the above variables in determining the restrained expansive and other qualities of concrete materials. Table 5.1 presents the fractional factorial designs of experiments. The averages of three tests on specimens from same batch was used as the test result for each condition of Table 5.1. The experiments were conducted in the random order presented in the Table.

Table 5.1. Fractional Factorial Design of Experiments.								
Run Order	1	2	3	4	5	6	7	8
Block	1	1	1	1	1	1	1	1
Standard Order	2	13	12	22	8	20	15	1
VARIABLES								
A=Powder Type	b	b	a	b	b	b	a	a
B=Powder/Cement Ratio	1.0	1.0	1.5	1.0	1.5	1.5	1.5	1.0
C=Cement Type	SM	LF	SM	LF	LF	SM	LF	SM
D=Aggregate/Cement Ratio	4.0	4.5	4.5	4.0	4.0	4.0	4.5	4.0
E=Water/Cement Ratio	.45	.45	.45	.55	.45	.55	.45	.45
F=Air-Entraining Agent/Cement Ratio	.10	.05	.10	.10	.10	.10	.10	.05
RESPONSE								
R1=Set Time (Hours)								
R2=Slump (inches)								
R3=Air Content (%)								
R4=Unit Weight (Pcf)								
R5=Flexure (Ksi)								
R6=Compression (Ksi)								
R7=7-Day Expansion (%)								
R8=28-Day Expansion (%)								

A to F = Variables

PPG-SB = a

St. Mary's = SM

R1 to R8 = Response

PPG-WB = b

Lafarge = LF

Table 5.1. Fractional Factorial Design of Experiments (Cont'd).								
Run Order	9	10	11	12	13	14	15	16
Block	1	1	1	1	1	1	1	1
Standard Order	4	5	25	32	11	24	6	28
VARIABLES								
A=Powder Type	b	a	a	b	a	b	b	b
B=Powder/Cement Ratio	1.5	1.0	1.0	1.5	1.5	1.5	1.0	1.5
C=Cement Type	SM	LF	SM	LF	SM	LF	LF	SM
D=Aggregate/Cement Ratio	4.0	4.0	4.5	4.5	4.5	4.0	4.0	4.5
E=Water/Cement Ratio	.45	.45	.55	.55	.45	.55	.45	.55
F=Air-Entraining Agent/Cement Ratio	.05	.10	.05	.10	.05	.05	.05	.05
RESPONSE								
R1=Set Time (Hours)								
R2=Slump (inches)								
R3=Air Content (%)								
R4=Unit Weight (Pcf)								
R5=Flexure (Ksi)								
R6=Compression (Ksi)								
R7=7-Day Expansion (%)								
R8=28-Day Expansion (%)								

A to F = Variables

PPG-SB = a

St. Mary's = SM

R1 to R8 = Response

PPG-WB = b

Lafarge = LF

Table 5.1. Fractional Factorial Design of Experiments (Cont'd).								
Run Order	17	18	19	20	21	22	23	24
Block	1	1	1	1	1	1	1	1
Standard Order	31	29	21	30	23	10	26	19
VARIABLES								
A=Powder Type	a	a	a	b	a	b	b	a
B=Powder/Cement Ratio	1.5	1.0	1.0	1.0	1.5	1.0	1.0	1.5
C=Cement Type	LF	LF	LF	LF	LF	SM	SM	SM
D=Aggregate/Cement Ratio	4.5	4.5	4.0	4.5	4.0	4.5	4.5	4.0
E=Water/Cement Ratio	.55	.55	.55	.55	.55	.45	.55	.55
F=Air-Entraining Agent/Cement Ratio	.05	.10	.05	.05	.10	.05	.10	.05
RESPONSE								
R1=Set Time (Hours)								
R2=Slump (inches)								
R3=Air Content (%)								
R4=Unit Weight (Pcf)								
R5=Flexure (Ksi)								
R6=Compression (Ksi)								
R7=7-Day Expansion (%)								
R8=28-Day Expansion (%)								

A to F = Variables

PPG-SB = a

St. Mary's = SM

R1 to R8 = Response

PPG-WB = b

Lafarge = LF

Table 5.1. Fractional Factorial Design of Experiments (Cont'd).								
Run Order	25	26	27	28	29	30	31	32
Block	1	1	1	1	1	1	1	1
Standard Order	17	7	16	3	27	14	18	9
VARIABLES								
A=Powder Type	a	a	b	a	a	b	b	a
B=Powder/Cement Ratio	1.0	1.5	1.5	1.5	1.5	1.0	1.0	1.0
C=Cement Type	SM	LF	LF	SM	SM	LF	SM	SM
D=Aggregate/Cement Ratio	4.0	4.0	4.5	4.0	4.5	4.5	4.0	4.5
E=Water/Cement Ratio	.55	.45	.45	.45	.55	.45	.55	.45
F=Air-Entraining Agent/Cement Ratio	.10	.05	.05	.10	.10	.10	.05	.10
RESPONSE								
R1=Set Time (Hours)								
R2=Slump (inches)								
R3=Air Content (%)								
R4=Unit Weight (Pcf)								
R5=Flexure (Ksi)								
R6=Compression (Ksi)								
R7=7-Day Expansion (%)								
R8=28-Day Expansion (%)								

A to F = Variables

PPG-SB = a

St. Mary's = SM

R1 to R8 = Response

PPG-WB = b

Lafarge = LF

The coarse aggregate used was crushed limestone with 3/4-in (9-mm) maximum aggregate size. The fine aggregate was natural fine sand, and its ratio of fine-to-coarse aggregate was 1.0. The air entraining agent was Vinsol resin-based.

The properties of the two cements used are compared in Table 5.2.

Table 5.2. Properties of Cement Used.		
Properties	Cement Source	
	St. Mary's Type I	Lafarge Type I
Silicon Dioxide (SiO_2)	19.95	20.8
Aluminum Oxide (Al_2O_3)	5.11	4.7
Ferric Oxide (Fe_2O_3)	2.07	2.5
Calcium Oxide (CaO)	63.53	64.5
Magnesium Oxide (MgO)	2.52	1.8
Sulfur Trioxide (SO_3)	4.41	2.6
Total Alkalis (Na_2O)	0.87	0.65
Loss On Ignition (LOI)	1.56	1.7
Specific Gravity	3.15	3.15

Concrete mixtures prepared based on the experimental design of Table 5.1 were subjected to the following tests.

- * 7-Day and 28-Day Restrained Expansion (ASTM C-878)
- * Compressive Strength (ASTM C-39)
- * Flexural Strength (ASTM C-192)
- * Unit Weight (ASTM C-138)
- * Air Content (ASTM C-231)
- * Slump (ASTM C-143)
- * Set Time (ASTM C-403)

5.3 EXPERIMENTAL RESULTS AND ANALYSIS

The experimental results are presented in Table 5.3.

Table 5.3. Results of Fractional Factorial Design of Experiments.								
Run Order	1	2	3	4	5	6	7	8
Block	1	1	1	1	1	1	1	1
Standard Order	2	13	12	22	8	20	15	1
VARIABLES								
A = Powder Type	b	b	a	b	b	b	a	a
B = Powder/Cement Ratio	1.0	1.0	1.5	1.0	1.5	1.5	1.5	1.0
C = Cement Type	SM	LF	SM	LF	LF	SM	LF	SM
D = Aggregate/Cement Ratio	4.0	4.5	4.5	4.0	4.0	4.0	4.5	4.0
E = Water/Cement Ratio	.45	.45	.45	.55	.45	.55	.45	.45
F = Air-Entraining Agent/Cement Ratio	.10	.05	.10	.10	.10	.10	.10	.05
RESPONSE								
R1 = Set Time (Hours)	4.1	3.9	3.9	4.0	3.9	3.8	3.9	4.2
R2 = Slump (inches)	3.1	2.9	2.1	2.8	3.0	2.6	2.4	2.9
R3 = Air Content (%)	12	6.0	13	15	12	13	12	6.3
R4 = Unit Weight (Pcf)	338	340	335	330	340	333	336	344
R5 = Flexure (Ksi)	.46	.62	.50	.62	.47	.63	.47	.62
R6 = Compression (Ksi)	3.6	4.4	3.8	3.8	3.8	4.0	3.8	3.9
R7 = 7-Day Expansion (%)	.04	.040	.043	.04	.04	.05	.05	.043
R8 = 28-Day Expansion (%)	.04	.042	.048	.04	.05	.05	.05	.044

A to F = Variables

PPG-SB = a

St. Mary's = SM

R1 to R8 = Response

PPG-WB = b

Lafarge = LF

Table 5.3. Results of Fractional Factorial Design of Experiments (Cont'd).								
Run Order	9	10	11	12	13	14	15	16
Block	1	1	1	1	1	1	1	1
Standard Order	4	5	25	32	11	24	6	28
VARIABLES								
A=Powder Type	b	a	a	b	a	b	b	b
B=Powder/Cement Ratio	1.5	1.0	1.0	1.5	1.5	1.5	1.0	1.5
C=Cement Type	SM	LF	SM	LF	SM	LF	LF	SM
D=Aggregate/Cement Ratio	4.0	4.0	4.5	4.5	4.5	4.0	4.0	4.5
E=Water/Cement Ratio	.45	.45	.55	.55	.45	.55	.45	.55
F=Air-Entraining Agent/Cement Ratio	.05	.10	.05	.10	.05	.05	.05	.05
RESPONSE								
R1=Set Time (Hours)	3.7	4.1	4.0	3.9	4.0	3.9	3.9	3.8
R2=Slump (inches)	2.1	2.8	3.0	2.3	2.6	2.5	2.6	2.5
R3=Air Content (%)	6.5	12	6.0	15	6.3	6.2	6.2	6.0
R4=Unit Weight (Pcf)	344	329	345	330	341	340	341	346
R5=Flexure (Ksi)	.47	.51	.76	.57	.75	.57	.61	.63
R6=Compression (Ksi)	3.8	3.8	4.0	3.6	3.8	3.8	4.0	3.9
R7=7-Day Expansion (%)	.05	.04	.04	.05	.05	.05	.04	.05
R8=28-Day Expansion (%)	.05	.04	.04	.05	.05	.05	.04	.05

A to F = Variables

PPG-SB = a

St. Mary's = SM

R1 to R8 = Response

PPG-WB = b

Lafarge = LF

Table 5.3. Results of Fractional Factorial Design of Experiments (Cont'd).								
Run Order	17	18	19	20	21	22	23	24
Block	1	1	1	1	1	1	1	1
Standard Order	31	29	21	30	23	10	26	19
VARIABLES								
A = Powder Type	a	a	a	b	a	b	b	a
B = Powder/Cement Ratio	1.5	1.0	1.0	1.0	1.5	1.0	1.0	1.5
C = Cement Type	LF	LF	LF	LF	LF	SM	SM	SM
D = Aggregate/Cement Ratio	4.5	4.5	4.0	4.5	4.0	4.5	4.5	4.0
E = Water/Cement Ratio	.55	.55	.55	.55	.55	.45	.55	.55
F = Air-Entraining Agent/Cement Ratio	.05	.10	.05	.05	.10	.05	.10	.05
RESPONSE								
R1 = Set Time (Hours)	3.9	4.0	4.1	3.9	3.9	4.0	4.2	3.9
R2 = Slump (inches)	2.6	3.0	2.8	2.9	2.7	3.0	2.9	3.0
R3 = Air Content (%)	6.5	12	5.9	6.3	10	6.5	14	6.5
R4 = Unit Weight (Pcf)	343	328	340	341	331	342	338	345
R5 = Flexure (Ksi)	.84	.51	.84	.80	.61	.75	.63	.75
R6 = Compression (Ksi)	4.6	3.8	4.6	4.8	3.8	4.0	3.9	4.5
R7 = 7-Day Expansion (%)	.05	.04	.04	.04	.05	.04	.04	.05
R8 = 28-Day Expansion (%)	.05	.04	.04	.04	.05	.04	.04	.05

A to F = Variables

PPG-SB = a

St. Mary's = SM

R1 to R8 = Response

PPG-WB = b

Lafarge = LF

Table 5.3. Results of Fractional Factorial Design of Experiments (Cont'd).								
Run Order	25	26	27	28	29	30	31	32
Block	1	1	1	1	1	1	1	1
Standard Order	17	7	16	3	27	14	18	9
VARIABLES								
A=Powder Type	a	a	b	a	a	b	b	a
B=Powder/Cement Ratio	1.0	1.5	1.5	1.5	1.5	1.0	1.0	1.0
C=Cement Type	SM	LF	LF	SM	SM	LF	SM	SM
D=Aggregate/Cement Ratio	4.0	4.0	4.5	4.0	4.5	4.5	4.0	4.5
E=Water/Cement Ratio	.55	.45	.45	.45	.55	.45	.55	.45
F=Air-Entraining Agent/Cement Ratio	.10	.05	.05	.10	.10	.10	.05	.10
RESPONSE								
R1=Set Time (Hours)	4.1	3.8	3.9	3.9	3.9	3.9	4.0	4.1
R2=Slump (inches)	2.9	2.1	2.0	2.0	2.1	3.0	2.9	3.0
R3=Air Content (%)	11	6.5	6.1	12	13	12	5.9	14
R4=Unit Weight (Pcf)	335	343	345	327	328	340	343	341
R5=Flexure (Ksi)	.62	.75	.76	.47	.51	.61	.47	.63
R6=Compression (Ksi)	3.5	4.8	4.8	3.7	3.6	3.8	3.8	4.0
R7=7-Day Expansion (%)	.04	.05	.05	.05	.05	.04	.04	.04
R8=28-Day Expansion (%)	.04	.05	.05	.05	.05	.04	.04	.04

A to F = Variables

PPG-SB = a

St. Mary's = SM

R1 to R8 = Response

PPG-WB = b

Lafarge = LF

Factorial analysis of variance of the 7-day and 28-day restrained expansion test results indicated that:

- * The only factor with statistically significant effects, at 95% level of confidence, on 7-day and 28-day expansions is powder content. The effect of powder type on expansion could be detected only at 90% level of confidence. The interaction curves presented in Figure 5.1 demonstrate the trends in the powder type and powder content effects on 7-day and 28-day restrained expansions.

- * There was an interaction only between powder type and air content in determining 28-day restrained expansion at 95% level of confidence; this interaction could be detected only at 90% level of confidence in the case of 7-day restrained expansion (see Figure 5.2). It should be noted that the higher level of air content used in this study is far in excess of typical values, and this interaction seems to be of little value.

One may conclude from the results of statistical analyses of restrained expansion test results that the powders present the potential to be effective in concrete mixtures with diverse constituents and proportions.

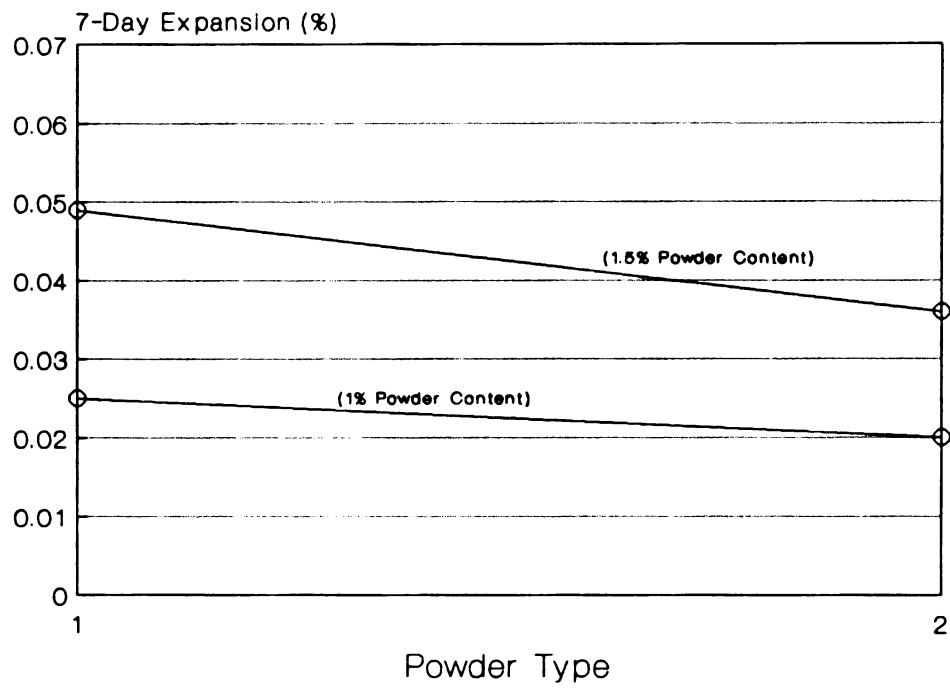


Figure 5.1 (a). Effects of Interactions of Powder Type and content in Determining 7-Day Restrained Expansions.

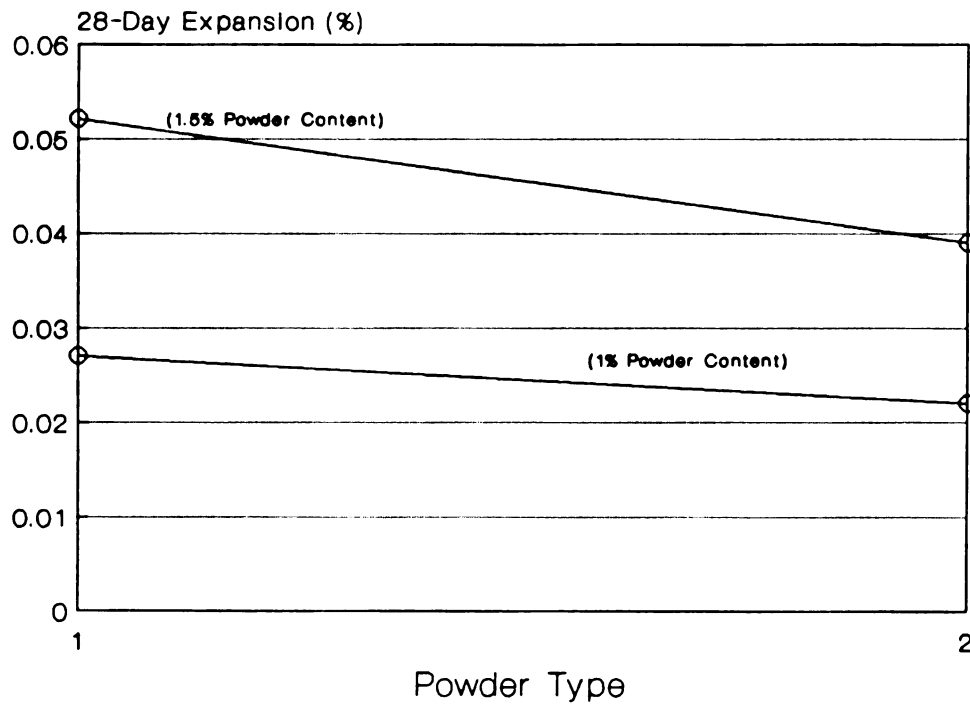


Figure 5.1 (b). Effects and Interactions of Powder Type and content in Determining 28-Day Restrained Expansions.

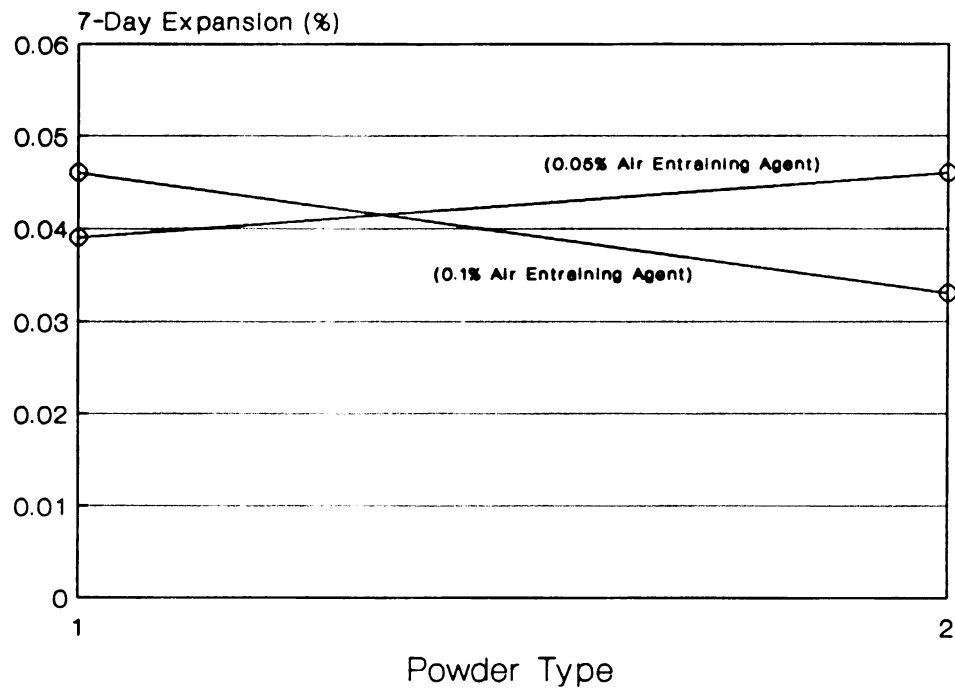


Figure 5.2 (a). The Interaction Between Powder Type and Air content in Determining 7-Day Restrained Expansions.

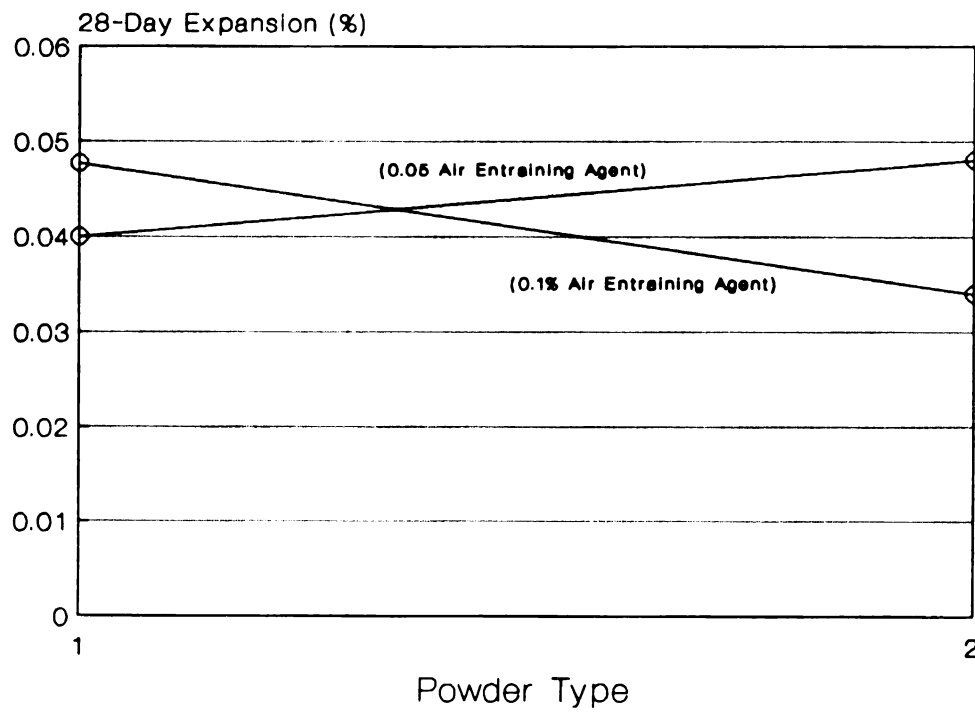


Figure 5.2 (b). The Interaction Between Powder Type and Air content in Determining 28-Day Restrained Expansions.

Factorial analysis of variance of the final set time test results suggested that:

- * At 95 % level of confidence, there were interactions between powder content and cement type, and powder content and aggregate content in determining final set time (see Figure 5.3 a & b, respectively). At 90% level of confidence, powder type and aggregate content also showed an interaction (Figure 5.3c). However, from a practical viewpoint, the actual changes in set time were relatively small and of little practical consequence.

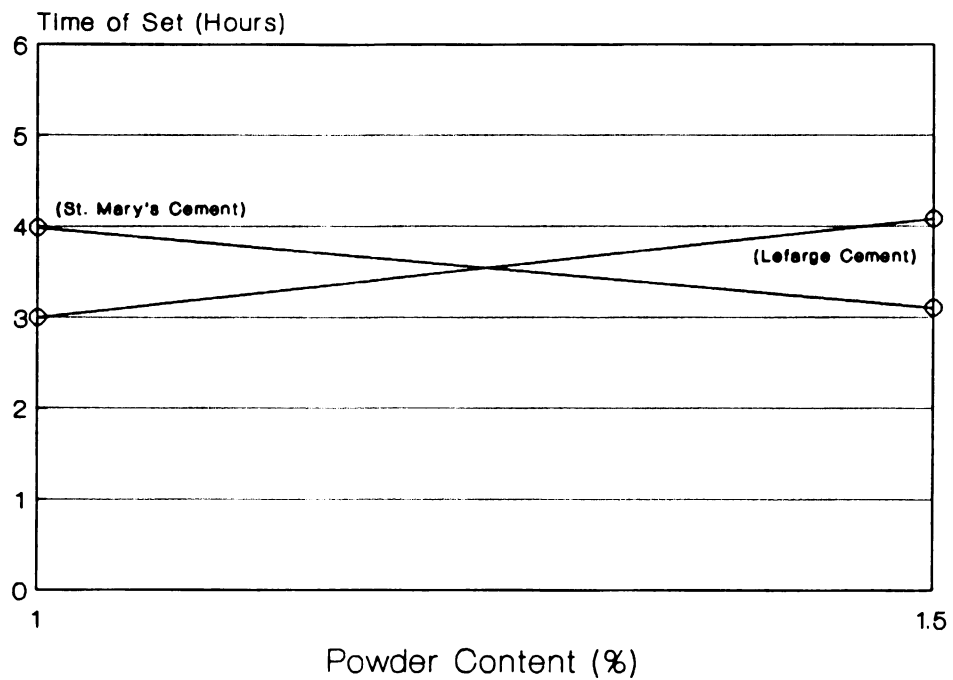


Figure 5.3 (a). Interaction of Different Factors (Powder Content & Cement Type) in Determining Final Set Time.

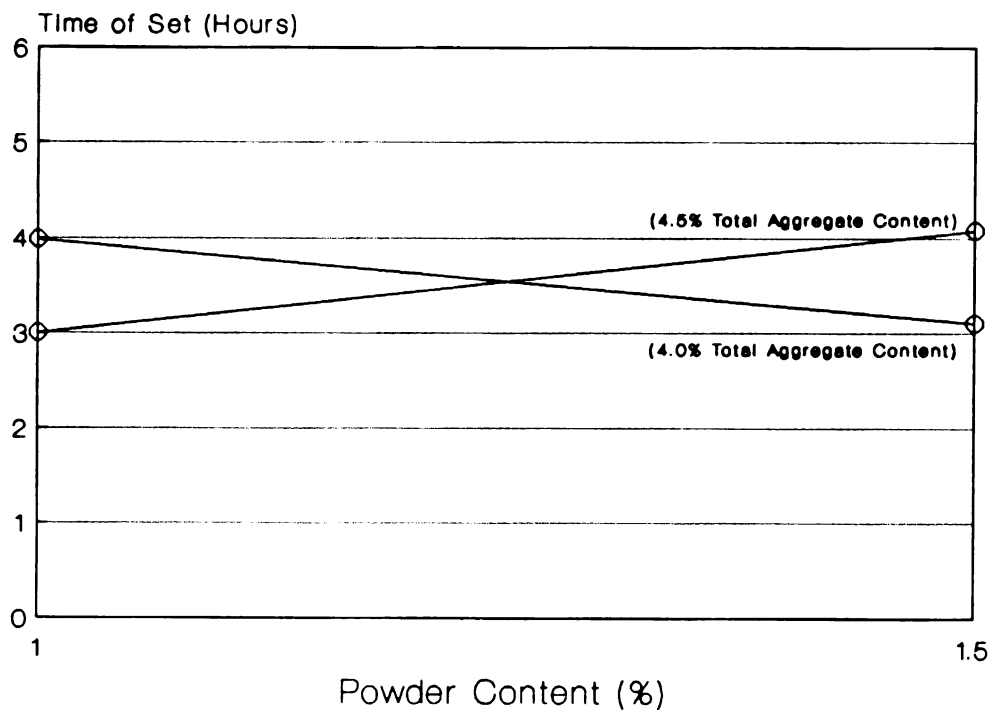


Figure 5.3 (b). Interaction of Different Factors (Powder Content & Aggregate Content) in Determining Final Set Time.

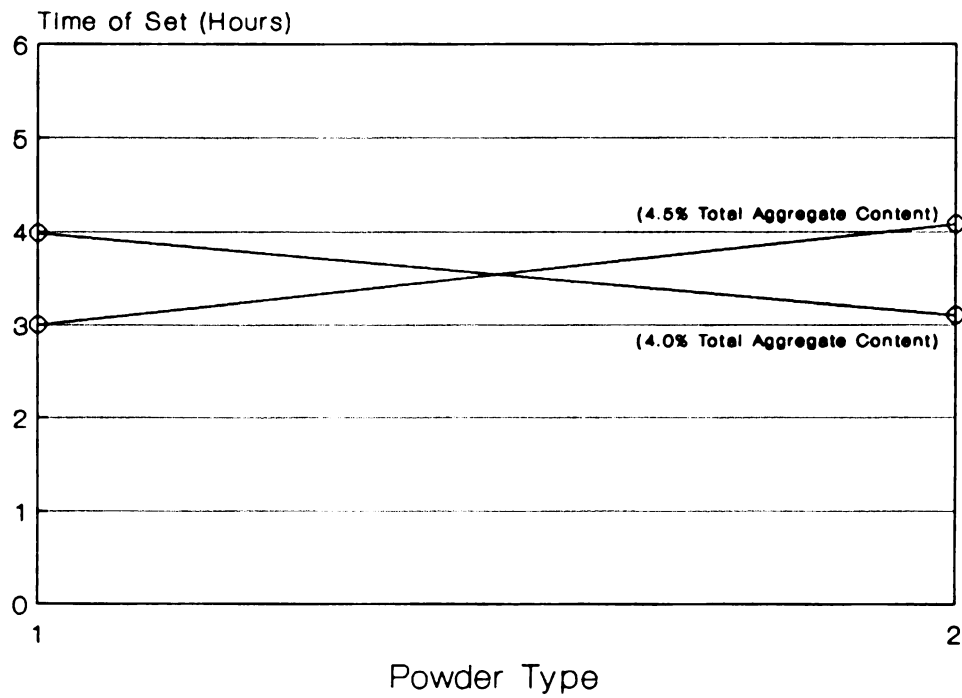


Figure 5.3 (c). Interaction of Different Factors (Powder Type & Aggregate Content) in Determining Final Set Time.

Factorial analyses of variance of the compressive and flexural strengths test results suggested that:

- * None of the effects and interactions involving powder type and content were influential determining the compressive strength of concrete.**

- * At 95 % level of confidence, there were interactions between powder type and air content, and between powder type and aggregate content in determining flexural strength as shown in Figures 5.4 a & b respectively. At 90 % level of confidence, water-cement ratio also influenced the flexural strength.**

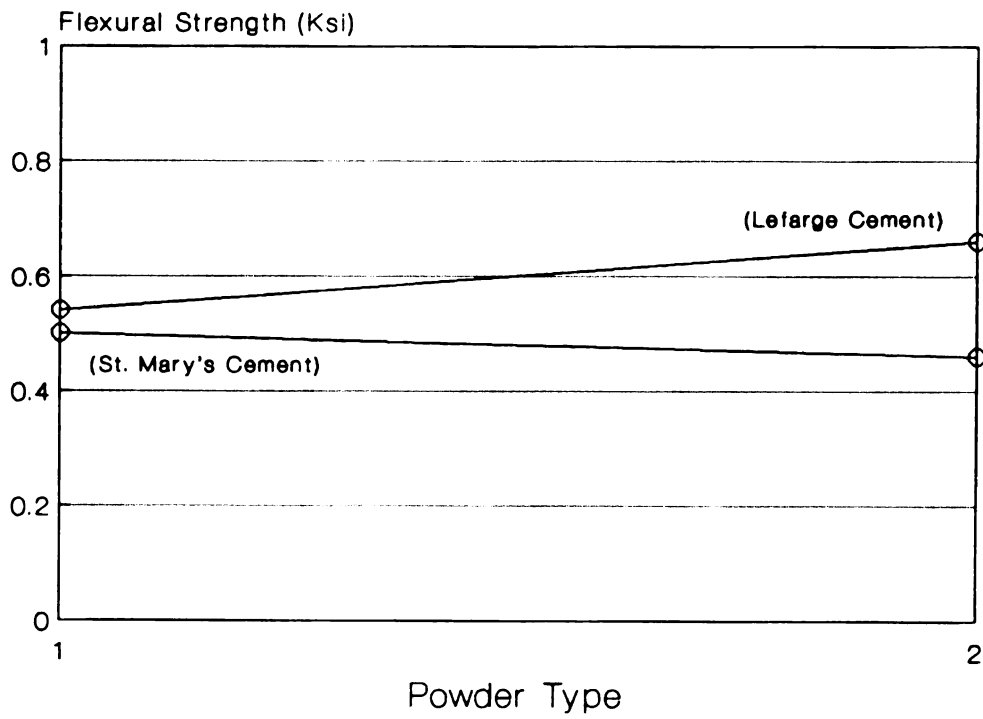


Figure 5.4 (a). Interactions Between Different Factors (Powder Type & cement Type) in Determining Flexural Strength.

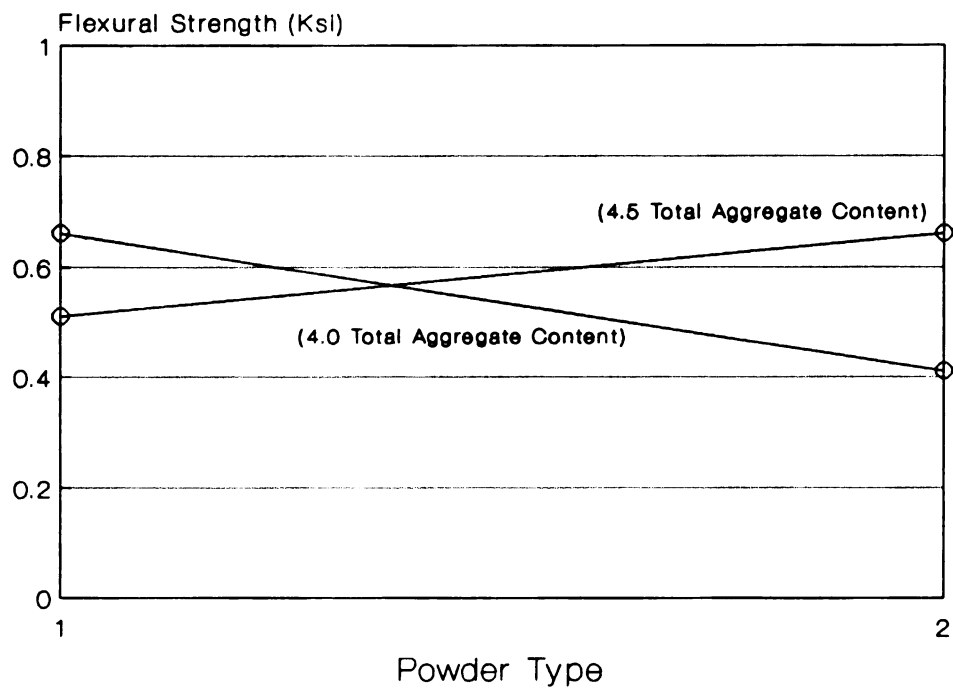


Figure 5.4 (b). Interactions Between Different Factors (Powder Type & Aggregate Content) in Determining Flexural Strength.

Factorial analyses of variance of the slump, air content and unit weight test results indicated that:

- * For slump, the only factor or interaction relating to powders which played a role at 95 % level of confidence was powder content (see Figure 5.5). A relatively small reduction in slump was observed when powder content was increased from 1 % to 1.5 % by weight of cement.**

- * At 95 % level of confidence, none of the effects or interactions associated with powder played a role in determining air content; only the dosage of air entraining agent was a factor in this case (see Figure 5.6).**

- * At 95 % level of confidence, the interaction between powder content and cement type influenced the unit weight of concrete (see Figure 5.7a); the actual effects were, however, relatively small and of little practical significance. Air content had the most significant effect, at 95 % level of confidence, on unit weight (see Figure 5.7b).**

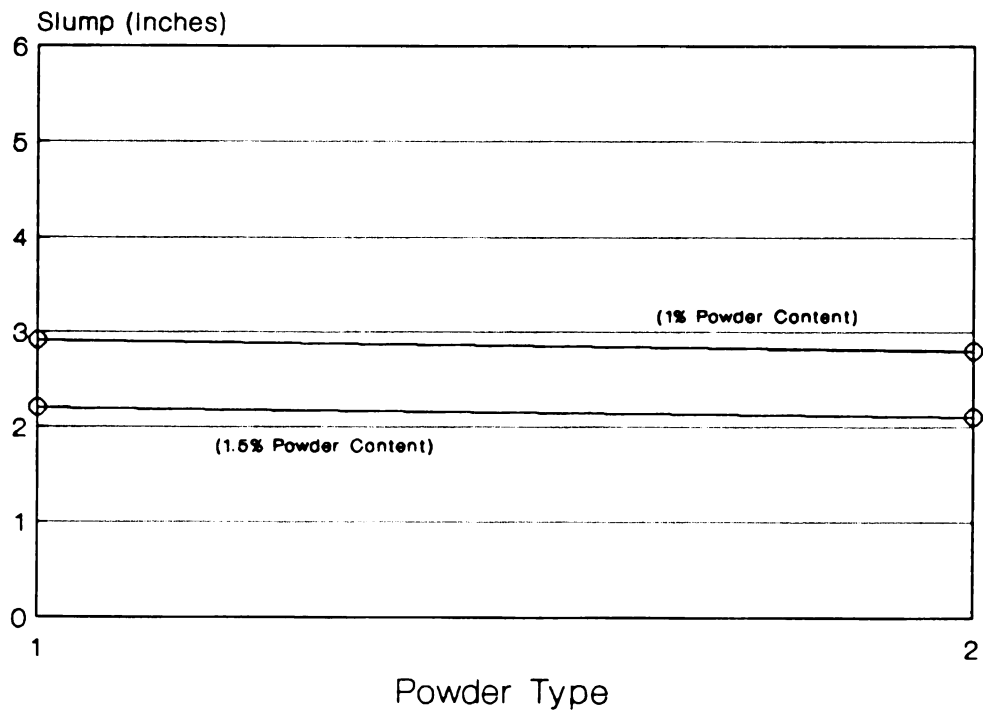


Figure 5.5. Effect of Powder Content on Slump.

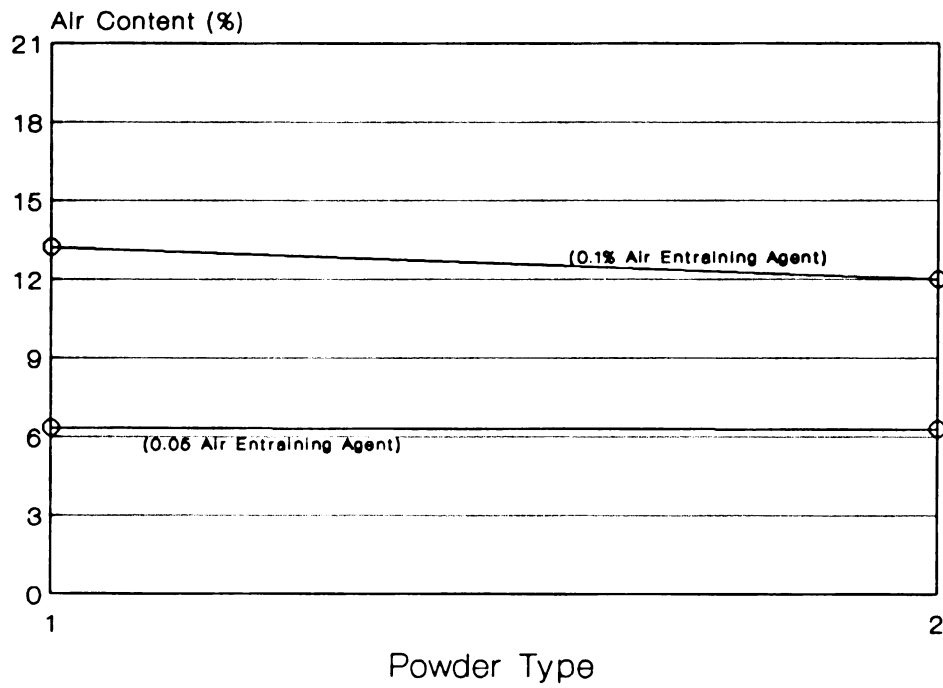


Figure 5.6. Effect of the Dosage of Air Entraining Agent on Air Content.

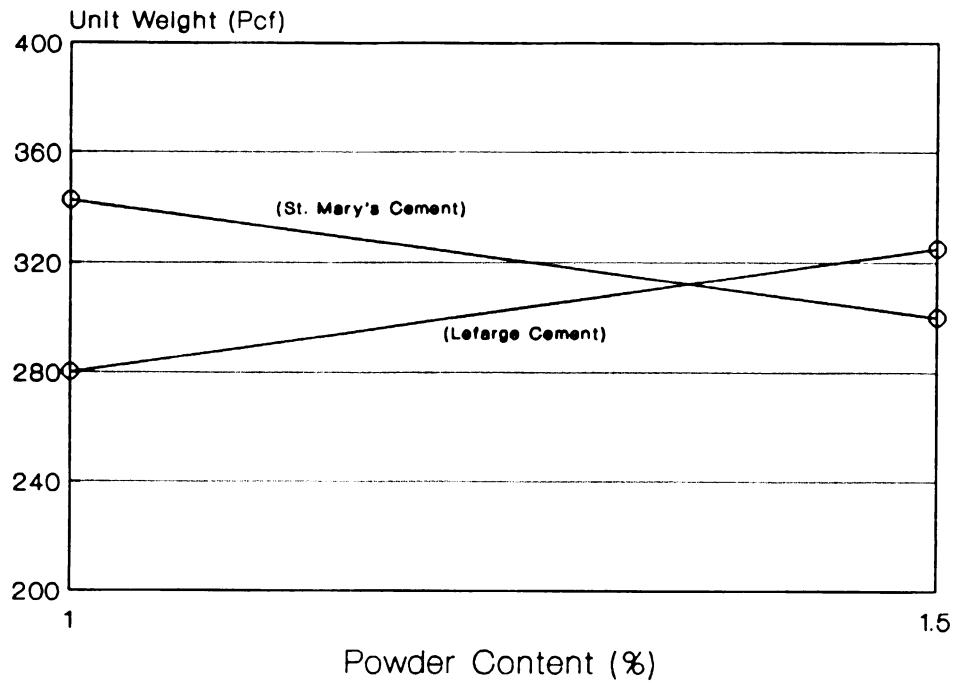


Figure 5.7 (a). Effects and Interactions of Powder Content and Cement Type in Determining Concrete Unit Weight.

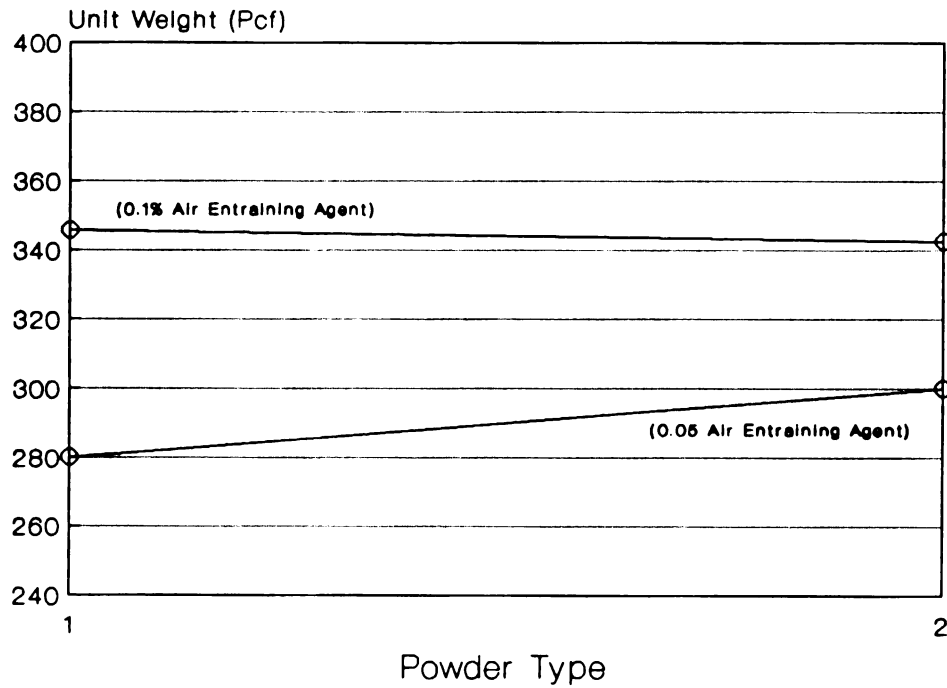


Figure 5.7 (b). Effects and Interactions of the Dosage of Entraining Agent in Determining Concrete Unit Weight.

5.4 SUMMARY AND CONCLUSIONS

A comprehensive experimental study, based on fractional factorial design of experiments, was conducted in order to determine the general applicability of paint sludge powders as expansive admixtures in concrete mixtures with diverse types and proportions of constituents. The effects and interactions of powder type and content, cement type, aggregate content, water cement ratio and dosage of air entraining agent on the following properties of concrete materials were investigated: 7-day and 28-day restrained expansion, set time, compression and flexural strengths, slump, air content and unit weight.

It was concluded that the paint sludge powders present the potential to be effective in providing sufficient restrained expansion in diverse concrete mixtures without any adverse effects on concrete material properties. It is, however, advisable to evaluate the performance of the powders in conjunction with the concrete mixtures used in a specific job before implementing a project involving the use of paint sludge powders as expansive admixtures.

CHAPTER VI

DURABILITY CHARACTERISTICS

6.1 INTRODUCTION:

Concrete is inherently a durable material. If properly designed for the environment to which it will be exposed, and if carefully produced with good quality control, concrete is capable of providing maintenance-free performance for decades without the need for protective coatings, except in highly corrosive environments. However, concrete is potentially vulnerable to attack in a variety of different exposures unless certain precautions are taken. Deterioration of concrete can be caused by the adverse performance of any one of the three major components: aggregate, paste, or reinforcement, and can be due to either chemical or physical causes. Although one particular environmental factor may initiate distress, other factors may contribute and aggravate the situation.

A major difficulty in studying durability is in predicting concrete behavior several decades in the future on the basis of short-term tests. Much of our understanding of durability has come through a direct study of actual field problems. A major challenge facing the modern concrete technologist is the more accurate prediction of concrete durability under a variety of service conditions.

Since durability under one set of conditions does not necessarily mean durability under another, it is customary to include a general reference to the environment when defining durability. According to ACI Committee 201, durability of portland cement concrete is defined as its ability to resist weathering action, chemical attack, abrasion, or any other process of deterioration; that is, durable concrete will retain its original form, quality, and serviceability when exposed to its environment.

No material is inherently durable; as a result of environmental interactions, the microstructure and, consequently, the properties of materials change with time. A material is assumed to reach the end of service life when its properties under given conditions of use have deteriorated to an extent that the continuing use of the material is ruled either unsafe or uneconomical.

For several reasons, such as the restrained expansion of concrete, lack of bleeding, and little or no microcracking by drying shrinkage, the shrinkage-compensating concrete is expected to provide a more dense and essentially impermeable mass than does Portland cement concrete of an equivalent water/cement ratio in the range of 0.4 to 0.6. Consequently, laboratory and field experience has shown that, in general, Type K cement concretes possess a significantly higher resistance to abrasion, erosion, and chemical attack by aggressive solutions. Tests have also shown that when Type K expansive cements are made with an ASTM Type II or V Portland cement clinker, the concrete shows a durability to sulfate attack that is similar to concrete made with a sulfate-resisting Portland cement. This has been an important consideration in the use of

Type K cement concrete for sanitary structures.

Properly designed, placed, and cured shrinkage-compensating concrete mixtures offer as much resistance to freezing and thawing, and deicer scaling, as Portland cement concretes of comparable quality. Generally, a given amount of an air-entraining admixture will produce a volume of air and void spacing in a shrinkage-compensating concrete which is comparable to that of Portland cement concrete, all other conditions being the same.

Paint Sludge powder is a new source of expansion in concrete which act through mechanism different from theories of conventional type K expansive cement. The purpose of this phase of research was to ensure that shrinkage compensating concrete materials incorporating paint sludge powder as expansive admixture present desirable durability characteristics under diverse causes of chemical and physical deteriorations.

6.2 EXPERIMENTAL PROGRAM

Before a discussion of important aspects of durability of concrete, a few general remarks on the subject will be helpful. First, water, which is the primary agent of both creation and destruction of many natural materials, happens to be central to most durability problems in concrete. In porous solids, water is known to be the cause of many types of physical processes of degradation. As a vehicle for transport of aggressive ions, water can also be a source of chemical processes of degradation. Second, the physical-chemical phenomena associated with water movements in porous solids are

controlled by the permeability of the solid. For instance, the rate of chemical deterioration would depend on whether the chemical attack is confined to the surface of concrete, or whether it is also at work inside the material. Third, the rate of deterioration is affected by the type and concentration of ions in water and by the chemical composition of the solid. Unlike natural rocks and minerals, concrete is a basic material (because alkaline calcium compounds constitute the hydration products of portland cement paste); therefore, acidic water is expected to be particularly harmful to concrete.

Most of our knowledge of physical-chemical processes responsible for concrete deterioration comes from case histories of structures in the field, because it is difficult in the laboratory to simulate the combination of long-term conditions normally present in real life. Also, in practice, deterioration of concrete is seldom due to a single cause; usually, at advanced stages of material degradation, more than one deleterious phenomena are found at work. In general, the physical and chemical causes of deterioration are both present and even separation of the cause from the effect often becomes impossible. Therefore, a classification of concrete deterioration processes into neat categories should be treated with some care. Since the purpose of such classification is to explain, systematically and individually, the various phenomena involved, there is a tendency to overlook the interactions when several phenomena are present simultaneously.

In this research, the following durability characteristics were investigated:

- * Abrasion Resistance
- * Chloride Permeability
- * Freeze-Thaw Durability
- * Deicer Salt Scaling
- * Sulfate Attack
- * Corrosion of Embedded Steel
- * Hot Water Immersion

Shrinkage compensating concrete materials with 1.5% powder content by weight of cement were considered. Both PPG solvent-based and PPG water-based powders were investigated in addition to control concrete materials incorporating Type I and Type K Cements. The concrete mix proportions were as presented in Chapter 5; with water-cement ratio of 0.55.

6.3 TEST PROCEDURES

6.3.1 ABRASION RESISTANCE

Concrete surfaces can be subjected to various types of abrasive wear. For example, sliding or scraping can cause *attrition* and, in the case of hydraulic structures, the action of abrasive solids carried by water generally leads to erosion of concrete. For these reasons, it may be desirable to know the resistance of concrete to abrasion. However, this is difficult to assess as the damaging action varies depending on the cause

of wear, and no one test procedure is satisfactory in evaluating the resistance of concrete to the various conditions of wear. The abrasion test method described in ASTM C-944 was used in this investigation; the depth of wear of a specimen is used in this method as a measure of abrasion.

6.3.2 CHLORIDE PERMEABILITY

In concrete, the role of water has to be seen in a proper perspective because, as a necessary ingredient for the cement hydration reactions and as a plasticizing agent for the components of concrete mixtures, water is present from the beginning. Gradually, depending on the ambient conditions and the thickness of a concrete element, most of the evaporable water in concrete (all the capillary water and a part of the adsorbed water), will be lost, leaving the pores empty or unsaturated. Since it is the evaporable water which is freezable and which is also free for internal movement, a concrete will not be vulnerable to water-related destructive phenomena, provided that there is a little or no evaporable water left after drying and provided that the subsequent exposure of the concrete to the environment does not lead to resaturation of the pores. The latter, to a large extent, depends on the permeability of the material.

Rapid assessment of the chloride permeability of concrete was made in this investigation through the performance of the AASHTO T277-831 (ASTM C-1202) test. This test is concerned with quick assessment of the degree of permeability in concrete samples.

The concrete specimens are conditioned by vacuum saturation, and they are bonded into the test cell with a silicone adhesive. The side of the cell containing the top surface of the specimen is filled with 3.0% NaCl (that side of the cell is connected to the negative terminal of the power supply). The other side of the cell is filled with 0.3N NaOH solution (this side of the cell is connected to the positive terminal of the power supply). A voltage of 60 Volts DC is then applied in this test for six hours; the electrical current passed through the specimen is measured and the current is integrated over time to give the charge that passes (in Coulombs).

Table 6.1 presents the interpretation of Chloride Permeability output. Through this table, one can compare the chloride permeability of the concrete under test to other types of concrete. The higher the charge passed, the higher the permeability of the specimens.

Table 6.1. Interpretation of the Chloride Permeability Output (AASHTO T-277-831)	
Chloride Permeability	Charge Passed in Coulombs
High	4,000
Moderate	2,000 - 4,000
Low	1,000 - 2,000
Very Low	100 - 1,000
Negligible	100 or less

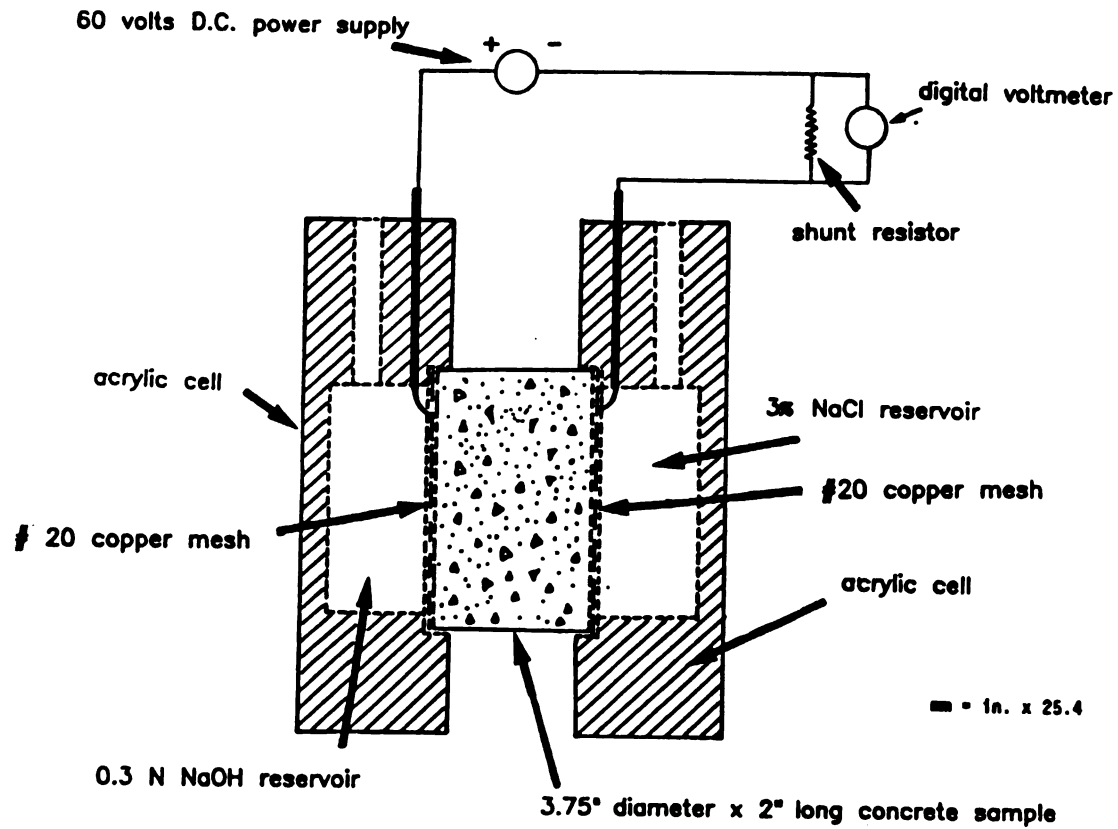


Figure 6.1. Chloride Permeability Test Set Up [Kuhlmann, et. al, 1984]

6.3.3 DE-ICER SALT SCALING

Concrete that is adequately air-entrained for frost resistance may nevertheless be damaged by repeated application of de-icing salts. Concrete that has suffered salt scaling becomes roughened and pitted due to the spalling of small pieces of mortar.

The exact causes underlying salt scaling are not known, but they probably involve more than one process. It has been suggested that the consumption of heat required to melt ice when the de-icer is applied causes a rapid drop in the temperature of the concrete just below the surface, which may cause damage from either the effects of rapid freezing or the stress caused by differential thermal strains. The additional free moisture now present at the surface of the concrete may encourage the growth of microscopic or macroscopic ice lenses near the surface, where ice formation can still occur. Osmosis has also been suggested as a mechanism of salt scaling. De-icing chemicals can accumulate in the concrete just below the surface of the concrete to form relatively concentrated solutions. When rainwater accumulates on the surface, the phenomenon of osmosis occurs, whereby water flows to equalize concentration differences. Considerable osmotic (hydraulic) pressures can be created by this effect, causing rupture of pastes. The most damage usually occurs in the late winter at places where rainwater can accumulate and remain for some time.

Scaling is most likely to occur on surfaces that have been overvibrated, trowelled too early and too long, subjected to plastic shrinkage, or where excessive bleeding occurred. Such surfaces tend to have a weak layer of paste or mortar either at the surface or just below, and may have microcracks or bleeding channels that can transport

surface solutions to lower levels. Careful attention to mix design, placing, and finishing should eliminate many potential problems. Table 6.2 indicates the recommended curing times for air-entrained pavements. If adequate moist curing is followed by a period of drying before de-icing chemicals are applied, scaling should not be a problem. ASTM C-672 is used for assessing scaling resistance of concrete surfaces. A solution of CaCl_2 (Calcium chloride) is ponded on the surface of the specimens, which are then subjected to freezing-thawing cycles. (The freeze-thaw cycles are not the same as in the freeze-thaw durability tests). Visual examination of the surface are made every five cycles.

Table 6.2. Minimum Moist-Curing Times to Develop Salt Scaling Resistance. [58]

Cement Type	Acceleration (CaCl_2)	Minimum Curing Period (days)		
		23°C (73°F)	4°C (39°F)	-4°C (25°F)
I	0	7	15	> 60
	2	7	7	30
II	0	7	12	35
	2	7	7	28
III	0	7	7	24

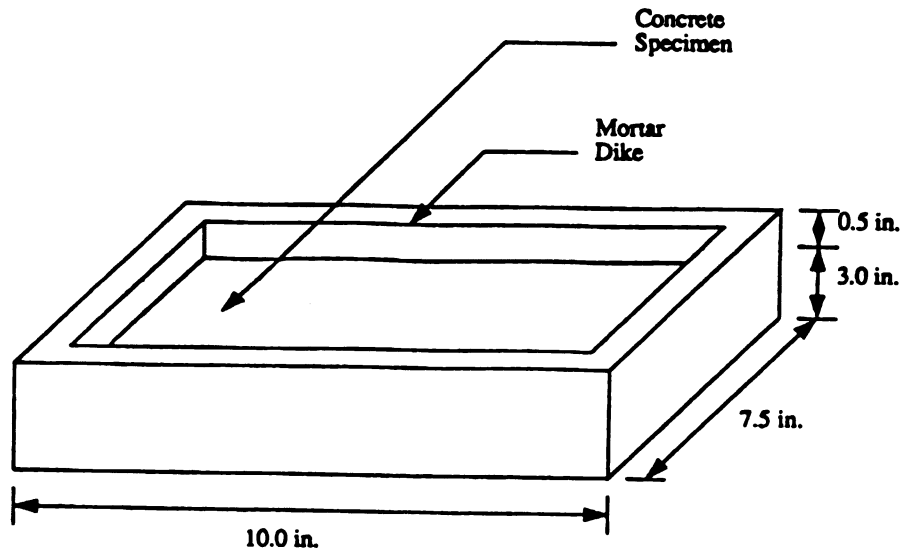


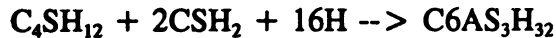
Figure 6.2. Specimen Used in Scaling Test. [58]

6.3.4 SULFATE ATTACK

Perhaps the most widespread and common form of chemical attack is the action of sulfates on concrete. Sulfates are often present in groundwater, particularly when high proportions of clay are present in the soil, and seawater has sulfates as a major constituent. Groundwater may have local concentrations of sulfates in the vicinity of industrial wastes such as mine tailings, slag heaps, and rubble fills. Sulfates present in rainwater from air pollution, or produced by biological growths, may cause slow deterioration even in concrete above ground.

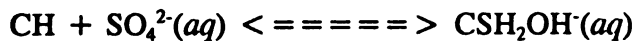
Sulfate attack is actually a rather complex process which may involve a number of secondary processes. However, laboratory and field experiences have definitely

established a correlation between the C_3A content of a Portland cement and its susceptibility to sulfate attack. The major cause is sulfoaluminate corrosion, in which ettringite is formed from monosulfoaluminate:



This is accompanied by a very large increase in solid volume which causes a volume expansion within the paste and which generates accompanying internal stresses and ultimately leads to cracking.

Sulfoaluminate attack is initiated by an initial reaction between sulfate ions and Calcium hydroxide:



This reaction can be described as a gypsum corrosion, since it is also an expansive reaction, but gypsum corrosion only contributes directly to concrete deterioration at concentrations of sulfate ions above 1000 ppm and remains of secondary importance until much higher levels are reached (in excess of 4000 ppm). At low levels of sulfate concentration, the importance of the reaction expressed in the above equation is that it encourages the penetration of sulfate ions into the concrete and concentrates them in a form in which they can react directly with monosulfoaluminate.

At present, no standard ASTM test exists for assessing the sulfate resistance of a concrete, and predictions of performance can not be made with certainty. There is still no satisfactory rapid test for sulfate attack, and many different approaches have been used. ASTM C-452, which is proposed for research purposes only, uses an abnormally high gypsum content in the cement to test for sulfate resistance. It is a test for the

cement only, not for concrete. This procedure has several drawbacks, the major objections being that the test is too slow for use as a routine specification test is too slow for use as a routine specification test, that the behavior of concrete does not necessarily parallel the behavior of mortar bars, and that realistic exposure conditions are not reproduced. Furthermore, test that are used with portland cements doe not always adequately assess the behavior of cements blended with pozzolans or slags.

Restrained concrete expansion specimens (with sizes as indicated in ASTM C-878) were submerged in this investigation into water with 5% sodium sulfate solution, after 28 days of lime saturated moist curing. Expansion measurements were made at regular time intervals in order to determine if there is any expansion associated with sulfate effect.

6.3.5 FREEZE-THAW DURABILITY

Several different processes can contribute to paste behavior during freezing. These are generation of hydraulic pressure by ice formation, desorption of water from C-S-H, and segregation of ice. A 9% volume increase occurs as water turns to ice, but this change is insufficient to account for all of the dilation observed in cement paste and in concrete. Some of the dilation that occurs on first freezing is probably due to direct expansion of ice in microcracks, and this will be increased if progressive microcracking occurs during continued freezing and thawing.

However, the major dilation that occurs was attributed by Powers to the generation of *hydraulic pressure*. As ice forms in a capillary, the accompanying volume

increase causes the residual water to be compressed. This pressure can be relieved if the water can escape from the capillary to a free space by diffusing through unfrozen pores, but if the water has too far to move to an escape boundary, the capillary will tend to dilate and the surrounding material will come under stress (see Figure 6.3). The superposition of pressure from adjacent capillaries will eventually cause the tensile strength of the paste to be exceeded and rupture will occur. As the temperature is progressively lowered, more capillary water is involved in freezing, increasing the hydraulic pressures and thereby increasing microcracking and dilation. In a saturated non-air-entrained paste, the only free space is the exterior of the specimen, and the diffusion of water to the outside is too slow to relieve the hydraulic pressure. Thus, the inclusion of entrained air provides empty space within the paste to which the excess water can move and freeze without damage. The bubbles act as "safety valves" and the spacing factor determines the average distance the water must travel to reach the free space. This distance must not be too great if hydraulic pressure is to be relieved: hence the requirement of a critical spacing factor.

A number of different tests have been developed to assess the long-term frost resistance of concrete. These involve subjecting concrete to different freeze-thaw cycles and measuring the progressive internal damage by monitoring weight loss, length change (dilation), decrease in strength, or dynamic modulus of elasticity. Tests differ in the nature of the freeze-thaw cycle used and the condition of the specimen during the test, but all tests are arbitrary, since they involve only one set of conditions. As we have seen, the potential for frost damage depends on rate of freezing, w/c ratio, time of moist

curing, and degree of saturation, and these may vary widely in actual service conditions. Thus, predictions from freeze-thaw tests are not always a good indication of actual service performance, and results should be treated with caution.

The standard method of test described in ASTM C-666 prescribes a cooling and heating cycle between 4.4 and -17.8°C (40 to 0°F) at a rate of cooling in the range of 12.2 to 14.4 °C/h (22 to 26°F/h) Freezing can be done while the specimen is in air (procedure A) or immersed in water (procedure B), but it is thawed in water in both cases. The test is continued (either continuously or intermittently) until 300 cycles or until the dynamic modulus has reached 60% of its initial value, whichever occurs first. A durability factor (D.F.) can then be calculated:

$$D.F. = P \times N/300$$

where P is the percentage of the initial dynamic modulus after N cycles. There are no definite values of the durability factor for acceptance or rejection of the concrete subjected to this test. A value of less than 40 suggest that the concrete may be unsatisfactory, whereas above 60 it is likely to perform well, but there is no guarantee that this prediction will hold true.

A major objection to ASTM C-666 is the unrealistically high rate of freezing, since in the field rates exceeding 3°C/h (5°F/h) are rarely encountered. ASTM C-671 overcomes this objection by specifying one freeze-thaw cycle every 2 weeks, employing this rate of freezing, with a rapid thawing regime. Damage is monitored by measuring linear expansion during the tests, which is continued until critical dilation (i.e., until dilation begins to increase sharply) or the desired number of cycles is completed. This

test allows the user to specify the curing history of the specimen and the exposure conditions to most nearly match the expected service conditions.

The ASTM C-666 procedure B was adopted in this investigation.

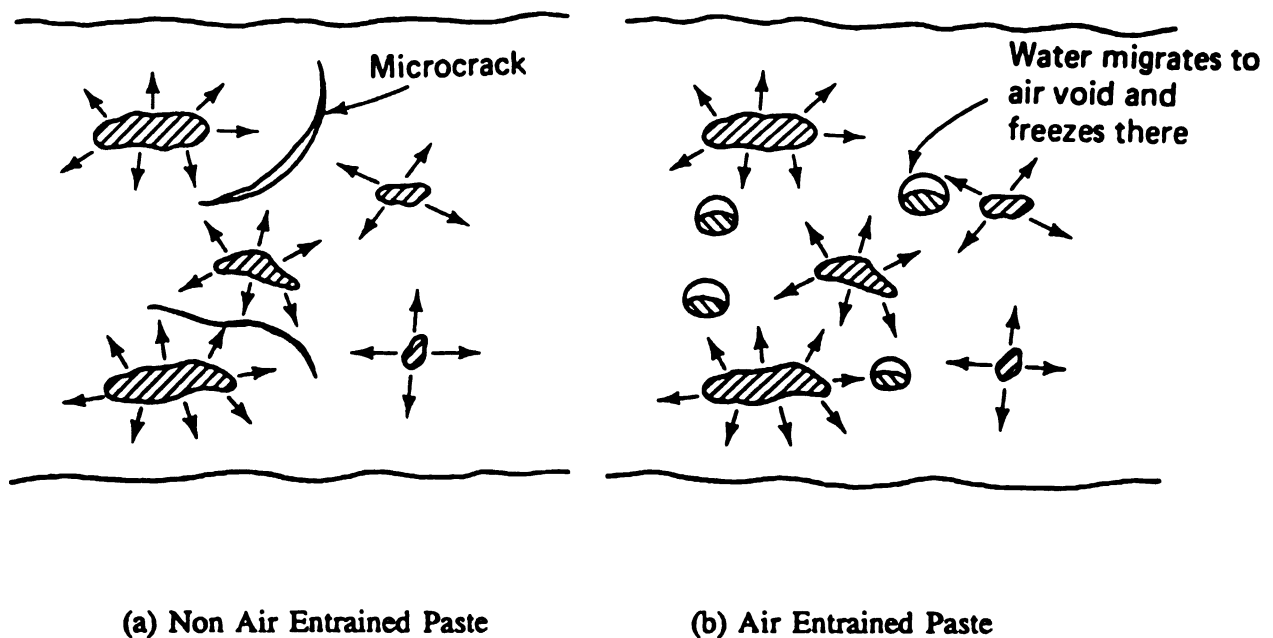


Figure 6.3. Creation of hydraulic pressure in frozen cement paste:
(a) non-air-entrained paste; (b) air-entrained paste. [58]

6.3.6 CORROSION OF EMBEDDED STEEL

The corrosion test were conducted using 75 x 150 mm (3 x 6 in) cylindrical specimens with 12 mm (0.5 in) diameter rebars placed along their longitudinal axis. cylindrical aluminum alloy molds were used with some modifications in order to be able to hold the steel reinforcement in the middle of the specimen and to provide uniform cover thickness throughout. In this arrangement (see Figure 6.8) the cylindrical part of the mold rests on a plate having a hole in the middle such that the center of the hole coincides with the cylinder axis along which the reinforcing bar is placed. Threaded rods support this plate to a base plate. The latter has a groove aligned with the hole in the upper plate so that the reinforcing steel bar passes through the hole in the upper plate and rests on the groove provided in the base plate. Before casting the specimens, the molds were coated with a thin layer of mineral oil and then a 12.5 mm (0.5 in) diameter steel bar was placed. The pouring of concrete into the molds was performed at three layers of one-third the cylinder's height. External vibration by means of a vibrating table was applied to compact the concrete, and troweling was performed to smoothen the finished surface. The specimens were then covered with a wet burlap topped with a polyethylene sheet to prevent water evaporation from the unhydrated concrete. Demolding of specimens from the molds was done after 24 hours. The specimens were then placed in moist curing room for 60 days before corrosion measurements.

In the corrosion test program, the specimens were placed in a tank and partially immersed in a 5% sodium chloride solution. The level of solution was kept at twice the cover thickness. Corrosion was monitored by obtaining half-cell potentials of the

embedded steel at regular time intervals using millivoltmeter. ASTM C-876 provides the classification shown in Table 6.3 for assessing the results of the half cell test:

Table 6.3. ASTM C-876 Half-Cell Test Classification	
Measured Potential (mV)	Statistical Risk of Corrosion (%)
Less than -350	90
Between -350 and -200	50
Greater than -200	10

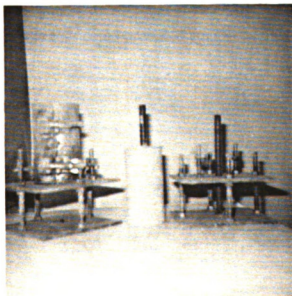


Figure 6.4. Arrangement of Molds for Corrosion Specimen

6.3.7 HOT WATER IMMERSION

Any deleterious chemical reactions, which could possibly take place between powders and cement, would be potentially accelerated when the specimens are immersed in hot water. After 7 days of moist curing, three specimens from each mix were immersed in hot water at 60 °C (140 °F) for 21 days before flexural strength measurements were made.

6.4 EXPERIMENTAL RESULTS AND DISCUSSIONS

6.4.1 ABRASION RESISTANCE

The abrasion test results are presented in Figure 6.5. Analysis of variance of results followed by multiple comparisons indicated that, at 95% level of confidence, all the concrete mixtures considered produced comparable resistance to abrasion effects.

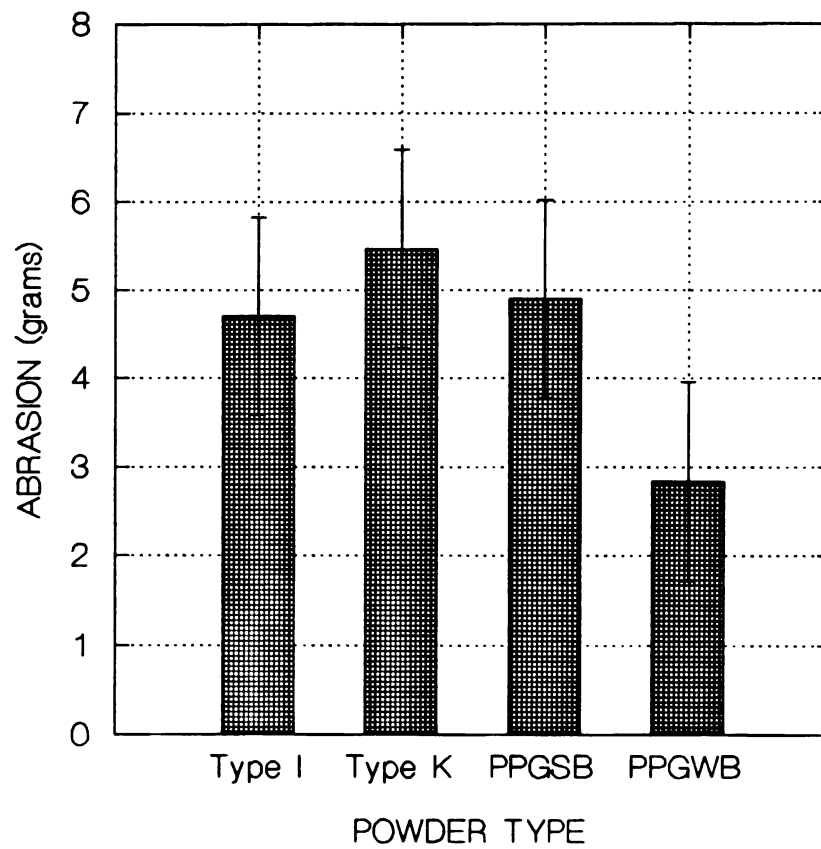


Figure 6.5. Abrasion Resistance Test Results (Means and 95% Confidence Intervals - 3 replications).

6.4.2 CHLORIDE PERMEABILITY

The rapid chloride permeability test results are presented in Figure 6.6. Analysis of variance and multiple comparison of results indicated that, at 95 % level of confidence:

- * Type I cement produced the highest permeability, statistically different from all others except for the concrete with PPG Water-based paint sludge powder.
- * Type K cement and PPG Solvent-based powders produced the lowest, and statistically comparable, levels of permeability.

In general, using conventional Type I Portland cement concrete as a basic, the presence of powders in concrete either did not change or improved permeability characteristics.

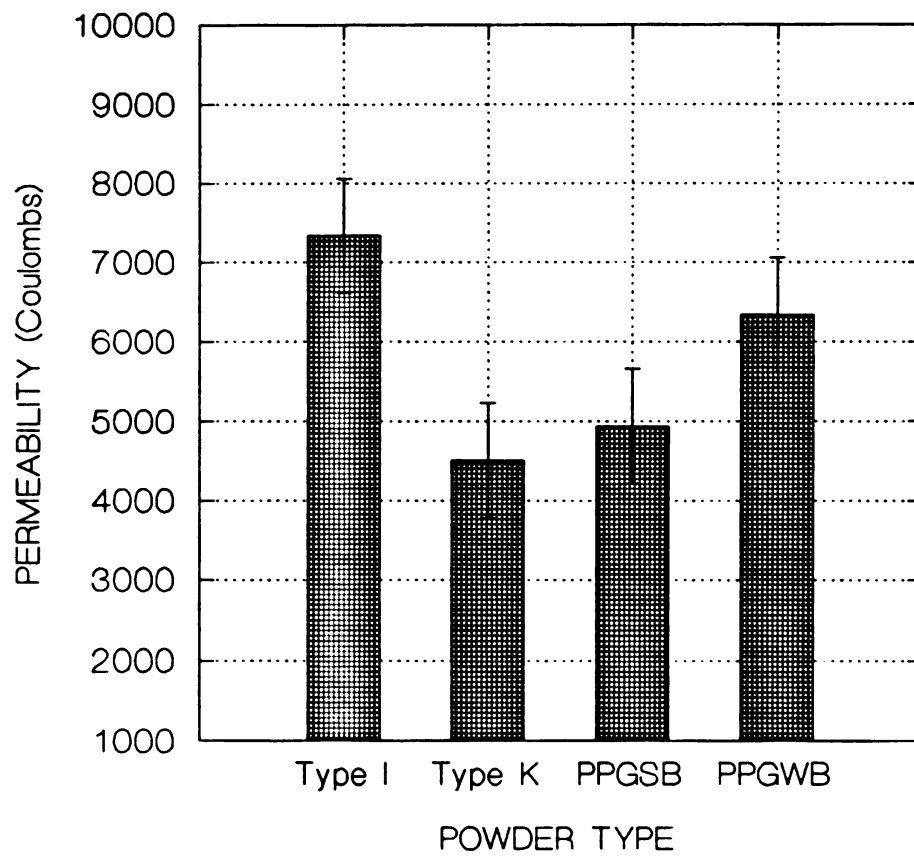


Figure 6.6. Rapid Chloride Permeability Test Results (Means and 95% Confidence Intervals - 3 replications).

6.4.3 FREEZE-THAW DURABILITY

The durability factors obtained from the freeze-thaw durability tests are presented in Figure 6.7. Statistical analysis of the results suggested that, at 95% level of confidence, the two powders produced durability factors which were higher than those obtained with Type I and Type K cements. Type I and Type K cements produced comparable freeze-thaw durability factors.

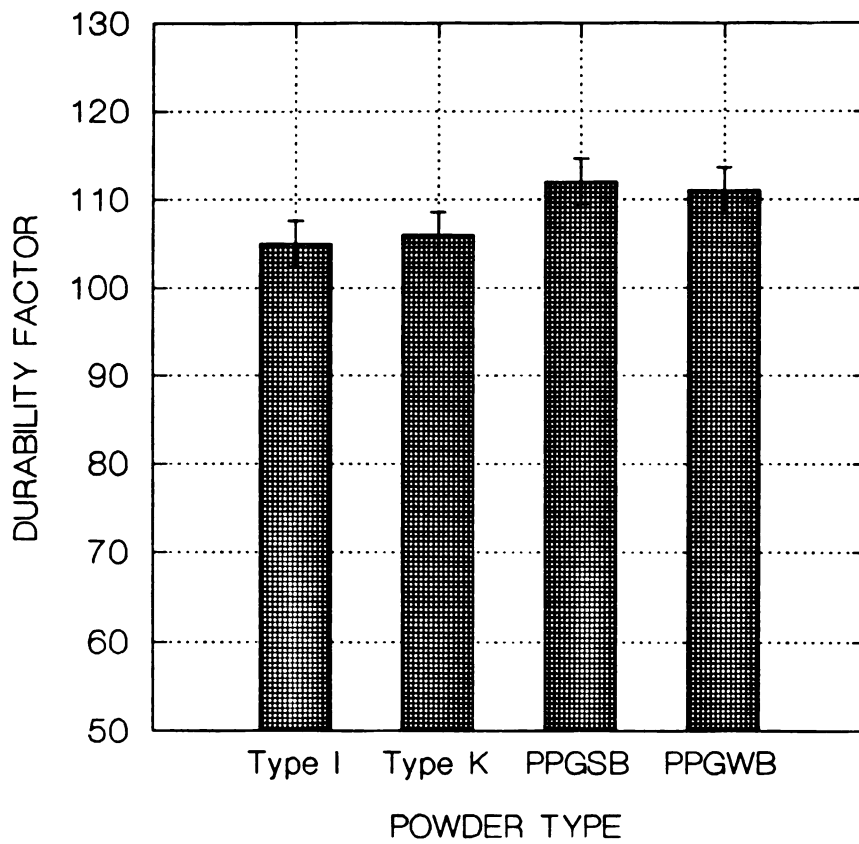


Figure 6.7. Freeze-Thaw Durability Results (Means and 95% Confidence Intervals).

6.4.4 SCALING RESISTANCE

Analysis of variance of the scaling results presented in Table 6.4 indicated that all the concrete mixtures conducted in this investigation produced, at 95% level of confidence, comparable resistance to deicer salt scaling.

Table 6.4 Scaling Resistance Test Results.		
Test Materials	Number of Cycles	Rating
Plain Concrete	50	1
Type K Cement	50	2
PPG Solvent-Based	50	1
PPG Water-Based	50	2

0 = No Scaling, 1 = Very Slight Scaling, 2 = Slight to Moderate Scaling.

6.4.5 SULFATE ATTACK

The sulfate resistance test results are presented in Figure 6.8. After 28-days of moist curing in lime saturated water, the specimens were then immersed in sulfate solution and the expansion measurements were taken daily for 180 days. The two powders produced concretes which showed improved sulfate resistance, at 95% level of confidence, when compared with Type K cement.

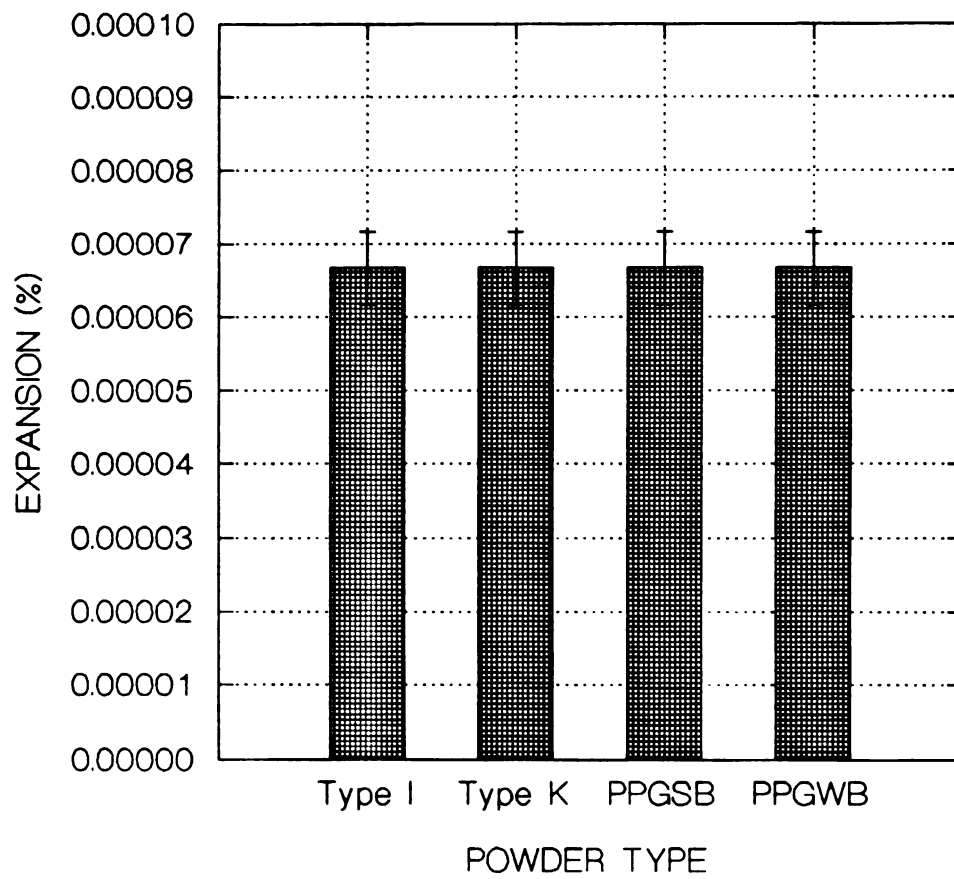


Figure 6.8. Sulfate Resistance Test Results (Means and 95% Confidence Intervals, 3 replication).

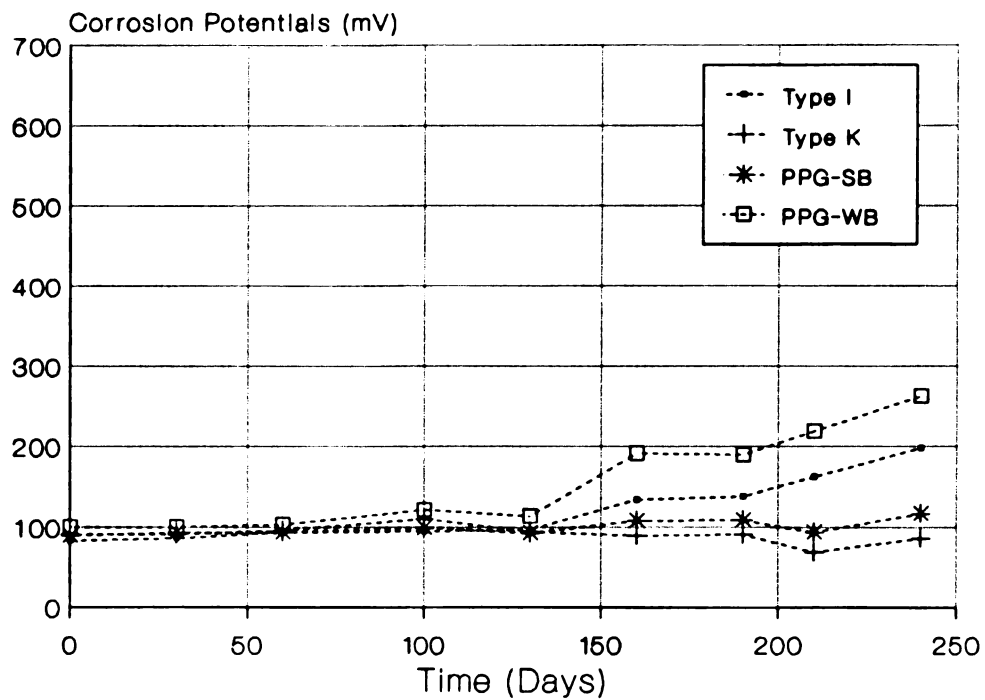


Figure 6.9. Corrosion Potentials Test Results (Means and 95% Confidence Interval).

6.4.6 CORROSION OF EMBEDDED STEEL

The corrosion test results are presented in Figure 6.9. Analysis of variance of the results suggested, at 95% level of confidence, that the corrosion protection qualities obtained with the powders are comparable to those obtained with Type I and K cements.

6.4.7 HOT WATER IMMERSION

The flexural strength test results after hot water immersion are presented in Figure 6.10. Analysis of variance of the results indicated that, at 95% level of confidence, all concrete mixtures considered in this investigation performed similarly after immersion in water.

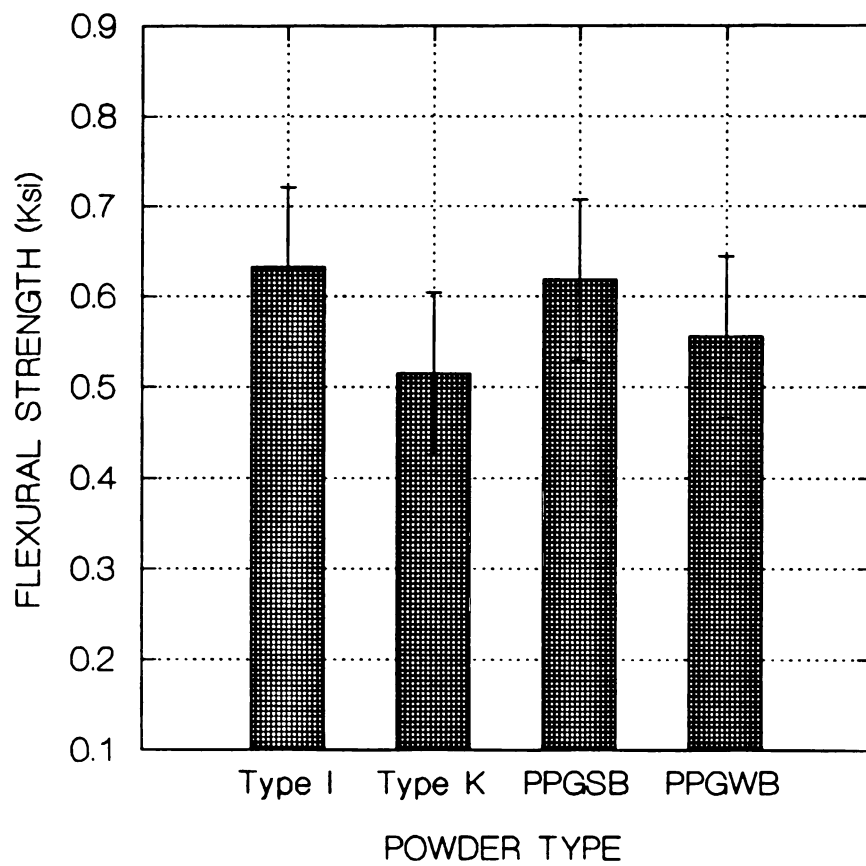


Figure 6.10. Hot Water Durability Test Results (Means and 95% Confidence Intervals, 3 replications).

6.5 SUMMARY AND CONCLUSIONS

Shrinkage compensating concrete materials incorporating two powder type (PPG Solvent-based and PPG Water-based) as well as concrete materials made with Type K cements, were subjected to various accelerated durability tests in order to determine their resistance to different chemical and physical causes of deterioration.

The durability related tests conducted were:

- * Abrasion Resistance
- * Chloride Permeability
- * Freeze-Thaw Durability
- * De-icer Salt Scaling
- * Sulfate Attack
- * Corrosion of Embedded Steel
- * Hot Water Immersion

The results of accelerated aging tests were analyzed statistically. It was concluded that shrinkage compensating concrete materials incorporating paint sludge powders durability characteristics which are generally comparable to those obtained with Type I and Type K cements. The resistance to abrasion, deicer salt scaling, corrosion and hot water immersion were statistically comparable in all concrete mixtures considered. The powders produced better freeze-thaw durability than Type I and K cements and better sulfate resistance than Type K cements; the chloride permeabilities obtained with conventional Type I Portland cement concrete.

CHAPTER VII

GENERAL SUMMARY AND CONCLUSIONS

7.1 INTRODUCTION

The feasibility of producing desirable restrained expansion qualities for the purpose of compensating for drying shrinkage effects using paint sludge powder (an industrial by-product) was demonstrated. The mechanisms responsible for the expansive qualities of this by-product were investigated. The physical, mechanical and durability characteristics of shrinkage compensating concrete incorporating the by-product were examined through a comprehensive experimental program.

7.2 DEVELOPMENT OF EXPANSIVE CEMENT WITH PAINT SLUDGE POWDER

Axially restrained expansion tests were conducted on mortar (ASTM C-806) and concrete (ASTM C-878) materials incorporating 0.5%, 1% and 1.5% paint sludge Powders (by weight of cement), and also on expansive cement and conventional cement mortar and concrete materials.

The mortar test results suggest that the PPG Solvent-based (PPG-SB) and PPG Water-Based (PPG-WB) powders at 1% substitution level (by weight of cement) are capable of satisfying the ASTM requirements on the restrained expansion, set time and air content of expansive cement. The increase in dosage from 1% to 1.5% provides an added safety for satisfying the ASTM requirements on restrained expansion.

ASTM C-878 does not specify any limits on concrete test results. Comparison with the corresponding values for Type K expansive cement thus provided the basis for the evaluation of powders. The powders produced performance characteristic which were essentially similar to those of Type K expansive cement.

It is interesting to note that the restrained expansion test results could reveal differences in the performance of the paint sludge powders which could not be detected through free expansion test results. It seems that the rate of expansion (rather than the total amount of free expansion) is the key factor distinguishing between the effects of different powders in mortar and concrete. Between-shipment variations in the end product qualities are negligible.

7.3 MECHANISM OF EXPANSION ASSOCIATED WITH PAINT SLUDGE POWDER

Chemical and mineralogical analyses of the powders and resulting concretes were undertaken in order to determine the sources of expansive qualities in powders. Chemical analyses indicated that calcium compounds dominate the chemistry of powders, and those powders with higher calcium compound contents produced larger restrained expansions. Calcium may produce expansion through hydration of hard-burnt calcium oxide (when occurring as CaO) or reaction with cement alkalis (when occurring as CaCO_3). Mineralogical analyses indicated that calcium in powders occurs dominantly in the form of CaCO_3 , suggesting that reaction of calcium carbonate with cement alkalis could be the cause of expansion; the fact that higher-alkali cements produced somewhat higher expansions with these powders further supported this hypothesis. Mineralogical analysis

of concrete suggested that powders with higher restrained expansive qualities also produced higher calcium hydroxide contents in hardened concrete.

7.4 EFFECTS OF MIX VARIABLES

A comprehensive experimental study, based on fractional factorial design of experiments, was conducted in order to determine the general applicability of paint sludge powders as expansive admixtures in concrete mixtures with diverse types and proportions of constituents. The effects and interactions of powder type and content, cement type, aggregate content, water cement ratio and dosage of air entraining agent on the following properties of concrete materials were investigated: 7-day and 28-day restrained expansion, set time, compression and flexural strengths, slump, air content and unit weight.

It was concluded that the paint sludge powders present the potential to be effective in providing sufficient restrained expansion in diverse concrete mixtures without any adverse effects on concrete material properties. It is, however, advisable to evaluate the performance of the powders in conjunction with the concrete mixtures used in a specific job before implementing a project involving the use of paint sludge powders as expansive admixtures.

7.5 DURABILITY CHARACTERISTICS

Shrinkage compensating concrete materials incorporating two powder type (PPG Solvent-based and PPG Water-based) as well as concrete materials made with Type K cements, were subjected to various accelerated durability tests in order to determine their

resistance to different chemical and physical causes of deterioration.

The durability related tests conducted were:

- * Abrasion Resistance
- * Chloride Permeability
- * Freeze-Thaw Durability
- * De-icer Salt Scaling
- * Sulfate Attack
- * Corrosion of Embedded Steel
- * Hot Water Immersion

The results of accelerated aging tests were analyzed statistically. It was concluded that shrinkage compensating concrete materials incorporating paint sludge powders present durability characteristics which are generally comparable to those obtained with Type I and Type K cements. The resistance to abrasion, deicer salt scaling, corrosion and hot water immersion were statistically comparable in all concrete mixtures considered. The powders produced better freeze-thaw durability than Type I and K cements and better sulfate resistance than Type K cements; the chloride permeabilities obtained with the powders comparable to those obtained with conventional Type I Portland cement concrete.

It may thus be concluded that the paint sludge powders (an industrial by-product) is capable of producing shrinkage compensating concrete which are comparable to those incorporating conventional (Type K) expansive cements. The lack of any high-volume application for this industrial by-product presents the opportunity to price it to be highly competitive and rapidly penetrate the existing markets of shrinkage compensating

concrete. Successful field projects and the flexibility in pricing may eventually lead to an increased market share of shrinkage-compensating concrete in the overall concrete market.

APPENDIX I

PRACTICAL GUIDELINES

I.1 INTRODUCTION

The paint sludge powders, when used in concrete at an appropriate dosage, produce restrained expansion qualities which are comparable to those obtained with conventional expansive cements. At these dosages, concrete incorporating the powders will present desirable short-term and long-term engineering qualities, similar to those expected from conventional concrete. Hence, design, and construction procedures and application fields for shrinkage-compensating concretes incorporating the powders would be comparable to those used for conventional shrinkage-compensating concrete, as described in chapters 1 and 2. One may use the powders in all current applications of Type K cement following conventional design procedures (including those applying to restraining reinforcement).

I.2 COST ANALYSIS

Shrinkage-compensating concrete incorporating Type K expansive cement or expansive admixtures add 10-20% to the cost when compared to conventional concrete; the exact value depends on the location and availability of expansive products. The lower end of this added cost indicates a price tag of approximately \$6 per cubic yard of concrete for adding expansive qualities to conventional concrete.

Assuming a typical cement contents of 600 lb/yd³ and a powder content of 1.5% by weight of cement, one may estimate an approximate powder dosage of 9 lb/yd³. Hence, to add less than \$6 per cubic yard to the price of concrete, an upper limit for the price of powder at the job site is approximately \$0.65 per lb.

One needs to deduct the packaging and transportation costs as well as the quality control expenses and the distributor's profits from this value before determining the sales price to a distributor.

I.3 TOXICITY CHARACTERISTICS AND LEACHING

This test ensures that the solubility and mobility of any contaminants that are possibly present in the powder are limited to satisfy the EPA requirements when the powder is used as an expansive admixture in portland cement concrete. Concrete materials incorporating 1.5% sludge powder (PPGSB and PPGWB) content were crushed in this test to pass a 0.38 inch (9.5 mm) screen; a buffered acidic leaching solution is then added and the sample is agitated for 18 hours. The leaching solution is then filtered for analysis, where the maximum concentrations of different contaminants in the leached solution are then measured and compared with EPA limits. The results of this analysis as presented in Table I.1 (performed by FIBERTECH Incorporated in Lansing, Michigan) indicates that shrinkage-compensating concrete incorporating the powders satisfied the EPA toxicity requirements.

Table I.1. Paint Sludge Powder Toxicity Results

Metals	Paint Sludge Powder (mg/L)		EPA Limit (mg/L)
	PPGSB	PPGWB	
Arsenic	< 200	< 200	200
Barium	< 100	< 100	100
Cadmium	< 100	< 100	100
Chromium	< 100	< 100	100
Copper	< 100	< 100	100
Lead	< 100	< 100	100
Selenium	< 200	< 200	200
Silver	< 100	< 100	100
Zinc	< 100	< 100	100
Mercury	< 10	< 10	10
Aluminum	< 2,000	< 2,000	2,000
Beryllium	< 2,000	< 2,000	2,000
Boron	< 4,000	< 4,000	4,000
Manganese	< 50	< 50	50
Molybdenum	< 2,000	< 2,000	2,000
Nickel	< 200	< 200	200

REFERENCES

1. Cohen, M.D., "Micromechanics of Expansive Mechanisms in Expansive Cement Concrete," Stanford University, 1981.
2. Richards, C.W., and Helmuth, R.A., "Micromechanical Models for Free and Restrained Expansion," Expansive Cement Concrete, Technical Report No. 191, Jan. 1975, pp. 1-36
3. Aroni, S., Polivka, M., and Bresler, B., "Expansive Cement and Expanding Concrete," Structures and Materials Research, July 1966, pp. 1-74.
4. Gustafarro, A.H., Greening, N., and Klieger, P., "Expansive Concretes Laboratory Testing of Freeze-Thaw and Surface Scaling Resistance," Journal of PCA Research, Jan. 1966, pp. 10-36.
5. Hoff, G., and Mather, K., "A Look at Type K Shrinkage-Compensating Cement Production and Specifications," Book of ASTM Standards, Part 13, SP 64-9, 1977, pp. 153-180.
6. Cohen, M., Olek, J., and Mather, B., "Silica Fume Improves Expansive-Cement Concrete," Concrete International, March 1991, pp. 31-37.
7. Simms, F., "Expansive Cements for Crack-Resistant Concrete," Civil Engineering ASCE, June 1966, pp. 46-47.
8. Civil Engineering - ASCE, "Expansive Cements in the United States - a Three Year Review," Civil Engineering-ASCE, Nov. 1967, pp. 43-45.
9. ACI Committee 223, "Expansive Cement Concretes - Present State of Knowledge," ACI Journal, Title No. 67-35, Aug. 1970, pp. 583-609.
10. Mindess, S., and Young, J.F., "Concrete," 1981, pp. 38-45
11. Bayasi, S., and Abifaher, R., "Properties of Shrinkage-Compensating Silica Fume Concrete Made with Type K Cement," Concrete International, April 1992, pp. 35-37.
12. Bertero, V., "Curing Effects on Expansion and Mechanical Behavior of Expansive Cement Concrete," ACI Journal, 1967, pp. 84-97.

13. Mehta, P.K., "Mechanism of Expansion Associated with Ettringite Formation," *Cement and Concrete Research*, Vol. 3, 1973, pp. 1-6.
14. Cohen, M.D., and Mobasher, B., "Effects of Sulfate and Expansive Clinker Contents of Expansion time of Expansive Cement Paste," *Cement and Concrete Research*, Vol. 21, 1991, pp. 147-157.
15. Srawley, J.E., and Gross, B., "Compendium," *Engineering Fracture Mechanics*, Vol. 4, 1972, pp. 587-589.
16. Irwin, G.R., "Crack-Extension Force for a Part - Through Crack in a Plate," *Journal of Applied Mechanics*, Dec. 1962, pp. 651-654.
17. Keer, L.M., and Freedman, J.M., "Tensile Strip with Edge Cracks," *Int. J. Engr Sci.*, Vol. 11, 1973, pp. 1265-1275.
18. Grudemo, A., "Microcracks, Fracture Mechanism, and Strength of the Cement Paste Matrix," *Cement and Concrete Research*, Vol. 9, 1979, pp. 19-34..
19. Baokun, Y., Wanchun, W., Lilin, H., Faming, Y., and Fuyin, C., "Proceeding of Second International Symposium on Cement and Concrete," *China Building Materials Academy*, Sept. 5-8, 1989, pp. 31-48.
20. Zheng, L., Xuehua, C., and Mingshu, T., "Hydration and Setting Time of MgO Type Expansive Cement," *Cement and Concrete Research*, Vol. 22, 1992, pp. 1-5.
21. Mehta, P.K., and Hu, F., "Further Evidence for Expansion of Ettringite by Water Adsorption," *Journal of the American Ceramic Society*, March - April 1978, pp. 179-182.
22. Cohen, M.D., and Richards, C.W., "Effects of the Particle Size of Expansive Clinker on Strength - Expansion Characteristics of Type K Expansive Cements," *Cement and Concrete Research*, Vol. 12, 1982, pp. 717-725.
23. Swamy, R.N., and Al-Asali, M.M, "Expansion of Concrete Due to Alkali-Silica Reaction", *ACI Materials Journal*, Title No. 85-M5, Jan.-Feb. 1988, pp. 33-40.
24. Bryant, A.H., and Vadhanavikkit, C., "Creep, Shrinkage-Size, and Age at Loading Effects," *ACI Materials Journal*, Title No. 84-M14, March-April 1987, pp.117-123.

25. Bazant, Z.P., and Kim, J., "Effects of Specimen Size and Shape on Drying Shrinkage of Concrete," *ACI Materials Journal*, Jan.-Feb. 1988, pp. 63-67.
26. ACI Committee 207, "Effect of Restraint, Volume Change, and Reinforcement on Cracking of Mass Concrete," *ACI Materials Journal*, Title No. 87-M31, May-June 1990, pp. 271-295.
27. Dias, W.P., Khoury, G.A., and Sullivan, P.J., "Shrinkage of Hardened Cement Paste at Temperatures up to 670 C(1238 F)," *ACI Materials Journal*, Title No. 87-M22, May-June 1990, pp. 204-209.
28. Smadi, M.M., Slate, F.O., and Nilson, A.H., "Shrinkage and Creep of High, Medium, and Low-Strength Concretes, Including Overloads," *ACI Materials Journal*, Title No. 84-M25, May-June 1987, pp. 224-233.
29. Almudaiheem, J.A., and Hansen, W., "Effect of Specimen Size and Shape on Drying Shrinkage of Concrete," *ACI Materials Journal*, Title No. 84-M16, March-April 1987, pp. 130-134.
30. Hansen, W., and Almudaiheem, J., "Ultimate Drying Shrinkage of Concrete Influence of Major Parameters," *ACI Materials Journal*, Title No. 84-M24, May-June 1987, pp. 217-222.
31. Russell, H.G., "Design of Shrinkage - Compensating Concrete Slabs," *Research and Development Bulletin*, RDO34.01D, 1973.
32. ACI Committee 211, "Standard Practice for Selecting Proportions for Normal, Heavyweight, and Mass Concrete," *ACI 211*. pp. 1-81.
33. Paillere, A.M., Buil, M., and Serrano, J.J., "Effect of Fiber Addition on the Autogenous Shrinkage of Silica Fume Concrete," *ACI Materials Journal*, March-April 1989, pp. 139-144.
34. Almudaiheem, J.A., and Hansen, W., "Prediction of Concrete Drying Shrinkage from Short-Term Measurements," *ACI Materials Journal*, July-August 1989, pp. 401-408.
35. Smadi, M.M., Slate, F.O., and Nilson, A.H., "Shrinkage and Creep of High, Medium, and Low-Strength Concretes, Including Overloads," *ACI Materials Journal*, Title No. 84-M25, May-June 1987, pp. 224-233.

36. Rilem Committee TC69-MMC, "Conclusions Structural Analysis and for Formulation of Standard Design Recommendations on Creep and Shrinkage," *ACI Materials Journal*, Nov.-Dec. 1987, pp. 578-581.
37. Swamy, R.N., and Al-Asali, M.M., "Engineering Properties of Concrete Affected by Alkali-Silica Reaction," *ACI Materials Journals*, Title No. 85-M41, Sept.-Oct. 1988, pp. 367-374.
38. Chatterji, S., and Jeffery, J.W., "A New Hypothesis of Sulphate Expansion," *Magazine of Concrete Research*, Vol. 15, No. 44, July 1963 pp. 83-86.
39. Swayze, M.A., "Volume Changes in Concrete," *Materials Research and Standards*, Sept, 1961, pp. 700-710.
40. Cohen, M.S. and Mather, B., "Sulfate Attack on Concrete - Research Needs," *ACI Materials Journal*, Title No. 88-M9, Jan.-Feb. 1991, pp. 62-69.
41. Wills, M.H., "How Aggregate Particle Shape Influences Concrete Mixing Water Requirement and Strength," *Journal of Materials*, pp. 843-865.
42. Bertero, V., and Brester, B., "A Review of Research on Expansive - Cement Concretes. Part 4: The Behavior of Expansive -Cement Concrete Under Load," pp.493-498.
43. Brown, C.B., and Szabo, Z.S., " A Study of Bond Between Steel and Restrained Expanding Concrete," *Magazine of Concrete Research*, Vol. 20, Nov. 1962, March 1968, pp. 3-12.
44. Monfore, G.E., "Properties of Expansive Cement Made with Portland Cement, Gypsum, and Calcium Aluminate Cement," *Journal of the PCA Research*, May 1964, pp. 2-8 .
45. Li, S., "Expansive Cement Concretes - A Review," *Journal of the American Concrete Institute*, Title No. 62-43, June 1965, pp. 689-706.
46. Bentur, A., and Ish-Shalom, M., "Proposed Mechanism of Ettringite Formation and Expansion in Unrestrained Paste of Pure Expansive Component," *Cement and Concrete Research*, Vol. 4, 1974, pp. 709-721.
47. Bentur, A., and Ish-Shalom, M., "Hydration of Unrestrained Paste of Expansive Component-Results," *Cement and Concrete Research*, Vol. 4, 1974, pp. 519-532.

48. Nalamura, T., Sudoh, G., and Akaiwa, S., "Supplementary Paper 4-74 Mineralogical Composition of Expansive Cement Clinker Rich in SiO_2 and Its Expansibility," pp. 351-365.
49. Cohen, M.D., "Theories of Expansion in Sulfoaluminate - Type Expansive Cements: Schools of Thought," Cement and Concrete Research, Vol. 13, 1983, pp. 809-818.
50. Lobo, C., and Cohen, M.D., "Hydration of Type K Expansive Cement Paste and the Effect of Silica Fume: 1. Expansion and Solid Phase Analysis," Cement and Concrete Research, Vol. 22, 1992, pp. 961-969.
51. Cohen, M.D., and Olek, J., "Procedure for Mixture Proportioning of Type-K Expansive Cement Blends for Use in Shrinkage - Compensation Mortars," ACI Materials Journal, Title No. 88-M59, Sept.-Oct. 1991, pp. 536-543.
52. Mor, A., "High - Performance Concrete Quality Control Tests Challenge Labs," Concrete Construction, May 1992, pp. 363-365.
53. Mor, A., "High Performance Concrete Becoming a Practical Option," Concrete Construction, May 1992, pp. 351-353.
54. Hover, K.C., and Shaeles, C.A., "Influence of Mix Proportions and Construction Operations on Plastic Shrinkage Cracking in Thin Slabs," ACI Materials Journal, Nov.-Dec. 1988, pp. 495-504.
55. Ming-shu, Zheng Liu and Su-feng Ham, "Mechanism of Alkali-Carbonate Reaction: "Proceedings of the 7th International Conference, Ottawa, Canada. 1986 pp.275-279.
56. Grattan-Bellew P.E., and Lefebvre P.J., "Effect of confinement of Deterioration of Concrete Made with Alkali-Carbonate Reactive Aggregate: "Proceedings of the 7th International Conference; Ottawa, Canada. 1986 pp. 280-285
57. Chrysler Corporation, Warren Truck Assembly Plant Paint Sludge Powder Production Flow Chart, 1992.
58. Tlili, A. "Development, Characterization, and Fundamental investigation of Latex-Modified Steel Fiber Reinforced Concrete" - A Ph.D. Dissertation, 1993. Department of Civil and Environmental Engineering, Michigan State University, East Lansing, Michigan 48824.

MICHIGAN STATE UNIV. LIBRARIES



31293010468274

MICHIGAN STATE UNIVERSITY LIBRARIES



3 1293 01046 8282

LIBRARY
Michigan State
University

**PLACE IN RETURN BOX to remove this checkout from your record.
TO AVOID FINES return on or before date due.**

DATE DUE	DATE DUE	DATE DUE
DEC 22 1994 11 15 1994	_____	_____
12 15 1994	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

**FLOWABLE FILL:
A COMPREHENSIVE ASSESSMENT OF MIX DESIGN VARIABLES AND
PROPERTIES**

By

Augustine Okwuegbu

Part II of

A DISSERTATION

**Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of**

DOCTOR OF PHILOSOPHY

Department of Civil and Environmental Engineering

1993

ABSTRACT

FLOWABLE FILL: A COMPREHENSIVE ASSESSMENT OF MIX DESIGN VARIABLES AND PROPERTIES

By

Augustine Okwuegbu

A comprehensive experimental investigation was conducted in order to determine the correlations between various properties of flowable fill and the effects of mix proportioning variables on material properties. The results can be helpful in mix proportioning and evaluation of flowable fill materials for different applications.

It was concluded that the flowable fill structure and strength development mechanism are different from those in conventional concrete. When considering a combination of flowable fill mixtures with and without aggregates, increased compressive strength does not necessarily lead to enhanced impermeability and penetration resistance. California Bearing Ratio (CBR) test results on flowable fill correlate well with penetration resistance and compressive strength.

The effects of aggregates on strength, penetration resistance, California Bearing Ratio and permeability tend to overshadow the corresponding effects of other factors such as cement-ash and water-ash ratios. While aggregates have positive effects on impermeability, penetration resistance and California Bearing Ratio, they tend to damage compressive strength.

Given the complexity of the effects and interactions of different variables in deciding various material properties, one needs to subject a flowable fill produced with relevant materials and mix proportions to the specific set of tests representing actual job requirements in order to make judgment on the merits of flowable fill in a specific application.

**DEDICATED TO:
ALL NIGERIAN CHILDREN**

ACKNOWLEDGMENTS

The research work was performed under the supervision of Dr. Parviz Soroushian. Throughout this work, Dr. Soroushian was a sincere friend more than anything else. The author is indebted to Dr. Soroushian for the achievements that could not be possible without his great leadership and sacrifices.

The author also would like to thank Dr. Thomas Maleck, Dr. Gerald Ludden and Dr. Mark Snyder for their help and precious advice during his doctoral program.

Sincere thanks are also extended for the financial support of the American Public Power Association, the Lansing Board of Water and Light and Michigan State University Power Plant.

Finally, my innermost appreciation to Siavosh Ravanbakhsh for his technical and administrative contributions to this research.

TABLE OF CONTENTS

CHAPTER I

INTRODUCTION	250
------------------------	-----

CHAPTER II

BACKGROUND	254
2.1 INTRODUCTION	254
2.2 MIX PROPORTIONS AND FRESH MIX PROPERTIES	255
2.3 PRODUCTION	257
2.4 PROPERTIES	258
2.5 APPLICATIONS	259
2.5.1 Placing Culverts Pipe Bedding	259
2.5.2 Preventing Erosion	260
2.5.3 Bridge Rehabilitation	261
2.5.4 Filling a Void Along a Wooden Culvert	262
2.5.5 Filling Bridge Abutment Voids	262
2.5.6 Filling of Underground Tanks	262
2.5.7 Protecting Under-Road Petroleum Pipes	263
2.5.8 Road Bed Backfill	263
2.5.9 Abandoned Sewer Fill	264
2.6 COST	265
2.7 QUALITY CONTROL	266
2.8 TRANSPORTATION AND PLACEMENT	266

CHAPTER 3

CORRELATIONS BETWEEN FLOWABLE FILL PROPERTIES	269
3.1 INTRODUCTION	269
3.2 EXPERIMENTAL DESIGN	269
3.3 EXPERIMENTAL RESULTS	273
3.4 ANALYSIS OF RESULTS	283
3.5 COMMENTS ON DURABILITY TESTING OF FLOWABLE FILL . . .	292
3.6 COMPARISON BETWEEN LABORATORY AND FIELD PERFORMANCE	292

CHAPTER 4

EFFECTS OF MIX PROPORTIONS ON MATERIAL PROPERTIES	295
4.1 INTRODUCTION	295
4.2 EXPERIMENTAL DESIGN	295
4.3 RESULTS AND DISCUSSION	295

CHAPTER 5

IDENTIFICATION OF STATISTICALLY INFLUENTIAL PROPORTIONING VARIABLES	305
5.1 INTRODUCTION	305
5.2 EXPERIMENTAL PROGRAM	305

CHAPTER 6

CONCLUSIONS	340
REFERENCES	343

LISTS OF FIGURES

Figure 3.1.	Experimental results (Means and 95% Confidence Interval)	273
Figure 3.1.	(a) Unit Weight	273
Figure 3.1.	(b) Flow	274
Figure 3.1.	(c) Slump	275
Figure 3.1.	(d) Air Content	276
Figure 3.1.	(e) Compressive Strength	277
Figure 3.1.	(f) Penetration Resistance	278
Figure 3.1.	(g) Water Permeability	279
Figure 3.1.	(h) Final Set Time	280
Figure 3.1.	(i) California Bearing Ratio (0.1 in., penetration)	281
Figure 3.1.	(j) California Bearing Ratio (0.2 in., penetration)	282
Figure 3.2.	Correlations Between Different Material Properties	286
Figure 3.2.	(a) Slump Vs. flow	286
Figure 3.2.	(b) 28-Day Penetration Resistance Vs. 28-Day Compressive Strength	286
Figure 3.2.	(c) Air Content Vs. 28-Day Penetration Resistance	287
Figure 3.2.	(d) Water Permeability Vs. 28-Day Compressive Strength	287
Figure 3.2.	(e) Water Permeability Vs. 28-Day Penetration Resistance	288
Figure 3.2.	(f) 28-Day California Bearing Ratio Vs. 28-Day Penetration Resistance 0.1 in)	288
Figure 3.2.	(g) 28-Day California Bearing Ratio Vs. 28-Day Penetration Resistance (0.2 in)	289

Figure 3.2.	(h) 28-Day California Bearing Ratio Vs. 28-Day Compressive Strength (0.1 in)	289
Figure 3.2.	(i) 28-Day California Bearing Ratio Vs. 28-Day Compressive Strength (0.2 in)	290
Figure 3.2.	(j) 28-Day California Bearing Ratio Vs. Water Permeability (0.1 in)	290
Figure 3.2.	(k) 28-Day California Bearing Ratio Vs. Water Permeability (0.2 in)	291
Figure 3.3.	Comparison of Compressive Strengths of Laboratory and Field Specimens.	294
Figure 4.1.	Trends in the Effect of Mix Proportions on Material Properties.	298
Figure 4.1.	(a) Cement Content Vs. 28-Day Compressive Strength	298
Figure 4.1.	(b) Cement Content Vs. Water Permeability	298
Figure 4.1.	(c) Cement Content Vs. 28-Day Penetration Resistance	299
Figure 4.1.	(d) Total Binder Content Vs. 28-Day Compressive Strength	299
Figure 4.1.	(e) Total Binder Content Vs. 28-Day Penetration Resistance	300
Figure 4.1.	(f) Total Binder Content Vs. California Bearing Ratio (0.1 in)	300
Figure 4.1.	(g) Total Binder Content Vs. California Bearing Ratio (0.2 in)	301
Figure 4.1.	(h) Cement-Ash Ratio Vs. Compressive Strength	301
Figure 4.1.	(i) Cement-Ash Ratio Vs. California Bearing Ratio (0.1 in)	302
Figure 4.1.	(j) Cement-Ash Ratio Vs. California Bearing Ratio (0.2 in)	302
Figure 4.1.	(k) Water-Binder Ratio Vs. Air Content	303
Figure 4.1.	(l) Water-Binder Ratio Vs. 28-Day Compressive Strength	303

Figure 4.1.	(m) Water-Binder Ratio Vs. California Bearing Ratio (0.1 in)	304
Figure 4.1.	(n) Water-Binder Ratio Vs. California Bearing Ratio (0.2 in)	304
Figure 5.1.	Trends in variable Effects on compressive strength	314
Figure 5.1.	(a) Three-Factor Interaction Cube Plot	314
Figure 5.1.	(b) Effect of Aggregate-Ash-Ratio and interaction with Water-Ash Ratio	315
Figure 5.1.	(c) Effect of Aggregate-Ash Ratio and Interaction with cement-Ash Ratio	316
Figure 5.2.	Trends in Variable Effects On Set Time	317
Figure 5.2.	(a) Three-Factor Interaction Cube Plot	317
Figure 5.2.	(b) Effect of Water-Ash Ratio and Interaction with Aggregate-Ash Ratio	318
Figure 5.2.	(c) Effect of Water-Ash Ratio and Interaction with Cement-Ash Ratio	319
Figure 5.3.	Trends in Variable Effects on Water permeability	321
Figure 5.3.	(a) Three-Factor Interaction Cube Plot	321
Figure 5.3.	(b) Effects of Aggregate Ash-Ratio and Interaction with Water-Ash Ratio	322
Figure 5.3.	(c) Effects of Aggregate-Ash Ratio and Interaction with Cement-Ash Ratio	323
Figure 5.4.	Trends and Variable Effects on 28-Day Penetration Resistance.	324
Figure 5.4.	(a) Three-Factor Interaction Cube Plot	324
Figure 5.4.	(b) Effect of Aggregate-Ash Ratio and Interaction with Water-Ash Ratio.	325

Figure 5.4.	(c) Effect of Aggregate-Ash Ratio and Interaction with Cement-Ash Ratio.	326
Figure 5.5.	Three-Factor Interaction Cube Plot Representing Variable Effects On Slump.	327
Figure 5.6.	Three-Factor Interaction Cube Plot Representing Variable Effects on Flow.	328
Figure 5.7	Trends in Variable Effects on Air Content.	328
Figure 5.7.	(a) Three-Factor Interaction Cube Plot Representing Variable Effects on Air Content	328
Figure 5.7.	(b) Effect of Aggregate-Ash Ratio and Interaction with Water-Ash Ratio	331
Figure 5.7.	(c) Effect of Aggregate-Ash Ratio and Interaction with Cement-Ash Ratio.	332
Figure 5.8.	Three-Factor Interaction Cube Plot Representing Variable Effects on Unit Weight.	333
Figure 5.9.	Trends in Variable Effects on California Bearing Ratio (CBR, 0.1 in.)	334
Figure 5.9.	(a) Three-Factor Interaction Cube Plot (0.1 in., Penetration)	334
Figure 5.9.	(b) Effect of Aggregate-Ash-Ratio and interaction with Water-Ash Ratio (0. 1 in.)	335
Figure 5.9.	(c) Effects of Aggregate-Ash Ratio and interaction with Cement-Ash Ratio (0.1 in.)	336
Figure 5.10.	Trends in Variable Effects on California Bearing Ratio (CBR, 0.2 in.)	337
Figure 5.10.	(a) Three-Factor Interaction Cube Plot (0.2 in., Penetration)	337
Figure 5.10.	(b) Effect of Aggregate-Ash-Ratio and interaction with Water-Ash Ratio (0.2 in.)	338
Figure 5.10.	(c) Effect of Aggregate-Ash-Ratio and interaction with Cement-Ash Ratio (0.2 in.)	339

LISTS OF TABLES

Table 1.1. Typical analysis of Class F fly ash	252
Table 2.1. Examples of Flowable Fill Mix Proportions	256
Table 2.2. Settlement and cost comparism	264
Table 3.1. Mix Proportions (lb/yd ³)	270
Table 3.2. Coal Ash Properties	270
Table 3.2 (a). Lansing Board of Water and Light Coal Ash Properties. . . .	270
Table 3.2 (b). Lansing Board of Water and Light Coal Ash Properties. . . .	271
Table 3.2 (c). Michigan State University Coal Ash Properties.	271
Table 3.3. Correlations Between Different Material Properties.	285
Table 4.1. Correlation Coefficients Between Mix Proprtions and Material Properties	296
Table 5.1. Fractional Factorial Experiments	306
Table 5.2. Results of 1/2 Fractional Factorial Experiments	308
Table 5.3. 2 ³ factorial design in 2 Blocks.	309
Table 5.4. Results of 2 ³ Factorial Experiments	311

CHAPTER I

INTRODUCTION

Flowable fill is defined as a cementitious material that is in a flowable state at placement and has a relatively low compressive strength (e.g. 120 psi, [8.3 MPa] or less at 28 days). Flowable fill is basically fly ash stabilized with small amounts of Portland cement. Developed for use as a backfill material, it has significantly better physical properties than conventional structural backfill and is generally economically competitive whenever backfill is required. It is used primarily for nonstructural applications below grade where low strength is required; in some cases it is intended to be no stronger than the surrounding soil after it has attained the final set. The type of flowable fill selected should be based on technical and economical considerations for the specific application. [1, 2, 3]

In mix proportioning of flowable fill binding components of the paste (i.e. cement and ash) can be varied in quantity and sand can be introduced in order to achieve the required performance in terms of strength development, self-consolidation, flow behavior characteristics, durability, economy, and ease of removal. Gravel generally has not been a component of the aggregate filler probably because of economical reasons. Throughout the U.S., gravel is usually at a premium while sand is generally in surplus. [4, 5]

Flowable fill has gained appreciable recognition as fill material because of its many inherent advantages. These advantages include flowing placement without

segregation, self-consolidation, controlled density, controlled strength, ease of excavation, and economy. The two primary advantages of flowable fill over traditional methods are its ease of placement and the elimination of settlement. Therefore, in difficult compaction areas or areas where settlement is a concern, flowable fill should be used.

Flowable fill has several characteristics which make it unique and superior to conventional backfilling materials. Flowable fills achieve strengths on the magnitude of 50 to 200 psi (3.5 to 13.8 Mpa) depending on the percentage of Portland cement used. These strengths far exceed most natural soils, therefore the replacement can be made stronger than the surrounding material. Although the strength of the flowable fill will be greater than the surrounding material, the flowable fill can still be excavated with conventional methods were it to become necessary at a later date. The second advantage of flowable fill over a conventional backfill operation is the ease of placement. Conventional backfilling operations require the fill to be hauled to the location, dumped, spread in typically 6 to 12-inch (15 to 30 cm) lifts and compacted possibly adding water to aid in the compaction process. With flowable fill, the ready mix truck arrives, discharges the material and leaves. There is little to no spreading required. In tight locations the conventional spreading and compaction is usually accomplished by hand, making the operation time consuming and costly. [5, 6, 7, 12, 13, 14]

Even though flowable fill has the above virtues, the local market was initially undeveloped due to its high cost (\$34 to \$36 per cubic yard in late 1980's). In order to

compete with sand backfill, the costs had to be driven down. The concrete industry was for a while geared up to handle only low carbon fly ash (generally class F fly ash with typical properties presented in Table 1.1), for the higher quality control needed for concrete mixes. Efforts were thus made to encourage the use of coal ashes with broader ranges of qualities. In order to reduce cost, efforts were also made to develop flowable fill mixtures that utilize fly ash and cement only, without sand. [8, 9]

Table 1.1 Typical analysis of Class F fly ash	
Chemical composition	ASTM C-618 (%)
Silicon Oxide, SiO_2	55
Aluminum oxide, Al_2O_3	26
Iron oxide, Fe_2O_3	6.9
Total, $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	70 Min.
Sulfur trioxide, SO_3	5 Max.
Calcium oxide, CaO	8.7
Magnesium oxide, MgO	1.8
Loss on Ignition (LOI)	12
Available Alkalis, Na_2O	1.5 Max.
Fineness, percent retained on #325 wet sieve	34 Max.

The objective of the work reported herein was to establish a basis for mix design and evaluation of flowable fill. The effects of various mix variables on the end product qualities were assessed, and correlations between different qualities of flowable fill were investigated.

CHAPTER II

BACKGROUND

2.1 INTRODUCTION

A review of literature on flowable fill indicates that the material has been developed mainly through field trials. There is a need to develop a sound technical basis for judicious design and use of the material.

Fuston, Krell and Zimmer (1979) [8] have reported on the development of a flowable fly ash material for backfill applications above or below water. The material developed was composed principally of fly ash, cement (4 to 5 percent by dry weight of fly ash), and an appropriate amount of water to produce the flowability required for a particular application. The strength ranged between 50 and 100 psi (345 to 690 KPa) at 28 days, and the dry density ranged from 70 to 85 lb/ft³ (1120 to 1360 kg/m³). The permeability was approximately 2×10^{-6} in/sec (5×10^{-6} cm/sec), which places it between typical soil backfill materials, (i.e., 10^{-1} to 10^{-3} for sand and 10^{-7} for clay materials). It was also reported that the flowable fly ash, which can be produced in pug mills, turbine-type mixers, and in central concrete mix plants, could be excavated if necessary with stable sidewalls. [10, 41]

Swaffar and Price (1987) [7] have reported on successful use of flowable fill for many applications, including support of a distressed tunnel internally prior to external grouting; they also reported some limitations in using the material, including

handling, mixing, and placing problems, stating: "The new material cannot be handled as conventional fill material ... the contractor must develop a feel for the flowable ash before working (easily) with it ... the finished surface should not be considered a wearing surface. The surface will be slippery during rain-fall, similar to a smooth clay." [1, 15]

2.2 MIX PROPORTIONS AND FRESH MIX PROPERTIES

The mix proportions used for flowable fill are selected to obtain low-strength materials in the 50 to 150 psi (345 to 1,035 KPa) range. This level of strength is similar to that of many recompacted soils and makes it suitable as a backfill material for utility trenches containing ducts, pipes and manholes, other excavations in streets and around foundations, and as fill for abandoned tunnels, sewers, and other underground cavities.

To achieve maximum utilization of the coal combustion by-products, the mixes are usually proportioned using a large quantity of the Class F fly ash, a small amount of cement, and water (see Table 2.1 for typical mix proportions). In most cases, the material is proportioned to allow for easy future removal with ordinary excavating equipment while leaving the trench or excavation walls intact. The strength of the material depends on the particular fly ash being used and on the amount of Portland cement being added. The total amount of water used has some effect, but high water contents cause only a small reduction in strength. [15, 17, 18]

Table 2.1. Examples of Flowable Fill Mix Proportions [13, 28]			
Ash Type	<u>Cement</u> (%) Ash	<u>Sand</u> (%) Ash	<u>Water</u> (%) Ash
Top Ash	3	0	0.544
Top Ash	4	0	0.549
Bottom Ash	4	0	0.719
Top + Bottom Ash	4	0	0.526
Top Ash	5	0	0.435
Bottom Ash	5	0	0.726
Top + Bottom Ash	5	0	0.531
Top Ash	6	0	0.560
Top Ash	8	0	0.570
Top Ash	10	0	0.581
Top Ash	10	5.5	0.327
Top Ash	30	8.5	0.381

As in concrete, slump provides a measure of consistency defined in ACI 116 as "the relative mobility or ability of freshly mixed concrete or mortar to flow". Flowable fill will generally be more flowable than concrete at the same slump primarily because of the lubrication action of the high volume of fly ash spheres. By definition, flowable fill is proportioned to produce very high slumps. General fill applications require a slump range of 6 to 11 in.(15 to 17.5 cm). Where greater flow is required, care should be taken to ensure that adequate fines (generally fly ash) are present to accept the flow without segregation. [5, 19]

Segregation and subsidence are two phenomena that are not a problem with flowable fly ash. Fly ash and cement particles are about the same size and therefore do not separate. Total saturation with water results in free water flowing from the material, not solids. Subsidence does occur, but is almost instantaneous in character. Experience with deep placements and checking the filling under slabs, in sewers, and tunnels confirms that this can be easily accommodated during construction. [9, 20, 21, 22]

It should be noted that a typical mix using 5 percent Portland Cement (by dry weight of the fly ash) has been established. Most soil procedures use the percent of cement as that of the total weight of all the material. This means that a flowable fly ash using 5 percent Portland cement by dry weight of fly ash is approximately equal to 2.3 percent Portland cement based on the total weight of all materials as would normally be reported by a soils laboratory. The point made here is that if the designer compares either costs or materials, all elements must be compared on a basis of equal units.

2.3 PRODUCTION

The flowable fill slurry mixes can be produced at a ready-mixed concrete plant, the mix materials are then loaded into the ready-mix truck in the usual manner using automated plant equipment. However, it is found that a more homogeneous mixture could be obtained if cement was loaded into the ready-mix truck first, then water, and then, after thoroughly mixing these materials, the fly ash. Additional mixing for a minimum of 15 minutes will produce a good, completely mixed, homogeneous slurry. This falls

well within the usually available delivery time of ready-mixed materials. Type I cement is usually used for the mixes. In the placed fill, periodic inspection for condition of set, settlement, bleed water, and shrinkage cracks are necessary to detect settlement, if any, as well as retention of bleed water. [23, 24, 25]

2.4 PROPERTIES

Generally, the 28-day strength of a 2 in. (5 cm) cube will be close to 100 psi (690 KPa) using 5 percent Portland cement by weight of fly ash. This value, which is equivalent to 14,000 lb/ft² (690 kPa), can be compared to engineered backfills where a figure of 3000 to 4000 lb/ft² (21 to 28 MPa) is commonly used. Modulus of rupture tests also prove that the material is capable of slab action, which is why flowable fly ash can bridge over soft boggy soils. [26]

Flowable fly ash develops strength at a reasonable pace. On a hot day, the flowable fly ash has supported the full 10 yd³ (7.6 m³) load of a ready-mixed truck after a few hours. [9]

Although the strength at the flowable fill will be generally greater than the surrounding materials, it can be excavated with conventional methods were it to become necessary. Excavation of materials over a year old in the field with strengths in excess of 300 psi (20.7 Kpa) has been accomplished successfully. [13, 27, 35]

The permeability of the material ranges from 1.9×10^{-6} in/sec to 3.3×10^{-7} in/sec (4.75×10^{-6} cm/sec to 8.25×10^{-6} cm/sec), which places it in the range of a clay

material. When the backfill is used as a liner because of environmental considerations, local materials should be tested at the site. Laboratory tests indicate that a 15 percent Portland cement content (by weight of dry fly ash) can be considered effective for flowable fly ash used in this manner. [13, 28, 41]

Corrosion of embedded iron and steel is controlled by the alkalinity of flowable fill. Most fly ashes are alkaline, a condition that is enhanced by the addition of Portland cement, thus providing an ideal environment to inhibit the corrosion of iron and steel.

2.5 APPLICATIONS

This Section presents many successful applications of flowable fill.

2.5.1 Placing Culvert Pipe Bedding [19, 32, 33]

Since the mid 1970's some county agencies in Iowa have been placing culverts on a controlled low strength material (e.g., flowable fly ash) bedding. This prevents water from getting between the bottom of the pipe and the bedding to erode the pipe support.

Excavation for pipe can be much smaller when using flowable fly ash as the backfill material because it obtains density without compaction. Some engineers specify that the controlled density fill be placed only to the midpoint of the pipe and then, if there is room for compaction equipment, to proceed with conventional compaction and

backfill methods. Other engineers require the flowable fill be placed the entire depth, from the pipe bed to the road bed level.

The Iowa Department of Transportation (IDOT) started specifying flowable mortar backfill materials for culverts in 1979.[4] Their procedure requires that excavation for pipe placement have one-to-one side slopes. An earthen fill is compacted to place the pipe in its flow line and 4-in. (100-mm) subdrain pipe is placed in the flow line. Granular backfill is compacted around the bottom and to one-half the height of the pipe, then flowable mortar is placed to a maximum depth of 5 ft (1.5 m) above the surface of the culvert pipe. The subgrade is brought up to the final grade level by normal compaction methods. [29, 30, 31]

Flowable fly ash is also used for backfilling by Iowa maintenance crews, who specify mixtures ranging from 100 to 500 psi (690 to 3448 KPa). In one area, crews commonly use a 100-psi (690-KPa) mix design for most applications, but reduce the strength when working around water lines because they often have to re-excavate these areas at a later date. This is done even though the 100-psi (690 KPa) flowable fill is readily excavated with a backhoe or an air spade. [34]

2.5.2 Preventing Erosion [4, 36]

Flowable fill was used during the 1980's for two erosion prevention projects in Iowa. The first problem occurred when a 50,000 ft² (4645 m²) department store parking lot was built next to a 12 to 14 ft (3.7 to 4.3 m) deep, V-shaped drainage ditch. The

ditch, 12 to 14 ft. (3.7 to 4.3 m) wide at the top, was lined with riprap and loose rock. The large volume of runoff water from the parking lot soon washed out the loose material in the riprap and the riprap, in turn, washed into the ditch. In rebuilding, the eroded ditch wall was relined with riprap and the top of the ditch was offset from the parallel edge of the parking area by 2 to 3 ft (0.6 to 0.9 m). Flowable fill was placed through the riprap, filling the voids, and a 2 to 3 ft wide by 4 in. thick (0.6 to 0.9 m x 100 mm) flowable fill cap was placed over the riprap. Approximately 40 yd³ (31 m³) of relatively high-strength flowable fill was used for the project. [2, 15]

2.5.3 Bridge Rehabilitation [44, 46]

IDOT has used flowable mortar to save many old or damaged bridges rather than removing and rebuilding them. The method involves positioning culvert pipes or a box culvert under a bridge and building earth dams over the pipes, parallel to the bridge sides. The cavity under the bridges and between the dams is then filled with flowable mortar. The mortar is brought up to within 6 in. (150 mm) of the lowest member of the bridge. After 72 hours, the final placement is made, through 4-in. (100 mm) holes sawed in the bridge deck, bringing the mortar up to the bottom of the bridge deck so the deck becomes a roadway with a solid base.

The guard rail can be removed and the bridge deck can be widened at this point, and shoulder material can be added to make a safety culvert. The entire project can be completed without detouring traffic.

2.5.4 Filling a Void Along a Wooden Culvert [1, 2]

The appearance of a drainage hole led to the discovery of a large void caused by water seeping into, through, and around an old wooden box culvert below the roadway. The void was blocked at both ends of the culvert, denying access for conventional repairs, but the void was easily filled with flowable fill through the original hole from the road surface. Thirty-one yd³ (24 m³) of flowable fill was placed to fill the void.

2.5.5 Filling Bridge Abutment Voids [1, 33]

IDOT has learned the importance of early detection and prompt filling of voids around bridge abutments. One void, under a county bridge abutment near Harlan, Iowa, was estimated to require 30 yd³ (23 m³) of flowable mortar to fill. However, when it was filled a short time later after a heavy rain, it required almost 300 yd³ (230 m³) of flowable fill.

2.5.6 Filling of Underground Tanks [42, 43]

IDOT began a project in 1987 to remove three abandoned underground fuel tanks, but determined that removal would endanger the adjacent maintenance garage. It was then filled with flowable mortar. The two 2000-gal (7.6-m³) and one 1000-gal (3.8-m³) tanks were filled with approximately 26 yd³ (20 m³) of flowable fill. The project was completed at a cost of \$1140, while removing the tanks was estimated to cost approximately \$8000 in 1987.

Since 1987, federal and state rules have been developed to specify underground storage tank closure procedures. Filling in-place tanks with an inert solid material, such as flowable fill, is the alternative to tank removal.

2.5.7 Protecting Under-Road Petroleum Pipes [2, 40]

A roadway over petroleum pipes was needed to accommodate construction traffic for a project at a petroleum plant. To avoid the possibility of contaminating soil and groundwater with leaking petroleum products from ruptured lines, flowable fill was chosen to stabilize and support the piping where the road was to pass over the lines. A rectangular area was excavated, then filled to totally envelope the pipes in a monolithic mass of flowable fill. After a three-day curing period, a 4-in. (100-mm) granular layer was placed over the area as a wearing course for the roadway.

2.5.8 Road Bed Backfill [44, 45]

Flowable fill has been used for bases and subbases. If the proper bearing capacity for a footing cannot be obtained due to poor soil conditions, a "spread footing" of flowable mortar often can solve the problem.

Flowable mortar was used to backfill under the pavement by using the soil outside with a 1:1 slope as a form to contain the flowable mortar. Granular backfill was used for other locations. Still other locations were backfilled with soil in 8-in (20 cm). lifts and compacted with a vibratory plate. The methods used are compared as follows:

Table 2.2 Settlement and cost comparism [9, 15]		
Type of Backfill	Settlement (in.)	Cost (\$)
Soil	1.5	1,790
Granular	0	1,880
Flowable Mortar	0.25	790

This flowable fill was noted to develop a compressive strength of 80 psi (552 Kpa), and it could be designed as a backfill material that does not shrink. Flowable mortar could be removed without a jackhammer.

2.5.9 Abandoned Sewer Fill [19, 45]

Flowable fly ash has been used to fill abandoned tanks, tunnels, and sewers. For the case of filling a sewer, the fill points were placed 250 ft. (75 m) apart and into the sewer. As fill was deposited at one point, the next point served as an air vent. This second point was also used to check on the flow of the material. Test borings made after the filling operation was complete showed the sewer to be full without voids. This same principle has been used to support slabs that have been undermined. In one case, flowable fly ash backfill was used under a slab supporting a fuel tank. The speed of the operation saved the entire plant from a complete shutdown.

2.6 COST

When selecting any construction material for a job, its effect on cost should be considered. Flowable fly ash costs vary with the availability and the market, but the material offers properties that can not be found in others, such as erosion resistance, placability in water, minimum testing costs, elimination of hand labor for compaction, scheduling opportunities, and other advantages. The following case histories are offered to suggest some of the cost items beside those of direct material costs that should be considered.

It is usually the conservation of time and reduced manpower requirements which make flowable fill a less costly alternative. Although flowable fill may cost more on a per cubic yard of material basis, this must be balanced against the reduction in manpower, the reduced equipment rental and the overall time savings. In some circumstances, however, disposal of the excavated material must be included in the cost of the flowable fill. [9, 15]

In a backfilling operation, the use of a cement/fly ash mix that requires no compaction, versus the granular backfill normally used, permits work operations during wet months. This and other factors result in substantial savings in materials, field labor, and time.

2.7 QUALITY CONTROL

The burden of ensuring the proper cement content and mixing should be on the producer. Field controls should include compressive tests on 2-in. (5-cm) cubes and the performance requirement that it supports the weight of construction equipment. Tests on various-sized samples in this strength range indicate that the 2-in. (5-cm) cube strength is comparable to a standard concrete cylinder of 6-in. (15-cm) diameter by 12-in.(30-cm) length and does not require any correction factor as in the case of concrete testing. Although the standard 6-in.(15-cm) cylinder has been used on projects, it has been determined that the standard 2-in. (5-cm) cube is virtually trouble free. The cube requires little storage space and often can be tested on simple soil compression machines. [38, 39, 47]

2.8 TRANSPORTATION AND PLACEMENT

Flowable fly ash has been placed by end-dumping and chuting from ready-mix trucks. The material also has been placed from conveyor lines and by pumping. In its first application, flowable fly ash was end-dumped and then bulldozed into water depths up to 20 ft.(6-m). When placing flowable fill in a cavity under a structure, pipe, or slab, the placement must be continuous or subsidence will take place and it will not be possible to move the material further into the cavity.

The following items are important to remember in the transportation and placement of flowable fill: [4, 11, 16, 33]

- * Pipe culverts may float in flowable fill.
- * Bulkheads are designed for liquid pressure.
- * Delivery should be continuous for filling confined spaces.
- * Drainage of water through the flowable mortar is a must.

The following limitations are exerted on construction by the nature of flowable fly ash [6, 20, 22]:

- * Mortar shall not be placed on frozen ground.
- * Batching, mixing, and placing may be started, if weather conditions are favorable, when the temperature is at least 34 degrees F and rising. At time of placement, mortar must have a temperature of at least 40 degrees F (4.4 degrees C). Mixing and placing shall stop when the temperature is 38 degrees F (3.3 degrees C) or less and falling.
- * Each filling stage shall be as continuous an operation as is practicable.

The following issues also need to be considered in the transportation and construction of flowable fill:

1. The temperature of the flowable fill mix, as manufactured and delivered, shall be at least 50 °F (10 °C).

2. Mixtures shall be transported to the point of placement in a revolving drum mixer or agitator.
3. During placement operations around manholes and in utility trenches, care shall be used to avoid dislocating any pipes due to fluid pressure from the flowable fill by even placing of the material. Any pipes within the backfill area should be considered for securing to avoid initial buoyant effect of flowable fill.
4. When Ready Mixed Flowable Fill is used in pavement cuts, the fill shall be placed to the top of pavement. After setting, the fill is to be removed to the bottom of a concrete pavement patch or to the top of bituminous base course.

As the specific gravity of fly ash varies, it is recommended to be sure and check the yield before using any mix on a project. [11, 12, 16]

CHAPTER 3

CORRELATIONS BETWEEN FLOWABLE FILL PROPERTIES

3.1 INTRODUCTION

Various test methods have been used to evaluate flowable fill. This phase of research was concerned with establishing the correlation between the results of these test methods in order to help identify the necessary tests for comprehensive evaluation of flowable fill.

3.2 EXPERIMENTAL DESIGN

Nine different flowable fill mixtures were tested for various aspects of their performance characteristics. Correlations were established between these characteristics.

The nine mixtures were produced using three different coal ashes and three different mix proportions. The three coal ashes used were:

- * Lansing Board of Water & Light (4/5 fly ash + 1/5 bottom ash)
- * Lansing Board of Water & Light (1/4 fly ash + 3/4 bottom ash)
- * MSU Power Plant (mixture of fly ash and bottom ash)

The three mix proportions are introduced in Table 3.1

Table 3.1. Mix Proportions (lb/yd ³)			
	Mix I	Mix II	Mix III
Cement	35	273	50
Ash	1380	1417	500
Sand	-	-	2600
Water	487	1008	487

Some properties of the Coal ashes used in this investigation are presented in Table 3.2

Table 3.2. Coal Ash Properties

Table 3.2 (a). Lansing Board of Water and Light Coal Ash Properties.	
Property	LBWL (20% Bottom Ash)
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	85.85
SO_3	0.63
Loss on Ignition (LOI)	9.52
Specific Gravity	2.18
Fineness	23.7

Table 3.2 (b). Lansing Board of Water and Light Coal Ash Properties.	
Property	LBWL (75% Bottom Ash)
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	74.85
SO_3	0.71
Loss on Ignition (LOI)	10.5
Specific Gravity	2.27
Fineness	28.5

Table 3.2(c) Michigan State University Coal Ash Properties.	
Property	MSU
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	80
SO_3	4.51
Loss on Ignition (LOI)	9.8
Specific Gravity	2.10
Fineness	20.7

The nine flowable fill mixtures considered were subjected to the following tests: unit weight (ASTM C-29), air content (ASTM C-231), slump (ASTM C-143), flow (ASTM C-230), set time (ASTM C-403), water permeability, California Bearing Ratio (ASTM D-1883, see Appendix I) with 0.1 in and 0.2 in (2.54 and 3.08 mm) penetration, and penetration resistance at 28 days (ASTM C-403, see Appendix I, modified in the sense that the test was performed at 28 days of age and the resistance to penetration was recorded).

3.3 EXPERIMENTAL RESULTS

The experimental results are presented in the following Figures 3.1.

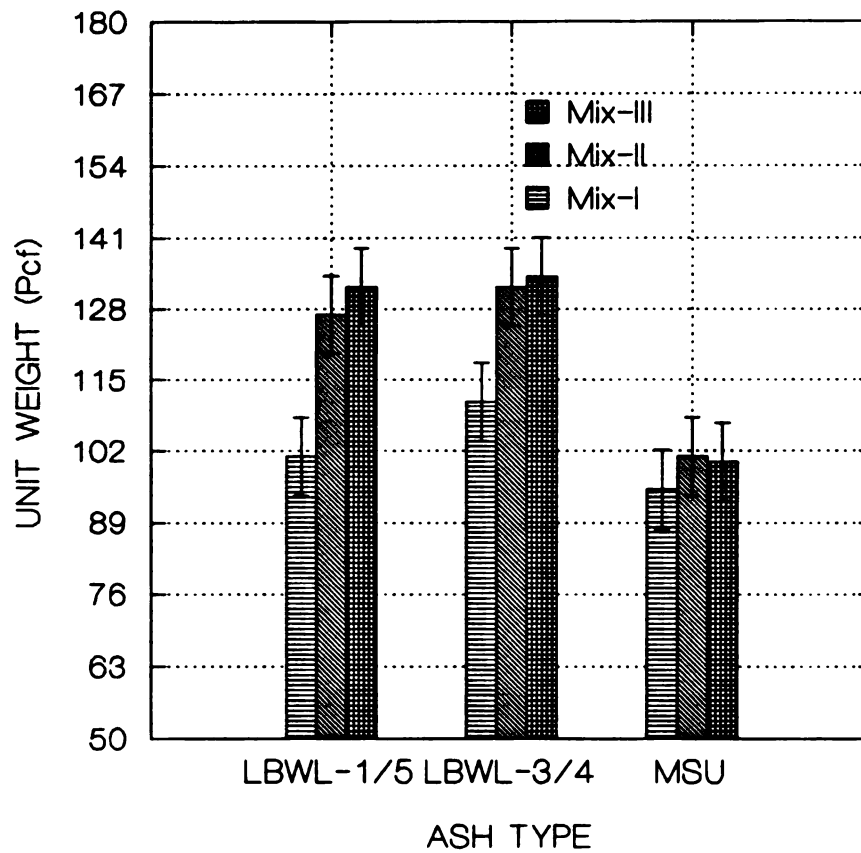


Figure 3.1 (a). Unit Weight

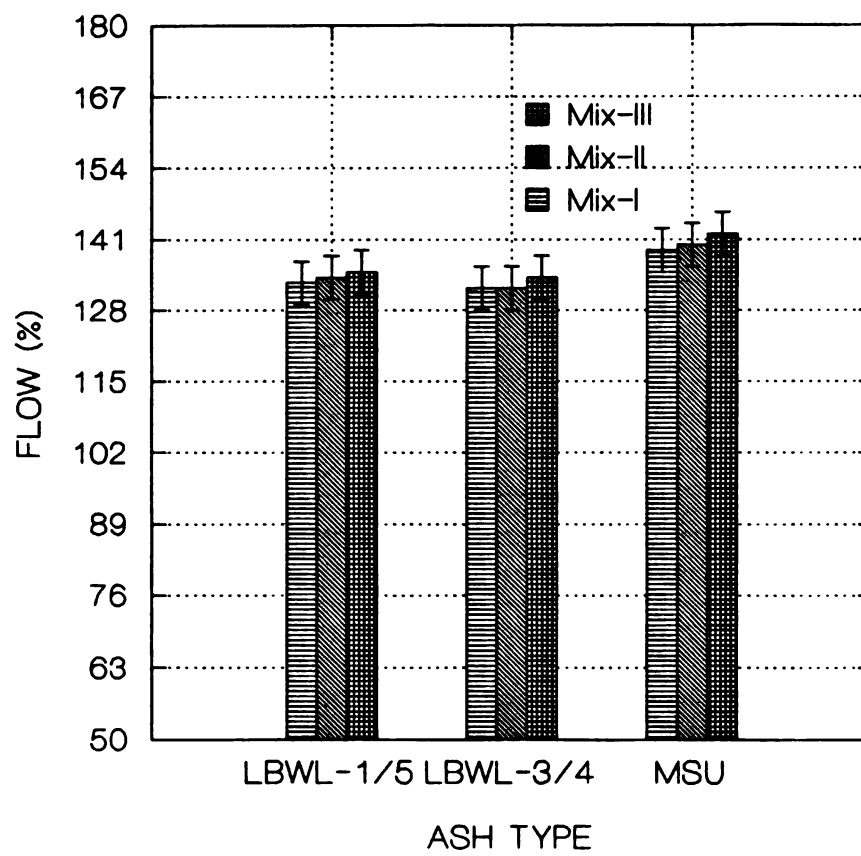


Figure 3.1 (b). Flow

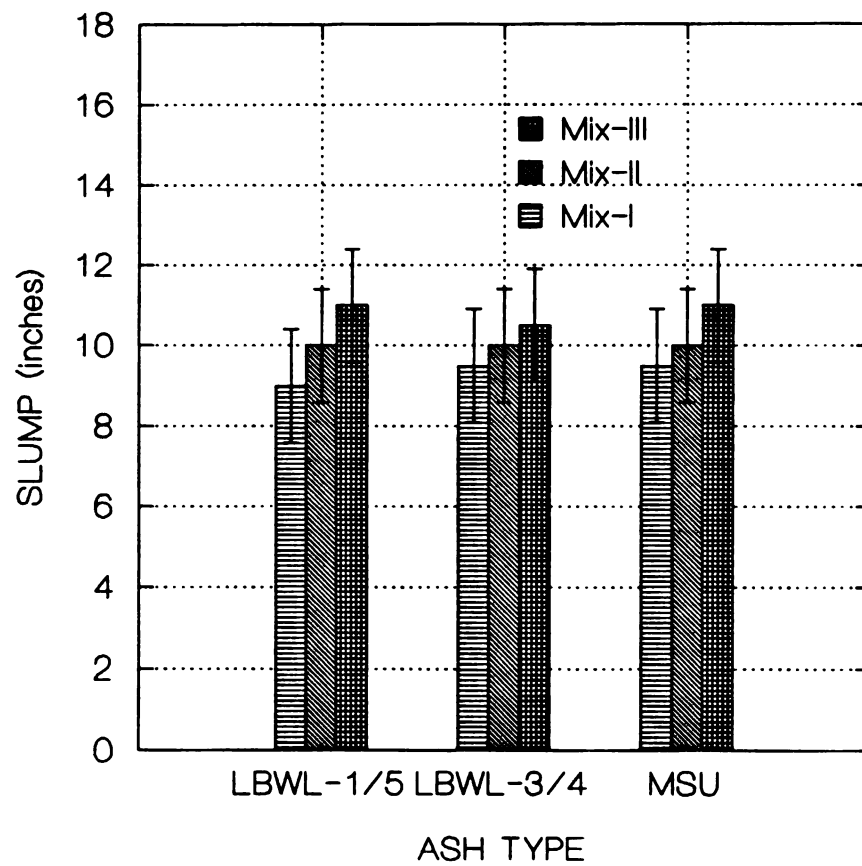


Figure 3.1 (c). Slump

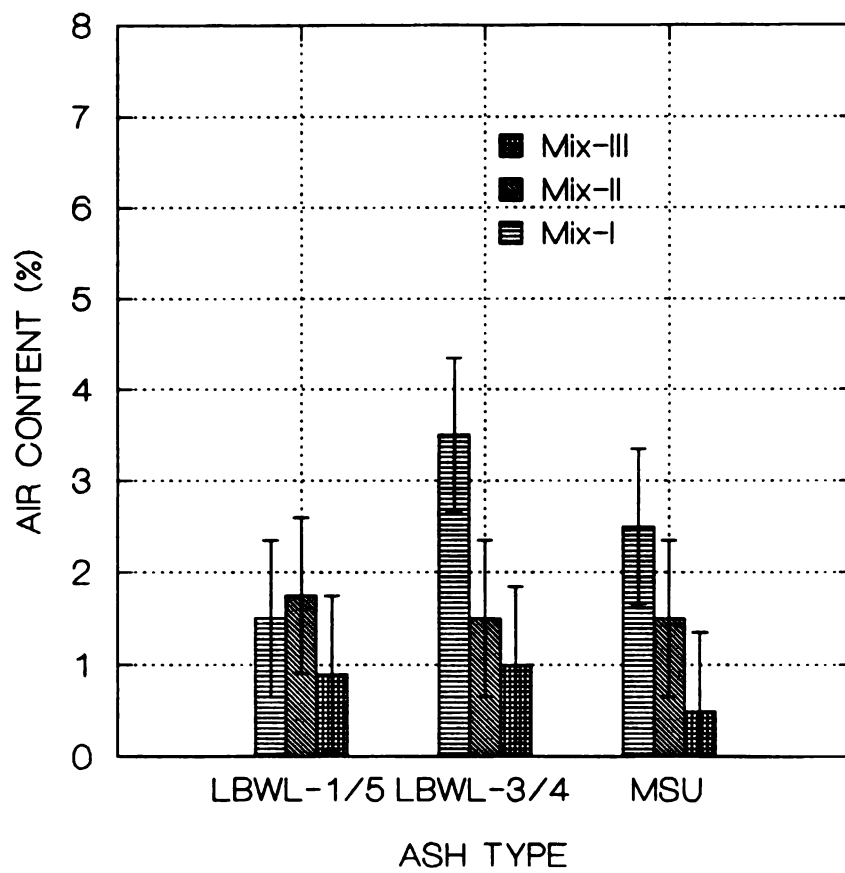


Figure 3.1 (d). Air Content

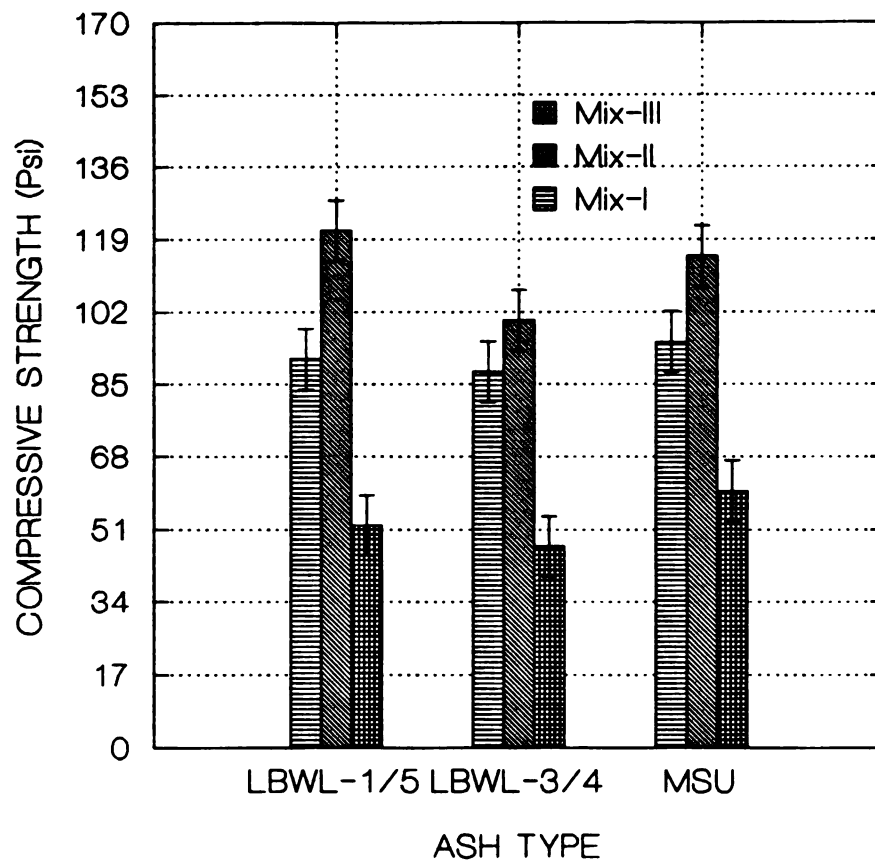


Figure 3.1 (e). Compressive Strength

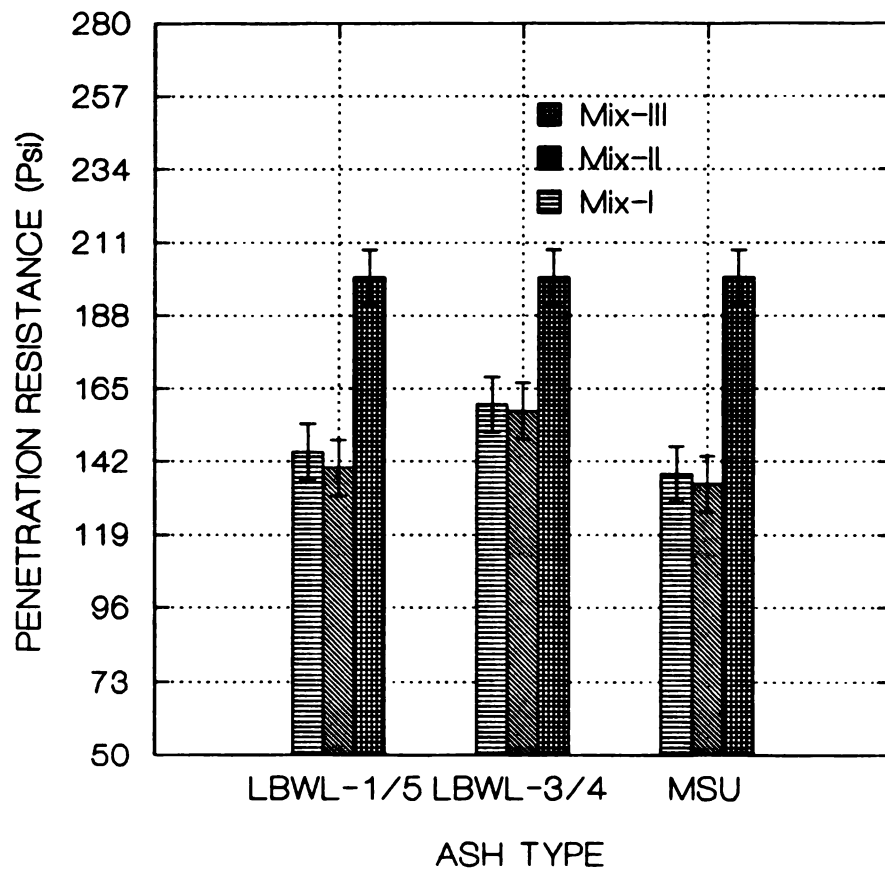


Figure 3.1 (f). Penetration Resistance

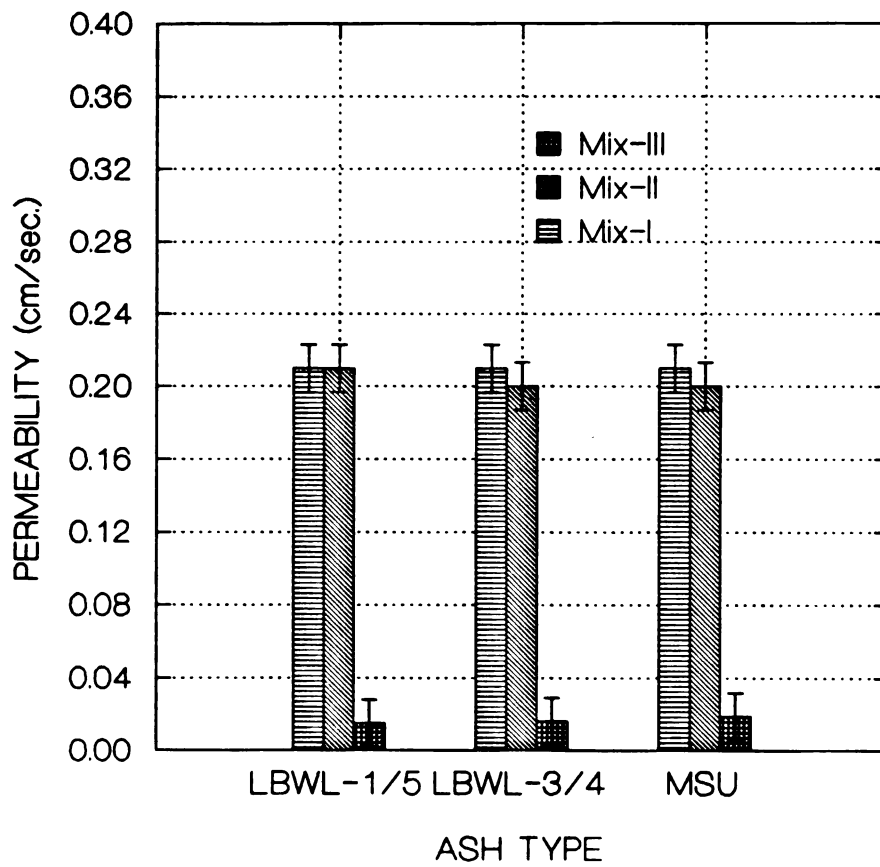


Figure 3.1 (g). Water Permeability

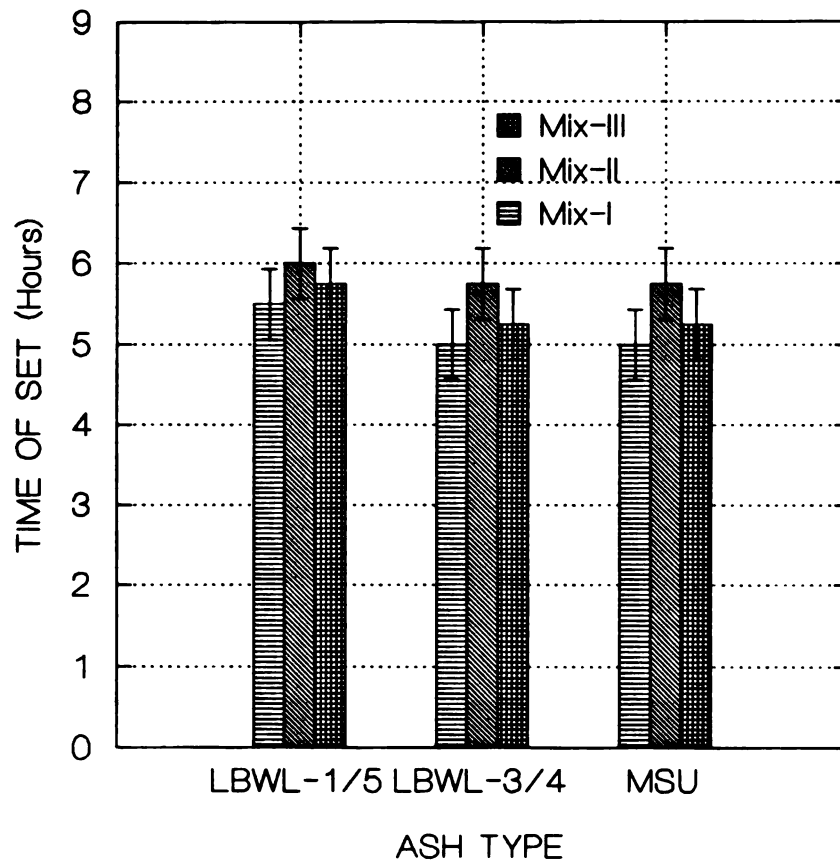


Figure 3.1 (h). Final Set Time

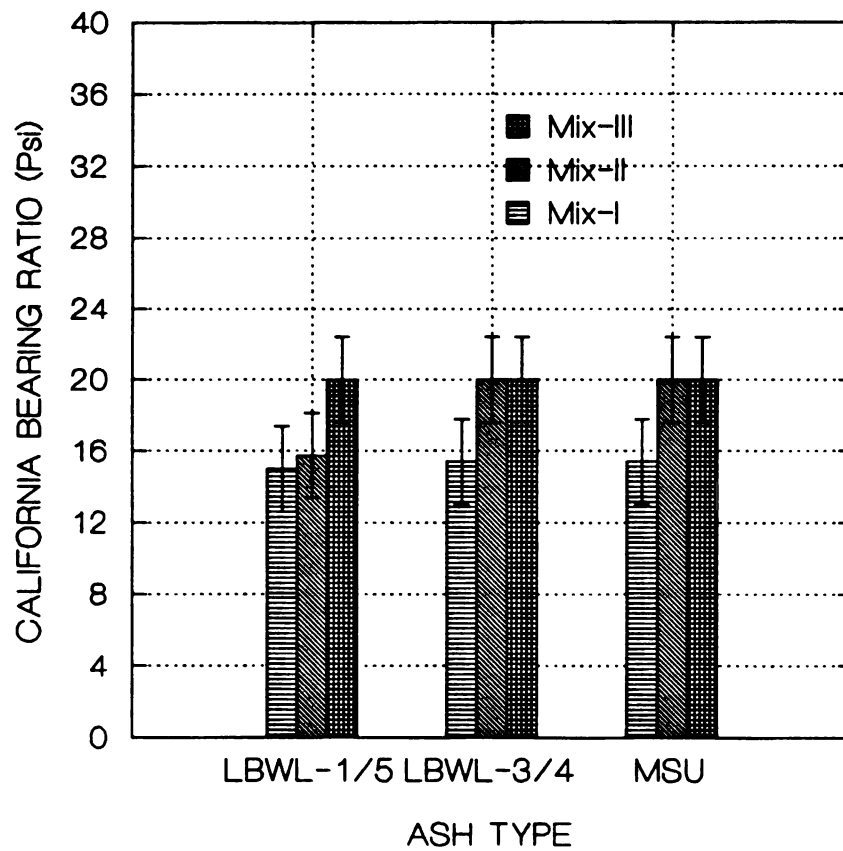


Figure 3.1 (i). California Bearing Ratio (0.1 in., penetration)

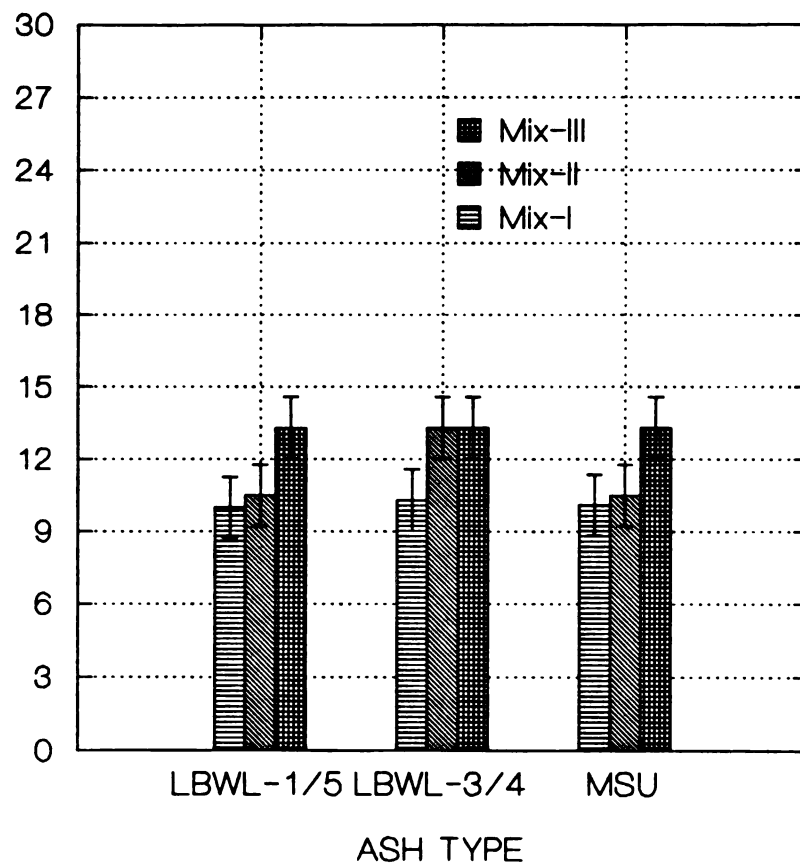


Figure 3.1 (j). California Bearing Ratio (0.2 in., penetration)

Figure 3.1. Experimental results (Means and 95% Confidence Levels)

Simple observation of the test results presented in Figure 3.1 suggests that coal ashes with diverse qualities and different combinations and fly ash and bottom ash produced flowable fills with comparable performance characteristics.

3.4 ANALYSIS OF RESULTS

The correlation coefficients presented in Table 3.3 have been derived based on the experimental results of Figure 3.1. Correlation coefficients (absolute values) greater than 0.4 and 0.7 reflect strong and very strong correlations, respectively.

An analysis of Table 3.3 suggests that:

- * Slump and flow of flowable fill are not strongly correlated (see Figure 3.2a).
- * 28-day penetration resistance of flowable fill is not strongly related to compressive strength (see Figure 3.2b).
- * Air content of flowable fill has a strong negative correlation with its 28-day penetration resistance (see Figure 3.2c).
- * There is no strong correlation between water permeability and strength or 28-day penetration resistance of flowable fill (Figure 3.2d & 3.2e).
- * Early-age compressive strength (say at 3 or 7 days of age) may not represent material properties at later ages (say 28 days).
- * California bearing ratio correlates well with 28-day penetration resistance (Figures 3.2 f & g), compressive strength (Figure 3.2 h & i), and water permeability (Figure 3.2 j & k).

The conclusions described above mark the differences between flowable fill and conventional concrete materials. Unlike conventional concrete, increased strength does not necessarily leads to reduced permeability. In notation used to present the permeability test results, E-7 and E-8 mean 10^{-7} and 10^{-8} respectively. Also, 28-day penetration resistance (suggested by some as a measure of flowable fill quality) does not represent the strength of the material.

Table 3.3. Correlations Between Different Material Properties.

	Unit Wt.	Air Content	Slump	Flow	Time of Set	Perm	CBR (0.1)	Pen
Unit Wt.	1	.424*	-.280	-.613*	.464*	-.409*	.148	.464
Air Cont.	.424*	1	-.696*	-.315	.400*	.299	-.717**	.509*
Slump	-.280	-.696*	1	.379	.190	.190	.818**	.639*
Flow	-.613*	-.315	-.315	1	-.170	-.212	.484*	.142
Time of Set	.464*	-.400*	.196	-.170	1	-.40*	.004	.343
Perm	-.409*	.299	-.058	.212	.40*	1	.072	.043
CBR (0.1)	.148	-.717**	.818**	.484*	.004	.072	1	.981**
Pen	.476*	-.590*	.639*	.142	.343	.043**	.981**	1
COMPRESSION								
1 Day	-.40*	.254	.254	-.369	.286	.379	-.748**	.199
3 Days	-.226	.385	-.501*	-.501*	.036	.478*	-.572*	.151
7 Days	-.315	.495*	-.646*	-.043	.359	-.036	-.698*	-.286
28 Days	-.269	.495*	-.615*	-.065	.391	-.112	.733**	-.371

* Strong Correlation

** Very Strong Correlation

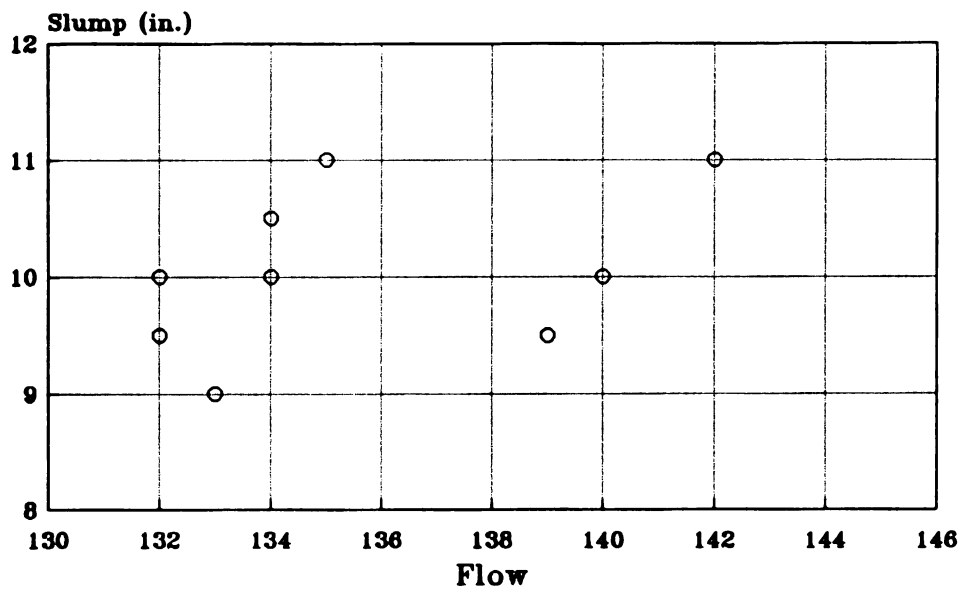


Figure 3.2 (a). Slump Vs. flow

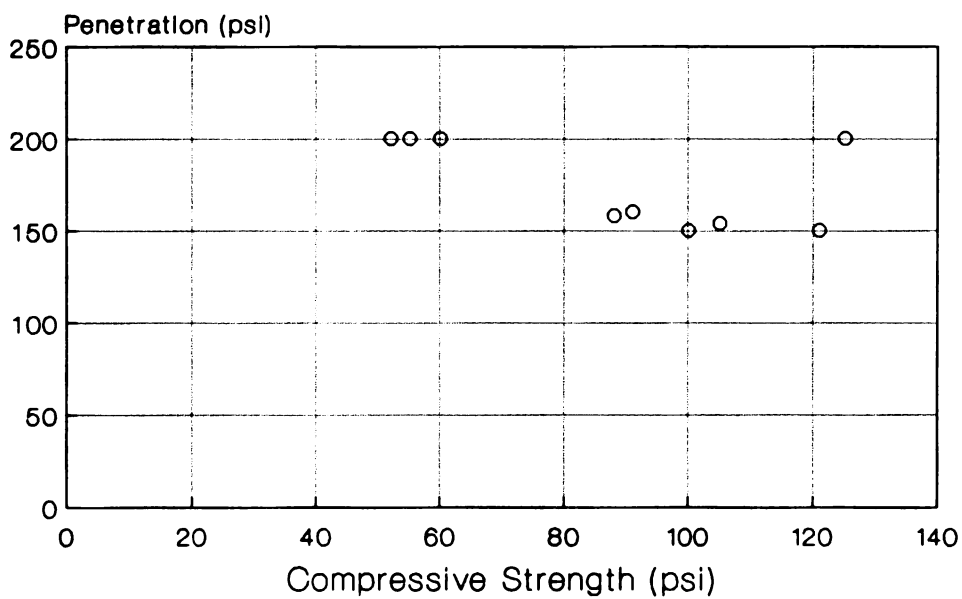


Figure 3.2 (b). 28-Day Penetration Resistance Vs. 28-Day Compressive Strength

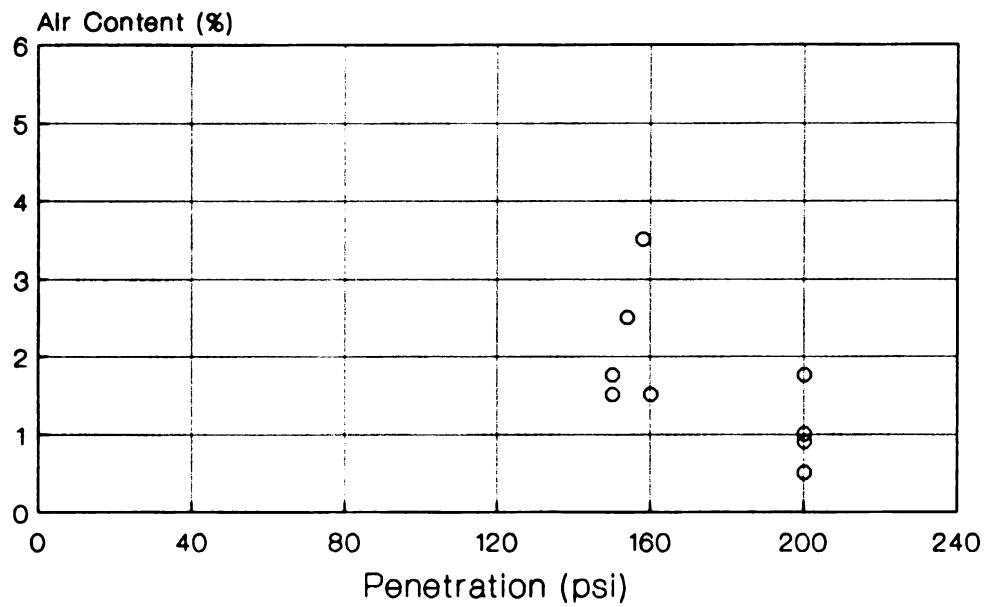


Figure 3.2 (c). Air Content Vs. 28-Day Penetration Resistance

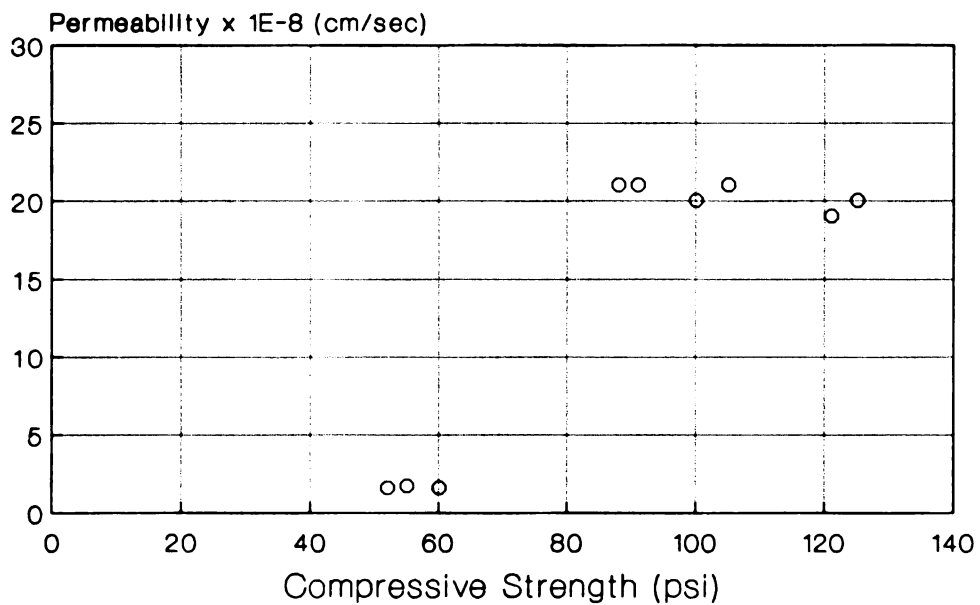


Figure 3.2 (d). Water Permeability Vs. 28-Day Compressive Strength

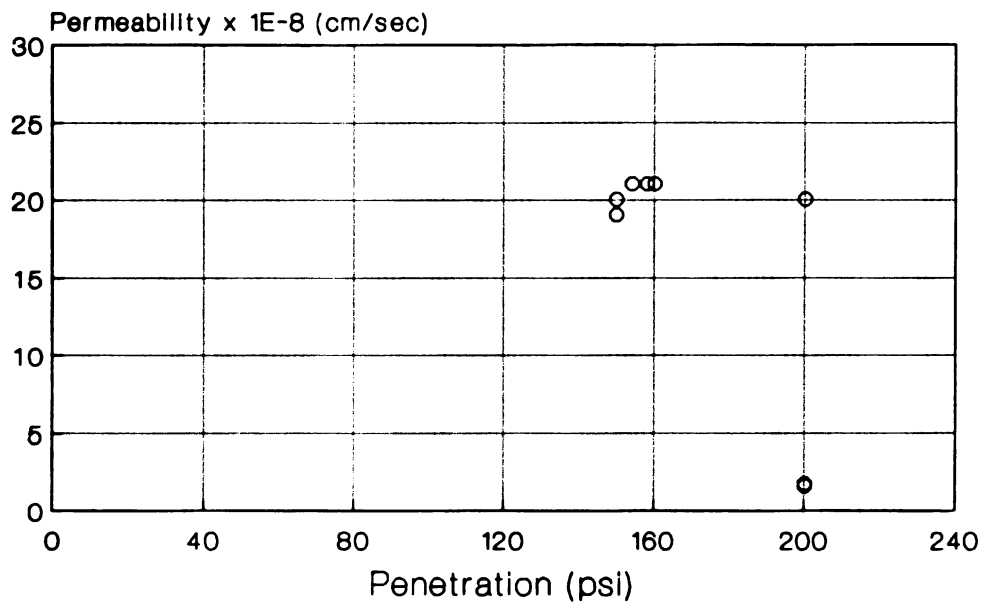


Figure 3.2 (e). Water Permeability Vs. 28-Day Penetration Resistance

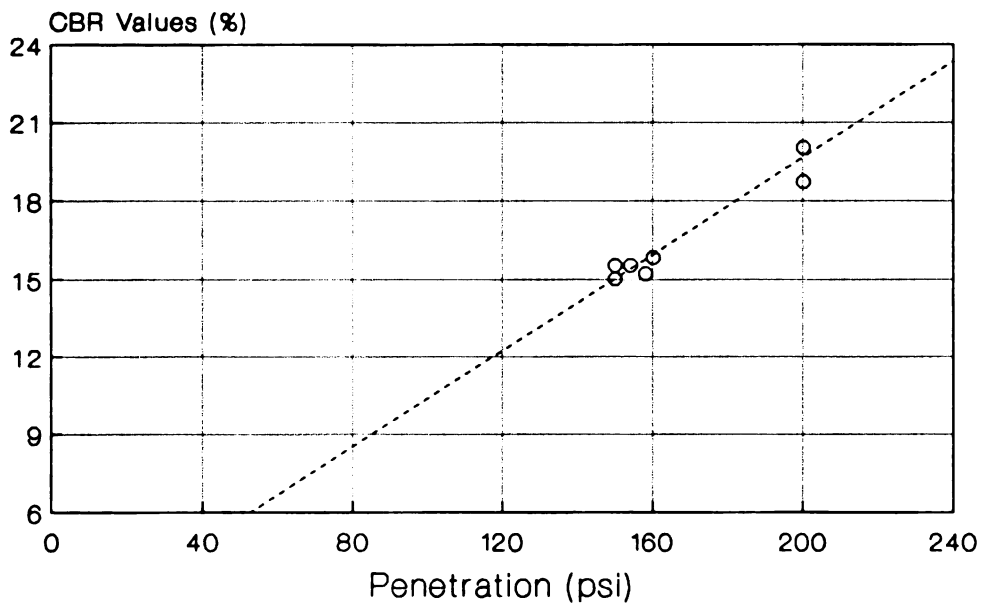


Fig. 3.2 (f). 28-Day California Bearing Ratio Vs. 28-Day Penetration Resistance (0.1 in)

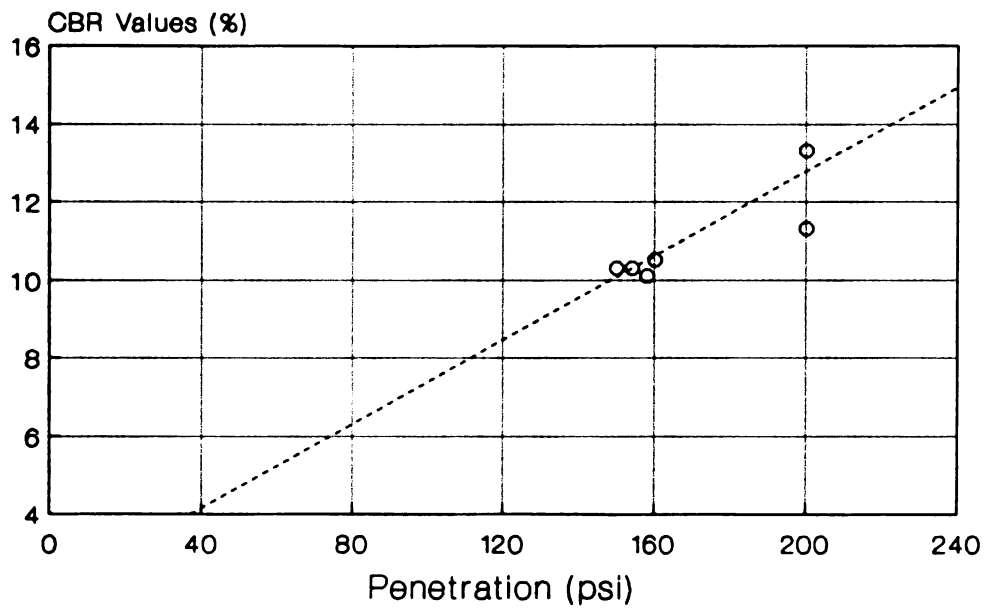


Fig 3.2 (g). 28-Day California Bearing Ratio Vs. 28-Day Penetration Resistance (0.2 in)

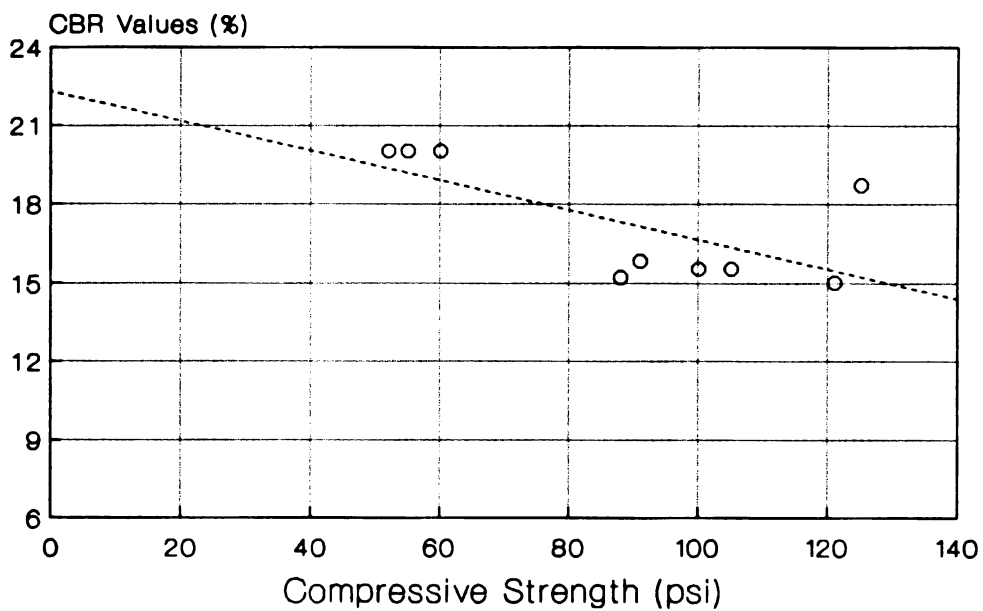


Fig. 3.2 (h). 28-Day California Bearing Ratio Vs. 28-Day Compressive Strength (0.1 in)

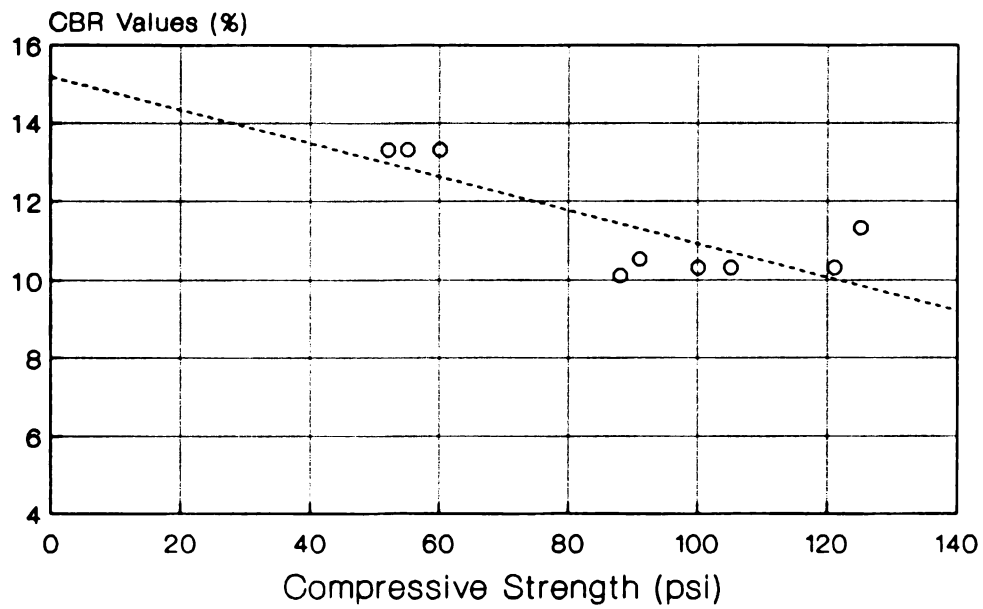


Fig. 3.2 (i). 28-Day California Bearing Ratio Vs. 28-Day Compressive Strength (0.2 in)

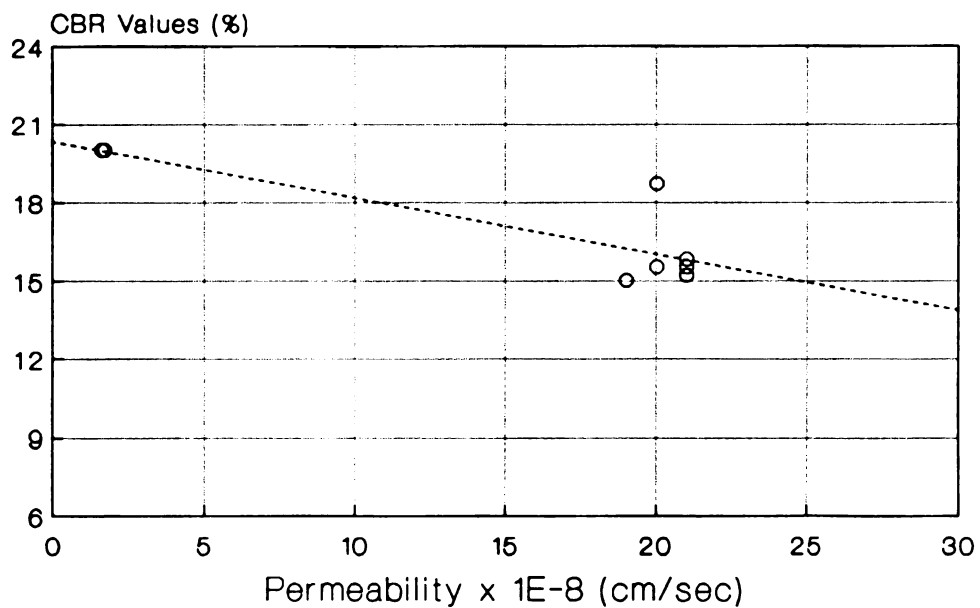
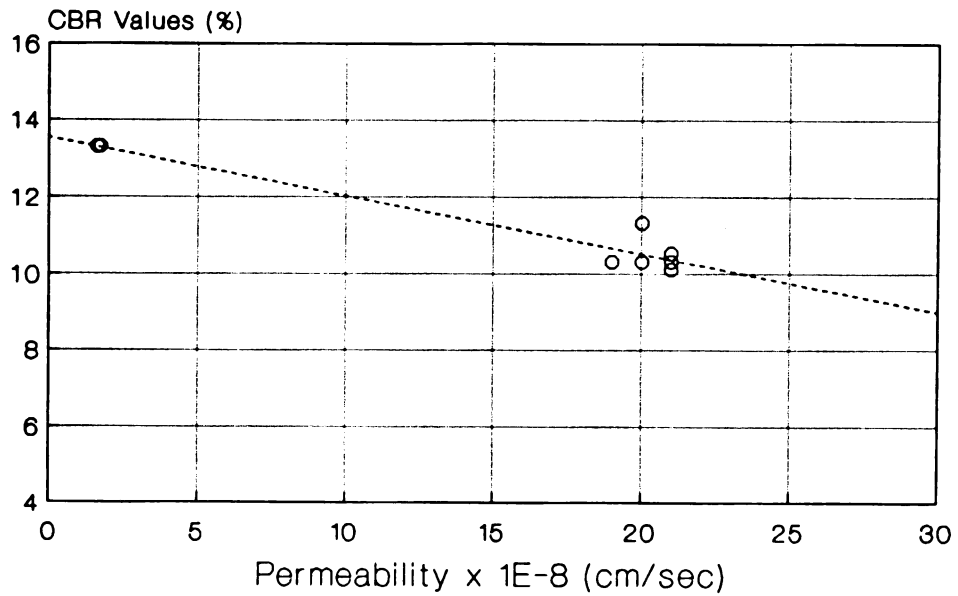


Figure 3.2 (j). 28-Day California Bearing Ratio Vs. Water Permeability (0.1 in)



(k) 28-Day California Bearing Ratio Vs. Water Permeability (0.2 in)

Figure 3.2. Correlations Between Different Material Properties.

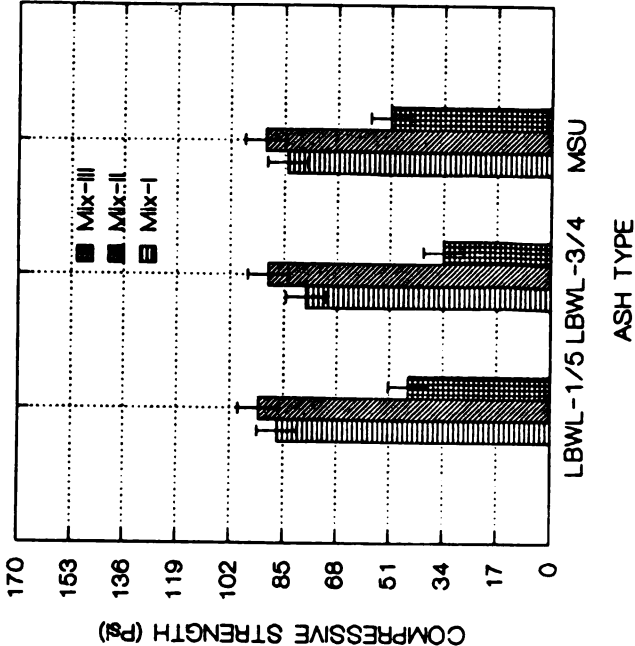
3.5 COMMENTS ON DURABILITY TESTING OF FLOWABLE FILL

Attempts were made to subject the flowable fill materials with mix proportions given in Table 3.1 to the following concrete durability tests: freeze-thaw (ASTM C-666 Procedure B: freezing and thawing in water) and abrasion resistance (ASTM C-944). None of these tests were found to be applicable to flowable fill. Under freeze-thaw test conditions of ASTM C-666, the flowable fill specimens deteriorated very rapidly after the first few cycles of freezing and thawing under water (durable concrete is expected to stand close to 300 cycles of freezing and thawing). Under the abrasive action specified in ASTM C-944 for concrete, the flowable fill specimens were fully abraded before the test could be completed. Obviously, flowable fill can not be tested as concrete which is expected to be durable when it is directly exposed to severe external effects.

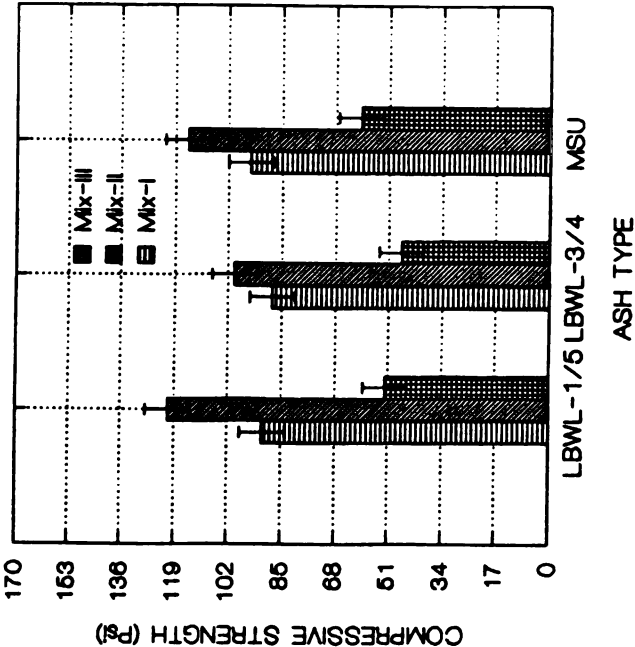
3.6 COMPARISON BETWEEN LABORATORY AND FIELD PERFORMANCE

Preparation of laboratory samples involves molding of small specimens while in field conditions flowable fill is generally placed in massive volumes. Also, exposure (curing) conditions are different in field. In order to investigate the effect of field placement and curing conditions, three 2 x 2 feet (0.609-m x 0.609-m) slabs with a thickness of 51 mm (2-in.) were placed outdoors in East Lansing in June 1993 with the three flowable fill mixtures of Table 3.1. The small thickness of slab was chosen to maximize any adverse effects of the outside environment. After 28 days (during which the slabs were subjected to several cycles of rain and sunshine), nine 51-mm (2-in) cube

specimens were cut out of each slab, and were tested for compressive strength. The compressive strength test results after 28 days of laboratory and field exposures are compared in Figure 3.3. Somewhat lower compressive strengths were obtained from the field specimens; this may be attributed to the fact that the thin slabs placed in the field could be adversely influenced by rain or repeated cycles of rain and heat. The damage is expected to be less in thicker placements of flowable fill.



(a) Laboratory



(b) Field

Figure 3.3. Comparison of Compressive Strengths of Laboratory and Field Specimens.

CHAPTER 4

EFFECTS OF MIX PROPORTIONS ON MATERIAL PROPERTIES

4.1 INTRODUCTION

Broad ranges of mix proportions have been used for flowable fill. These mixes are with or without sand, and have various cement/ash ratios; water content has been adjusted to produce the required flowability. It is not correct to assume that the relationships between mix proportions and material properties in the case of flowable fill are the same as those for concrete; this is due to the fact the mechanisms of strength development are different in flowable fill than in concrete. It is the intent of this phase of research to determine the trends in the effects of different mix proportioning variables on flowable fill properties. These trends would help refine the mix proportions of flowable fill for satisfying specific requirements.

4.2 EXPERIMENTAL DESIGN

The fly ash types, flowable fill mix proportions and experimental methods used in this phase of research are the same as introduced in Section 3.2 of Chapter 3.

4.3 RESULTS AND DISCUSSION

For the nine mixtures introduced earlier (Table 3.1, with three ash types), Table 4.1 presents the correlation coefficients between various mix proportioning

variables and different properties of flowable fill.

Table 4.1. Correlation coefficients between mix proportions and material properties.				
	Water/Binder	Cement Content	Binder Content	Cement/Ash
Unit Weight	0.514 *	0.270	-0.215	0.449 *
Air Content	-0.815 **	-0.007	-0.734 **	0.370
Slump	-0.954 **	-0.037	0.599 *	0.355
Flow	0.275	-0.052	-0.244	0.061
Time of Set	0.268	0.076	0.295	0.800 **
Permeability	-0.284	-0.380	0.458 *	0.458 *
CBR (0.1 in Penetration)	0.859 **	-0.246	-0.818 **	-0.494 *
CBR (0.2 in., Penetration)	0.907 **	-0.350	-0.923 **	-0.464 *
Penetration	0.709 **	0.289	-0.722 **	-0.722 **
COMPRESSION				
1-Day	-0.436 *	0.683 *	0.782 **	0.451 *
3-Days	-0.520 *	0.787 **	0.915 **	0.513 *
7-Days	-0.674 *	0.697 *	0.973 **	0.370
28-Days	-0.650 *	0.709 **	0.963 **	0.390

* Strong Correlation

** Very Strong Correlation

An analysis of Table 4.1 suggests that:

- * Increased cement content leads to an increase in compressive strength (Figure 4.1a), some reduction in water permeability (Figure 4.1b), but not much increase in penetration resistance (Figure 4.1c) and no improvement in California Bearing Ratio.
- * Increased total binder content causes an increase in compressive strength (Figure 4.1d); penetration resistance and California Bearing Ratio, however, tend to drop with increasing binder content (Figure 4.1 e, f, g). This may indicate that the presence of sand (reduced binder content) damages compressive strength but actually increases penetration resistance and California Bearing Ratio.
- * The effect of cement/ash ratio on compressive strength (Figure 4.1 h) is less obvious than the corresponding effect of cement content (Figure 4.1 a) and total binder content (Figure 4.1 d). This is also true for the effect of cement-ash ratio on California Bearing Ratio (Figures 4.1 i and j).
- * Increased water-binder ratio leads to reduced fresh mix air content (very strong correlation, Figure 4.1 k) and somewhat reduced compressive strength (Figure 4.1l). While California Bearing Ratio tends to increase with increasing water-binder ratio (Figure 4.1 m and n), the corresponding effects of sand in flowable fill may have overshadowed the effects of water-binder ratio.

One general conclusion that may be derived from the results presented in Table 4.1 and Figure 4.1 is that the mix proportioning variables influence flowable fill qualities in a different way than they influence concrete properties. This suggests that the mechanisms of structural development in flowable fill are different from those in concrete.

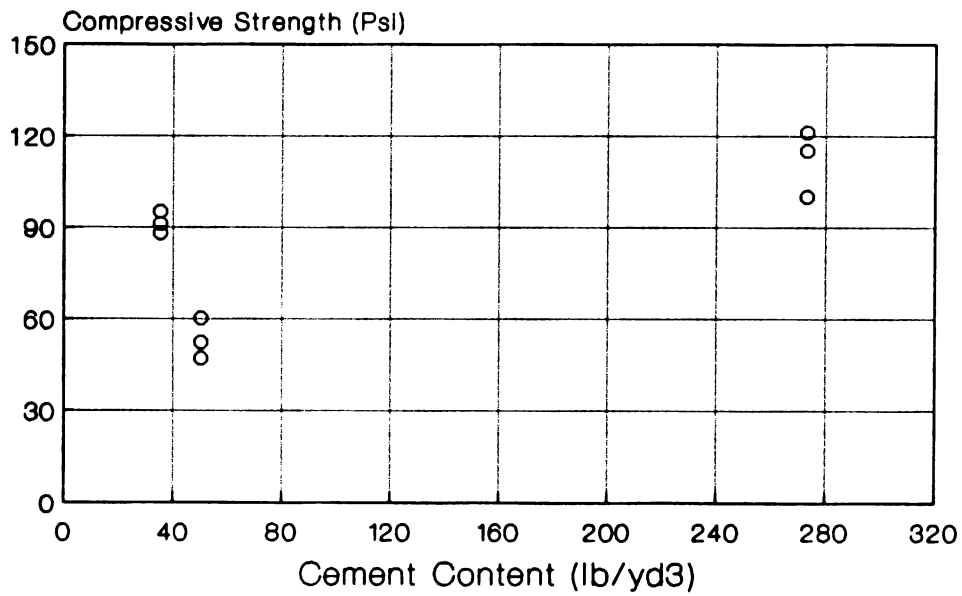


Figure 4.1 (a). Cement Content Vs. 28-Day Compressive Strength

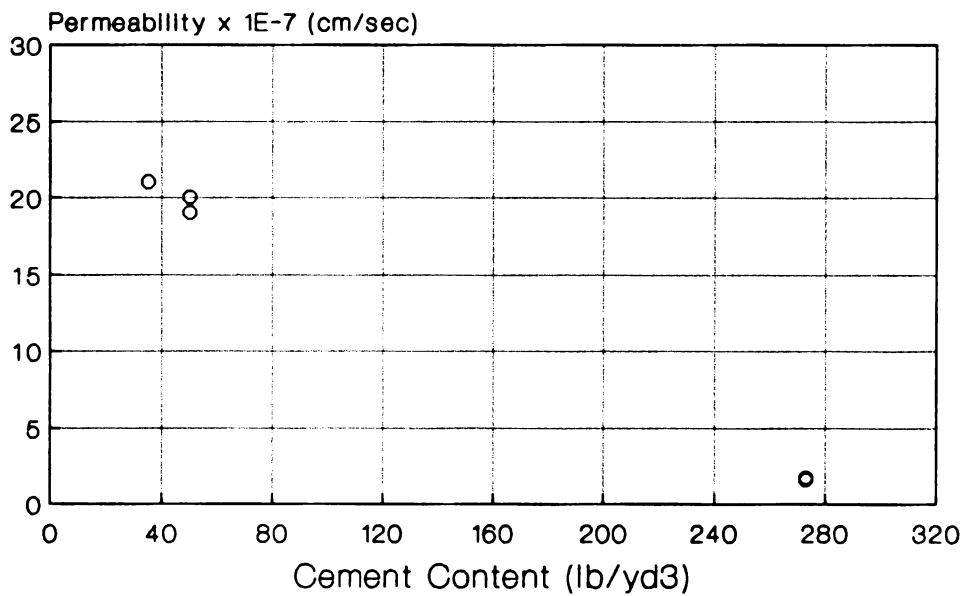


Figure 4.1 (b). Cement Content Vs. Water Permeability

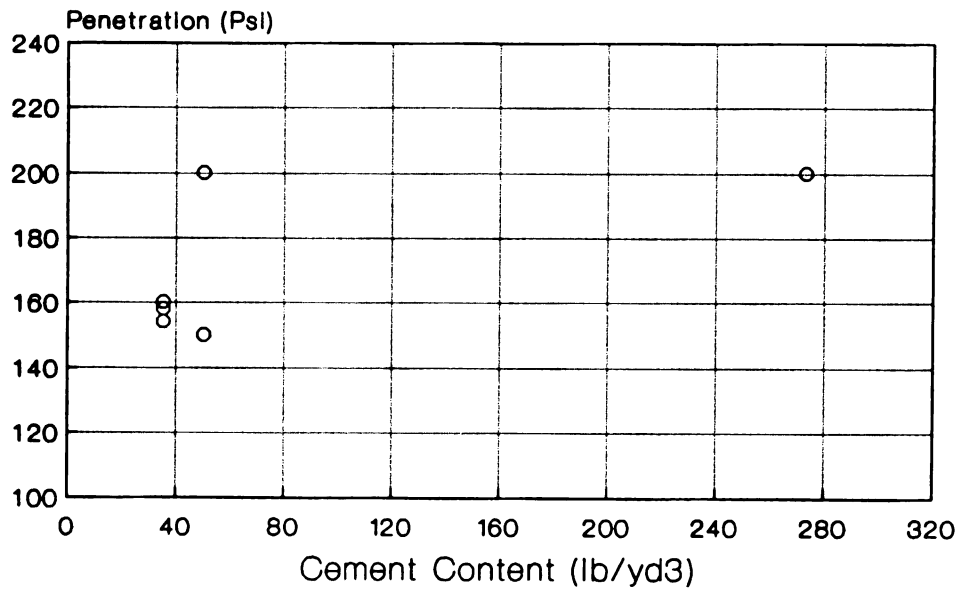


Figure 4.1 (c). Cement Content Vs. 28-Day Penetration Resistance

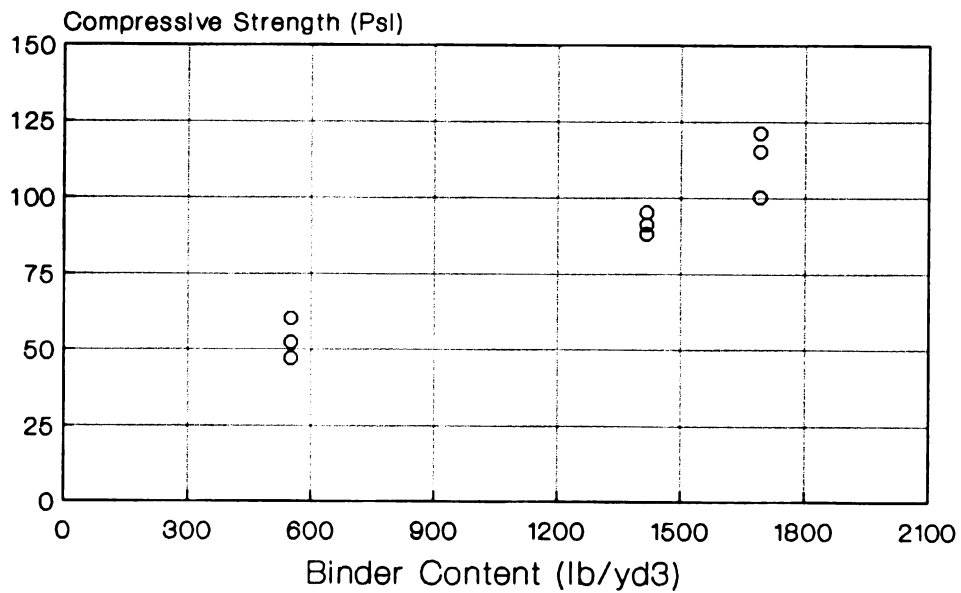


Figure 4.1 (d). Total Binder Content Vs. 28-Day Compressive Strength

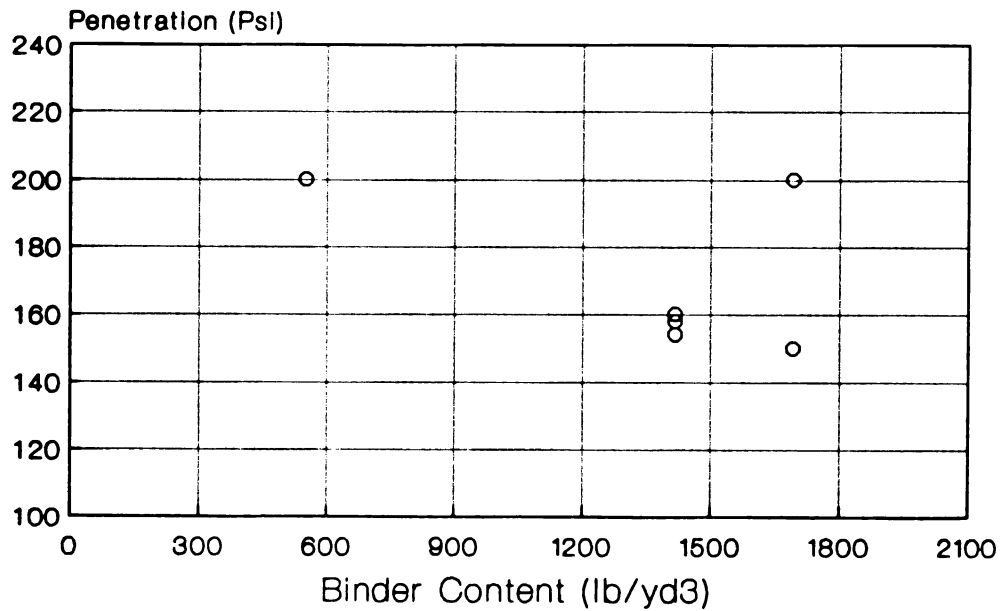


Figure 4.1 (e). Total Binder Content Vs. 28-Day Penetration Resistance

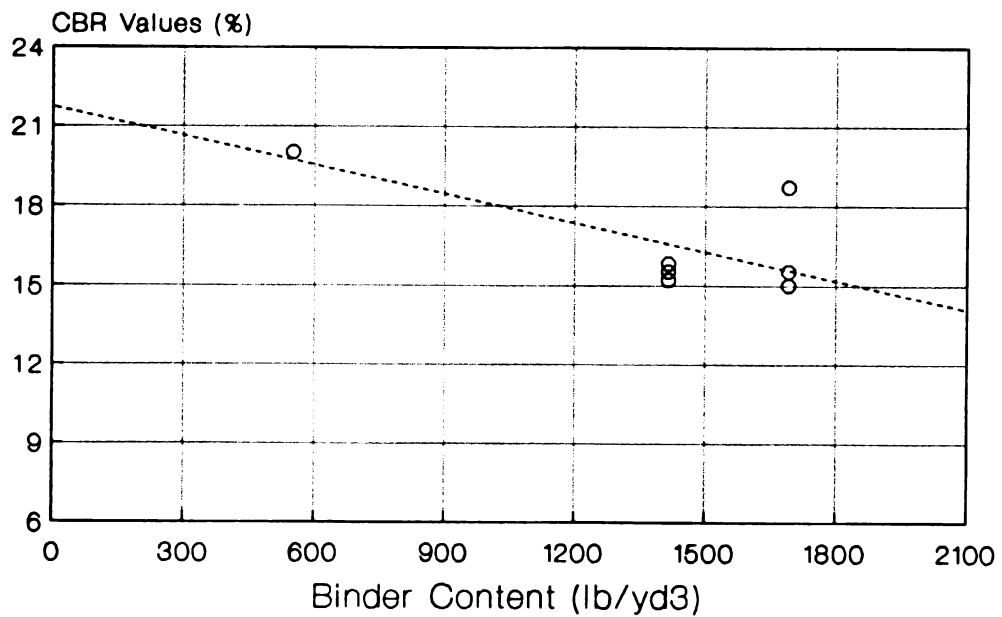


Figure 4.1 (f). Total Binder Content Vs. California Bearing Ratio (0.1 in)

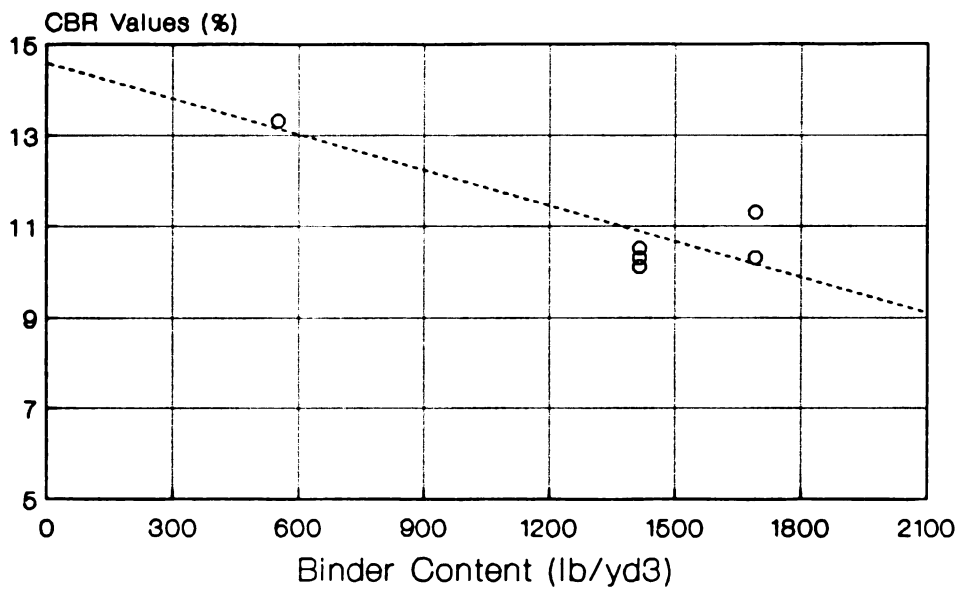


Figure 4.1 (g). Total Binder Content Vs. California Bearing Ratio (0.2 in)

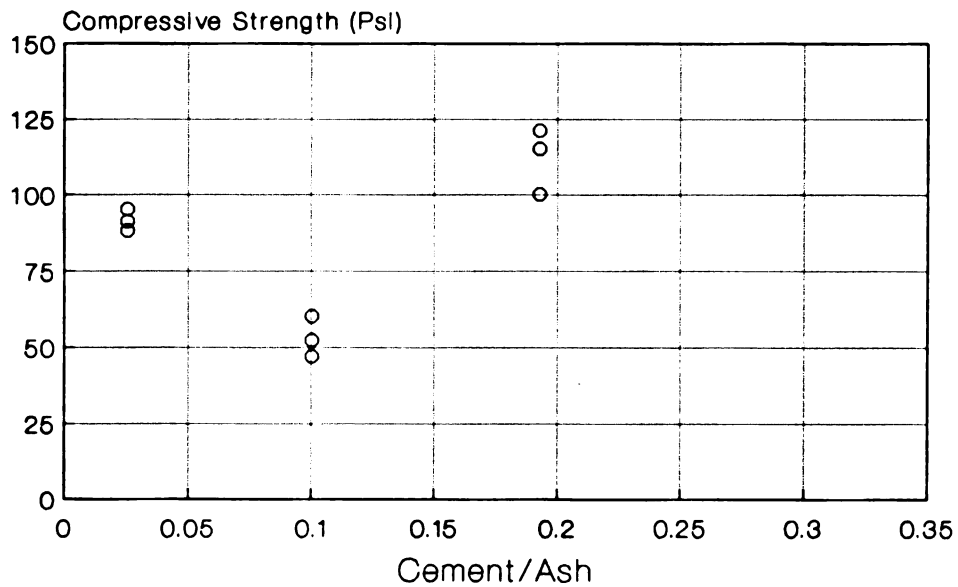


Figure 4.1 (h). Cement/Ash Ratio Vs. Compressive Strength

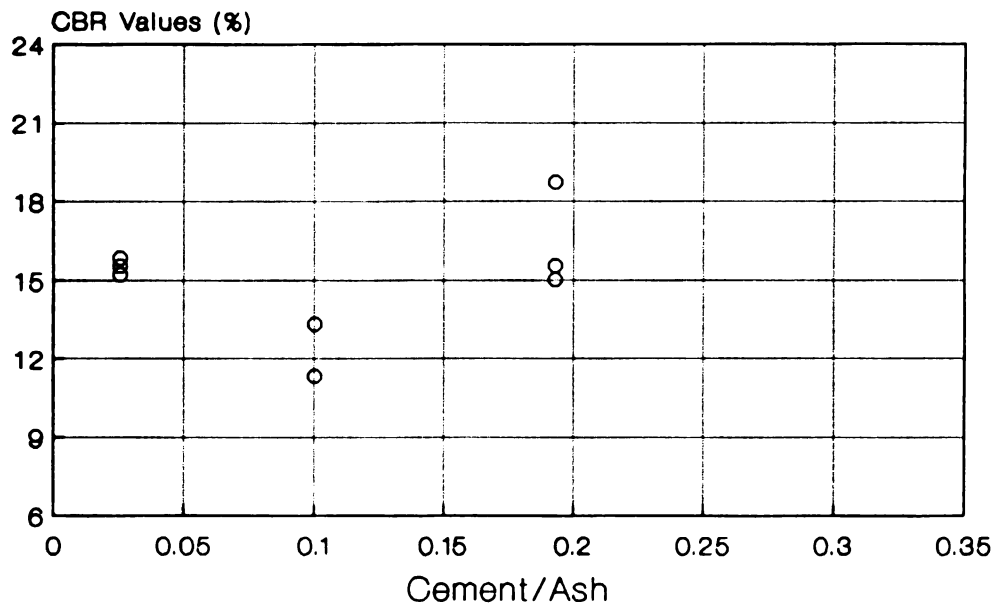


Figure 4.1 (i). Cement/Ash Ratio Vs. California Bearing Ratio (0.1 in)

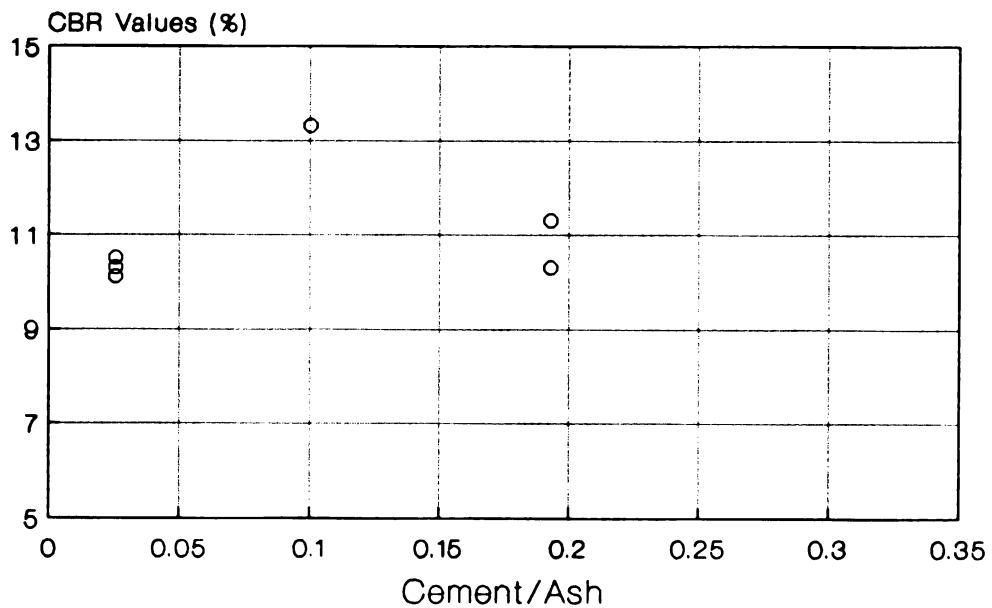


Figure 4.1 (j). Cement/Ash Ratio Vs. California Bearing Ratio (0.2 in)

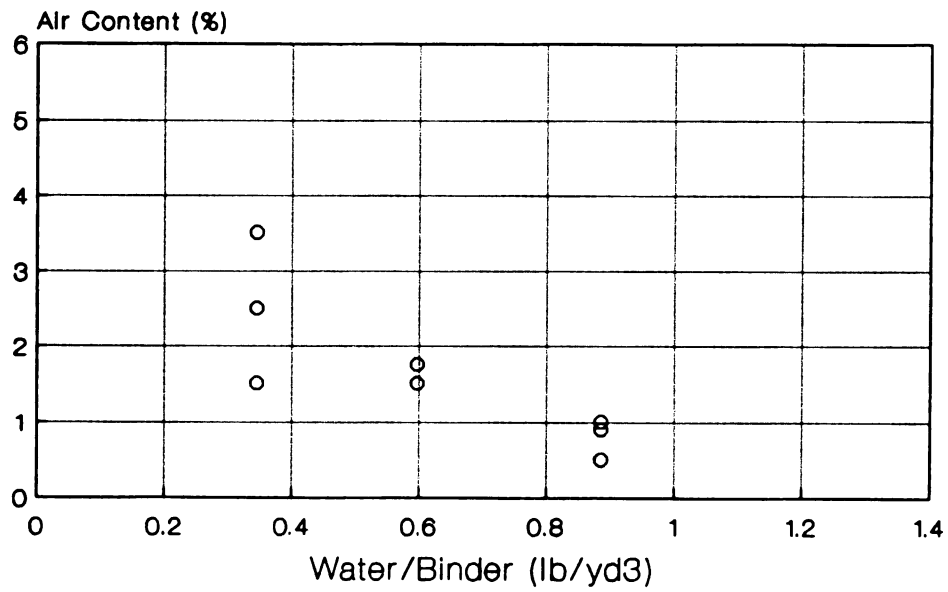


Figure 4.1 (k). Water/Binder Ratio Vs. Air Content

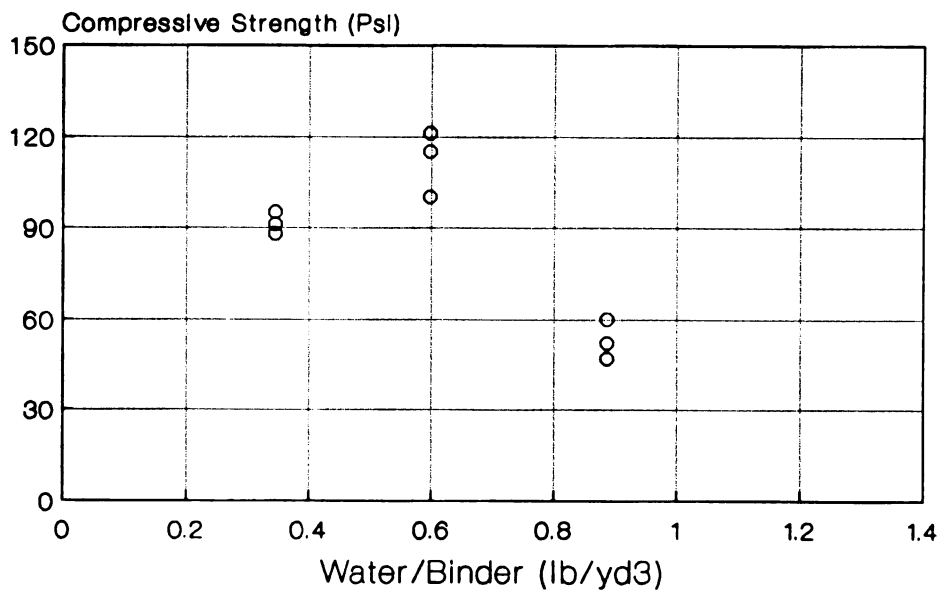


Figure 4.1 (L). Water/Binder Ratio Vs. 28-Day Compressive Strength

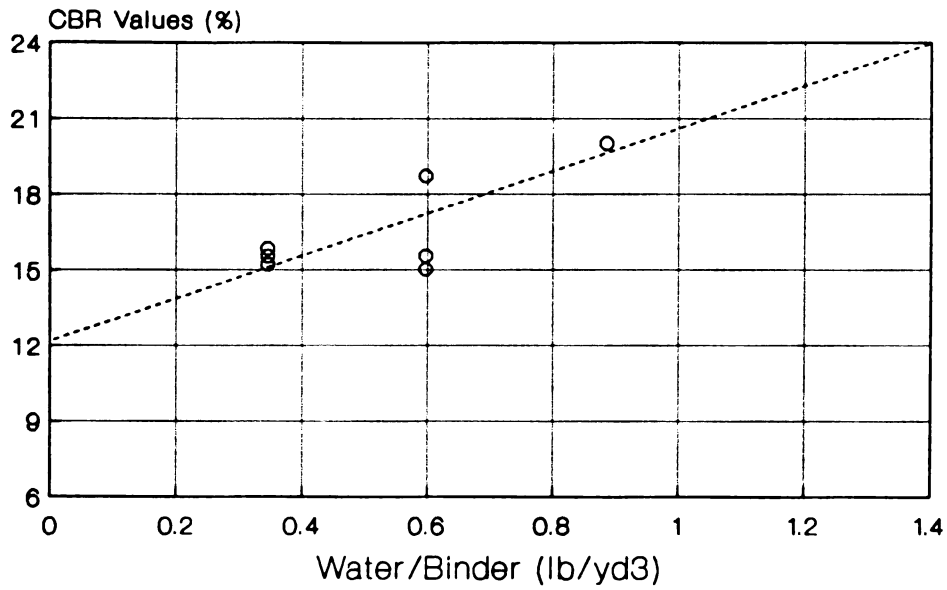


Figure 4.1 (m). Water/Binder Ratio Vs. California Bearing Ratio (0.1 in)

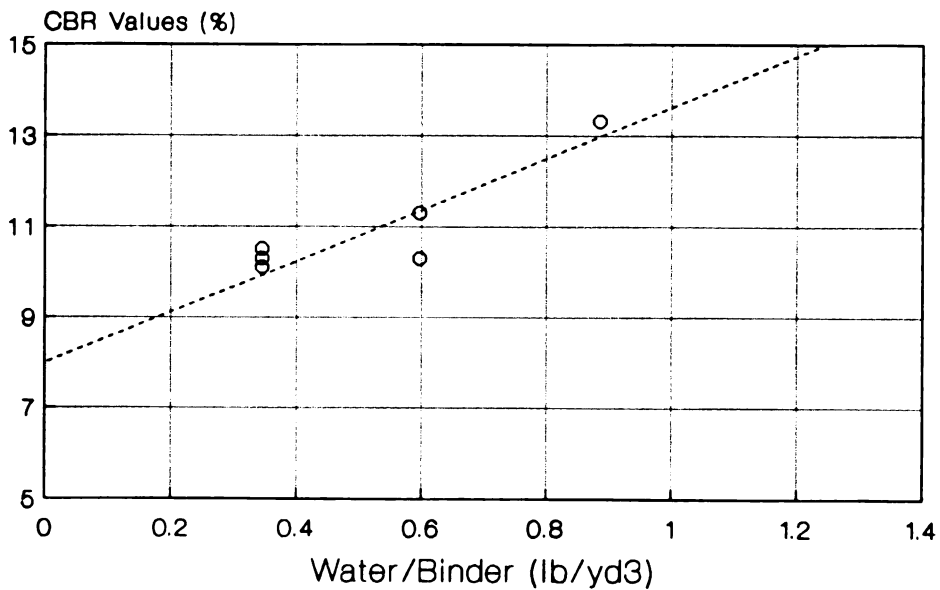


Figure 4.1 (n). Water/Binder Ratio Vs. California Bearing Ratio (0.2 in)

Figure 4.1. Trends in the Effect of Mix Proportions on Material Properties.

CHAPTER 5

IDENTIFICATION OF STATISTICALLY INFLUENTIAL PROPORTIONING VARIABLES

5.1 INTRODUCTION

The results of correlation analyses presented earlier suggest that one can not rely on the mix proportioning techniques used with conventional concrete to design flowable fill mixtures. This phase of research is concerned with identifying the key proportioning variables and establish the statistical effects/interactions of mix variables in determining flowable fill qualities.

5.2 EXPERIMENTAL PROGRAM

A systematic study based on factorial design of experiments was undertaken to identify the influential proportioning variables and the trends in their effects on flowable fill qualities. Four key proportioning variables were considered: ash type (MSU & Lansing Board of Water & Light with 1/5 bottom ash), water-ash ratio (0.5 and 1.25), aggregate-ash ratio (0 and 3), and cement-ash ratio (0.05 and 3). The 1/2 fractional factorial design of experiments for investigating the statistical significance of these variables is presented in Table 5.1. In these Tables, [MSU = Michigan State University and LB = Lansing Board of Water and Light].

Table 5.1. Fractional Factorial Experiments								
Run Order	1	2	3	4	5	6	7	8
Block	1	1	1	1	1	1	1	1
Standard Order	5	1	2	6	8	4	7	3
VARIABLES								
A = Water/Ash	0.5	0.5	1.25	1.25	1.25	1.25	0.5	0.5
B = Aggregate/Ash	0	0	0	0	3	3	3	3
C = Cement/Ash	0.3	0.05	0.05	0.3	0.3	0.05	0.3	.05
D = Ash Type	LB	MSU	LB	MSU	LB	MSU	MSU	LB
RESPONSE								
R1 = f'c (psi)								
R2 = Unit Weight (lb/yd ³)								
R3 = Set Time (Hours)								
R4 = Permeability (cm/sec)								
R5 = Penetration (psi)								
R6 = Slump (in)								
R7 = Flow (%)								
R8 = Air Content (%)								
R9 = CBR (%)								

A to D = Variables

R1 to R9 = Response

The mixtures of Table 5.1 were subjected to the following tests: slump (ASTM C-143), flow (ASTM C-230), air content (ASTM C-231), unit weight (ASTM C-29), set time (ASTM C-403), 28-day compressive strength (ASTM C-109), water permeability, and 28-day penetration resistance (Modified ASTM C-403) and California Bearing Ratio (ASTM D-1883). Test results for the eight mixtures of Table 5.1 are presented in Table 5.2.

Factorial analysis of variance of the test results presented in Table 5.2 indicated that, at 95% level of confidence, ash type (MSU vs. Lansing Board of Water & Light) had no statistically significant effect on any aspect of flowable fill performance. Consequently, further mix compositions were considered to accomplish a full fractional design of experiments with three variables (water/ash, aggregate/ash, and cement/ash ratios) in two blocks; ash type (MSU & Lansing Board) was used as a blocking variable.

The levels of the three variables investigated were: water/ash ratio (0.5 and 1.25), aggregate/ash ratio (0 and 3), cement/ash ratio (0.05 and 0.3). The resulting 2^3 factorial design in two blocks is presented in Table 5.3, and the experimental results are presented in Table 5.4.

Table 5.2. Results of 1/2 Fractional Factorial Experiments

Run Order	1	2	3	4	5	6	7	8
Block	1	1	1	1	1	1	1	1
Standard Order	5	1	2	6	8	4	7	3
VARIABLES								
A = Water/Ash	0.5	0.5	1.25	1.25	1.25	1.25	0.5	0.5
B = Aggregate/Ash	0	0	0	0	3	3	3	3
C = Cement/Ash	0.3	0.05	0.05	0.3	0.3	0.05	0.3	.05
D = Ash Type	LB	MSU	LB	MSU	LB	MSU	MSU	LB
RESPONSE								
R1 = $f'c$ (psi)	91	88	88	100	70	54	60	52
R2 = Unit Weight (lb/yd ³)	134	127	130	97	121	116	100	112
R3 = Set Time (Hours)	5.8	5.2	5.5	5.3	5.0	5.5	5.0	6.1
R4 = Permeability (cm/sec)	2.1 E-7	2.1 E-7	2.1 E-7	2.0 E-7	1.9 E-8	1.9 E-8	1.6 E-8	1.6 E-8
R5 = Penetration (psi)	160	158	155	150	200	200	200	200
R6 = Slump (in)	11	11	10	10	11	11	9	11
R7 = Flow (%)	135	137	131	138	140	137	140	133
R8 = Air Content (%)	1.7	1.8	1.6	2.5	1.2	1.0	0.4	0.9
R9 = California Bearing Ratio % (0.1 in.)	16	16	16	15	20	20	20	20
R10 = California Bearing Ratio % (0.2 in.)	11	10	10	10	13	13	13	13

Table 5.3. 2^3 factorial design in 2 Blocks.

[illegible]

Table 5.3. 2^3 factorial design in 2 Blocks (Cont'd).

[illegible]

Table 5.4. Results of 2³ Factorial Experiments

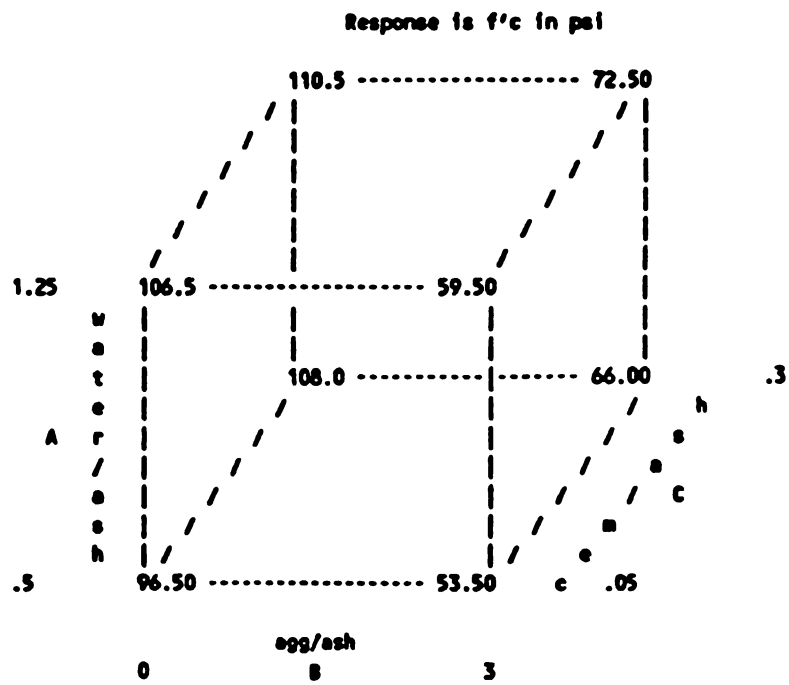
Run Order	1	2	3	4	5	6	7	8
Block	1	1	1	1	1	1	1	1
Standard Order	1	2	6	3	4	5	8	7
VARIABLES								
A = Water/Ash	0.5	1.25	1.25	0.5	1.25	0.5	1.25	0.5
B = Aggregate/Ash	0	0	0	3	3	0	3	3
C = Cement/Ash	.05	0.05	0.3	0.05	0.05	0.3	0.3	0.3
RESPONSE								
R1 = f'c (psi)	88	125	100	55	54	125	75	60
R2 = Unit Weight (lb/yd ³)	127	135	97	92	116	98	100	100
R3 = Set Time (Hours)	5.2	5.6	5.3	5.6	5.5	6.0	5.0	5.0
R4 = Permeability (cm/sec)	2.1 E-7	2.0 E-7	2.0 E-7	1.7 E-8	1.9 E-8	2.0 E-7	2.0 E-8	1.6 E-8
R5 = Penetration (psi)	158	152	150	200	200	155	200	200
R6 = Slump (in)	11	8.9	10	8.5	10.5	10	9	9
R7 = Flow (%)	137	134	138	135	137	138	140	140
R8 = Air Content (%)	1.8	1.6	2.5	0.6	1.0	1.7	0.7	0.4
R9 = California Bearing Ratio % (0.1 in.)	16	15	15	20	20	16	20	20
R10 = California Bearing Ratio % (0.2 in.)	11	10	10	13	13	10	13	13

Table 5.4. Results of 2³ Factorial Experiments (cont'd).

Run Order	9	10	11	12	13	14	15	16
Block	2	2	2	2	2	2	2	2
Standard Order	9	15	12	11	14	13	16	10
VARIABLES								
A = Water/Ash	0.5	0.5	1.25	0.5	1.25	0.5	1.25	1.25
B = Aggregate/Ash	0	0	3	3	0	0	3	0
C = Cement/Ash	.05	0.3	0.05	0.05	0.3	0.3	0.3	.05
RESPONSE								
R1 = f'c (psi)	105	72	65	52	121	91	70	88
R2 = Unit Weight (lb/yd ³)	99	115	100	112	130	134	121	130
R3 = Set Time (Hours)	6.2	6.1	5.4	6.1	5.7	5.8	5.0	5.5
R4 = Permeability (cm/sec)	2.1 E-7	1.6 E-8	2.0 E-8	1.6 E-8	1.9 E-7	2.1 E-7	1.9 E-8	2.1 E-7
R5 = Penetration (psi)	154	200	200	200	150	160	200	155
R6 = Slump (in)	11	11	10.5	11	8.5	11	10.5	10
R7 = Flow (%)	140	133	144	133	130	135	140	131
R8 = Air Content (%)	1.7	0.8	0.6	0.9	1.56	1.7	1.2	1.6
R9 = California Bearing Ratio % (0.1 in.)	15	20	20	20	15	16	20	16
R10 = California Bearing Ratio % (0.2 in.)	10	13	13	13	10	11	13	10

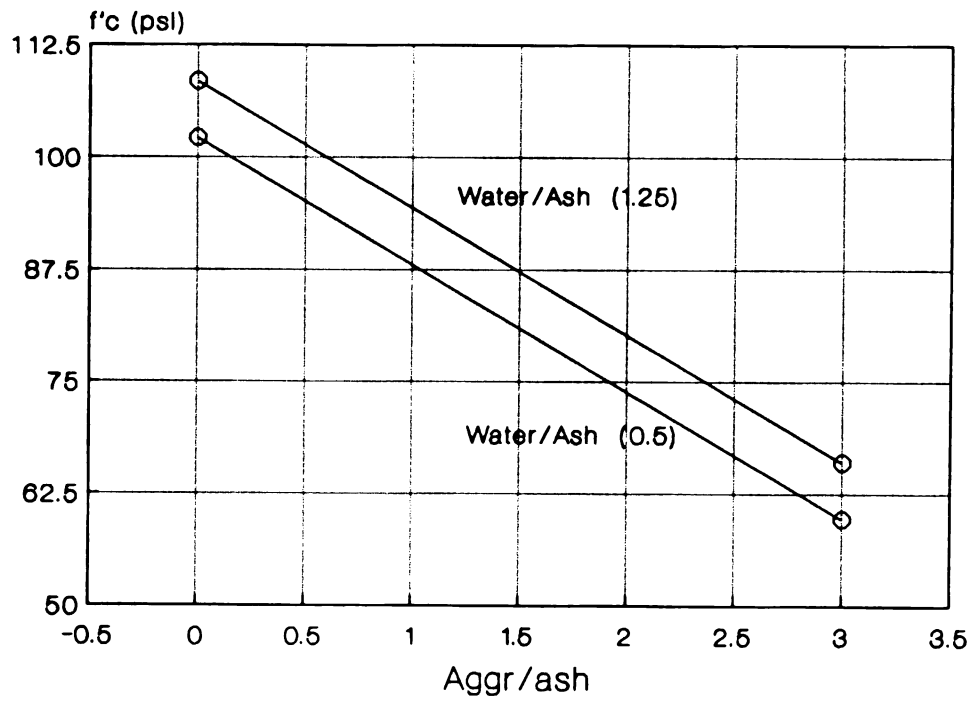
Factorial analysis of variance of the test data presented in Table 5.4 led to the following conclusions:

(1) Compressive Strength: The compressive strength of the flowable fill was influenced only by the aggregate/ash ratio, which represents total binder content (i.e., cement and fly ash), at 99.9% level of confidence; cement/ash ratio and water/ash ratio did not have any statistically significant effect on compressive strength (even if attempt is made to validate such effects at 80% level of confidence). The cube plot of Figure 5.1 a and the trends presented in Figures 5.1 b and 5.1 c confirm the significant effects of aggregate/ash ratio on the compressive strength of flowable fill. Increasing the aggregate/ash ratio from 0 to 3 (reflecting a reduction in total binder content) leads to approximately 40% reduction in compressive strength at constant water-to-ash and cement-to-ash ratios. In interpreting the cube plots one should note that the cube is plotted in a 3-D space with each axis representing one of the three variables considered. Each corner of the cube thus represents specific levels of the three variables, and moving along any of the three lines ending at a corner would change one of the variables with the other two kept constant.



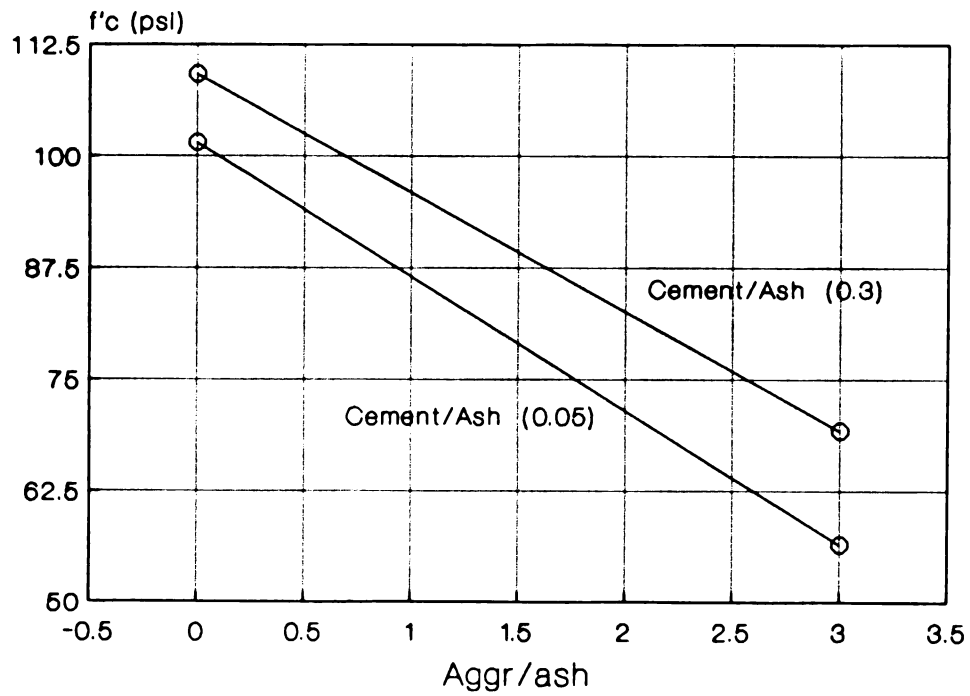
(a) Three-Factor Interaction Cube Plot

Figure 5.1. Trends in variable Effects on compressive strength.



(b) Effect of Aggregate/Ash Ratio and interaction with Water/Ash Ratio

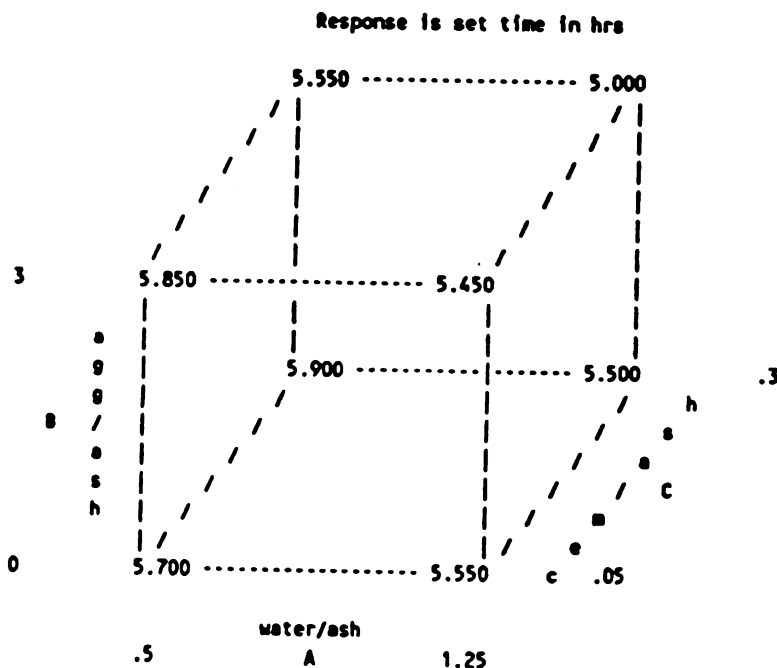
Figure 5.1. Trends in variable Effects on compressive strength.



(c) Effect of Aggregate/Ash Ratio and Interaction with Cement/Ash Ratio.

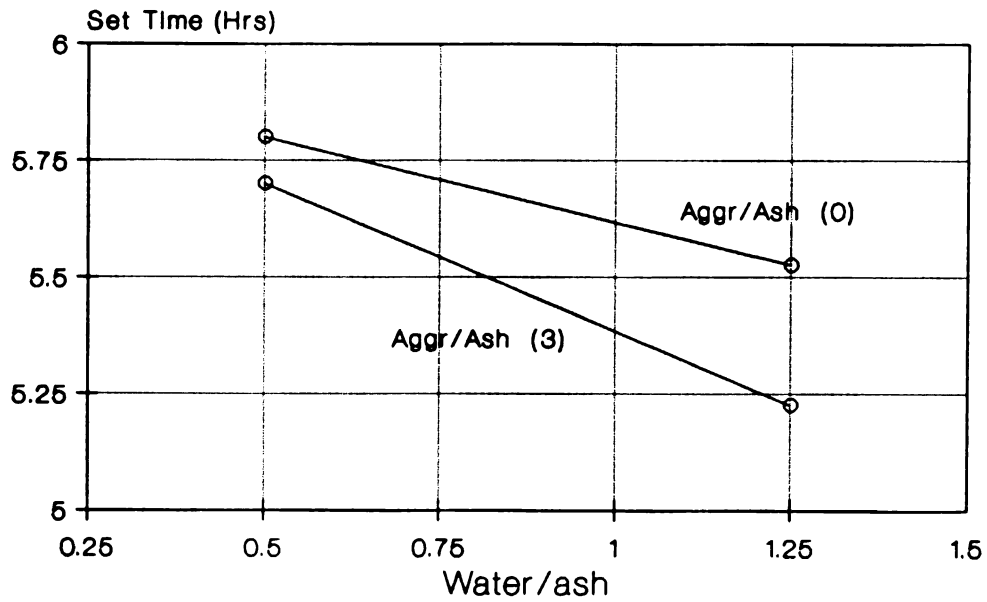
Figure 5.1. Trends in variable Effects on compressive strength.

(2) Set Time: The only factor with statistically significance effect (at 95% levels of confidence) on the set time of flowable fill is water/ash ratio. There was also no interaction between different factors. The increase in water/ash ratio from 0.5 to 1.25 caused a slight decrease of about 5% in set time. Figure 5.2 a presents the cube plot of the effects of different factors on set time, and figures 5.2 b and 5.2 c present the effects of water/ash ratio at different aggregate/ash and cement/ash ratios, respectively.



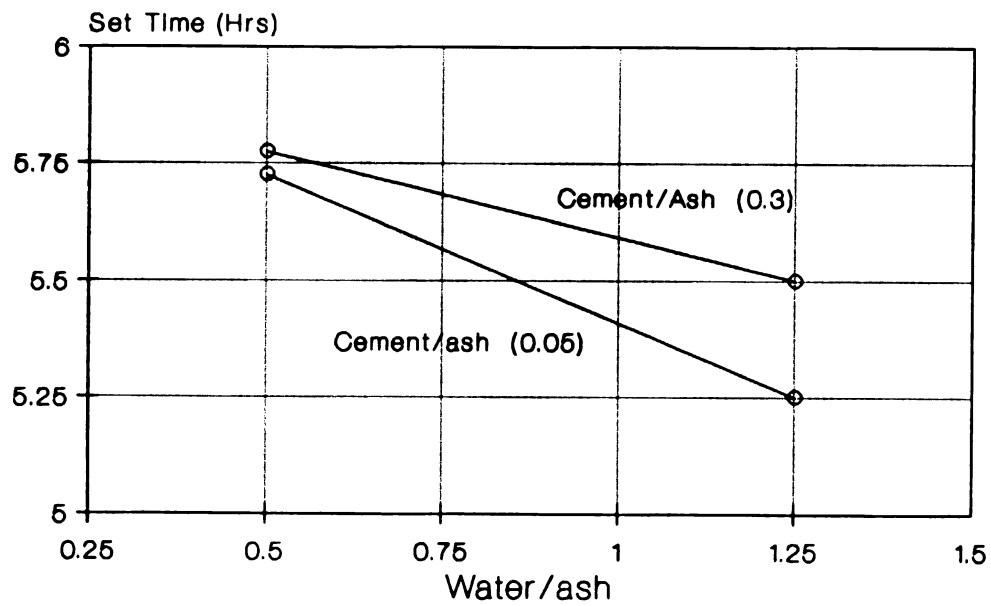
(a) Three-Factor Interaction Cube Plot

Figure 5.2. Trends in Variable Effects On Set Time



(b) Effect of Water/Ash Ratio and Interaction with Aggregate/Ash Ratio

Figure 5.2. Trends in Variable Effects On Set Time



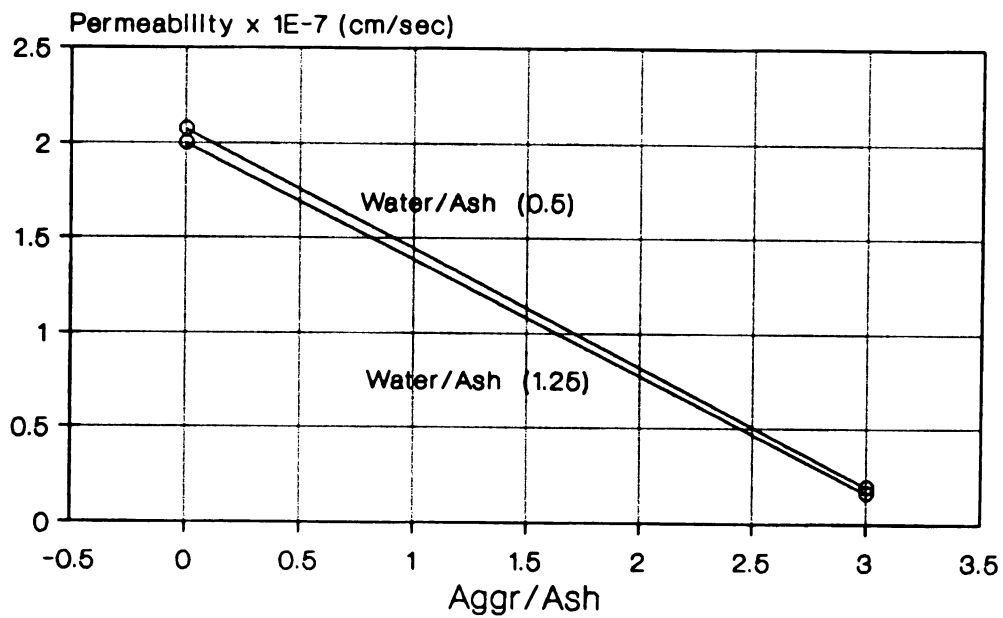
(c) Effect of Water/Ash Ratio and Interaction with Cement/Ash Ratio

Figure 5.2. Trends in Variable Effects On Set Time

(3) Water Permeability: Aggregate/ash ratio is the only factor with statistically significant effect on flowable fill permeability (at 99.9% level of confidence). An increase in aggregate/ash ratio from 0 to 3 reduced water permeability by an order of magnitude. Figure 5.3 a shows the cube plot of the effects of different factors on water permeability, and Figures 5.3 b and 5.3 c show the effects of aggregate/ash ratio of different water/ash and cement/ash ratios.

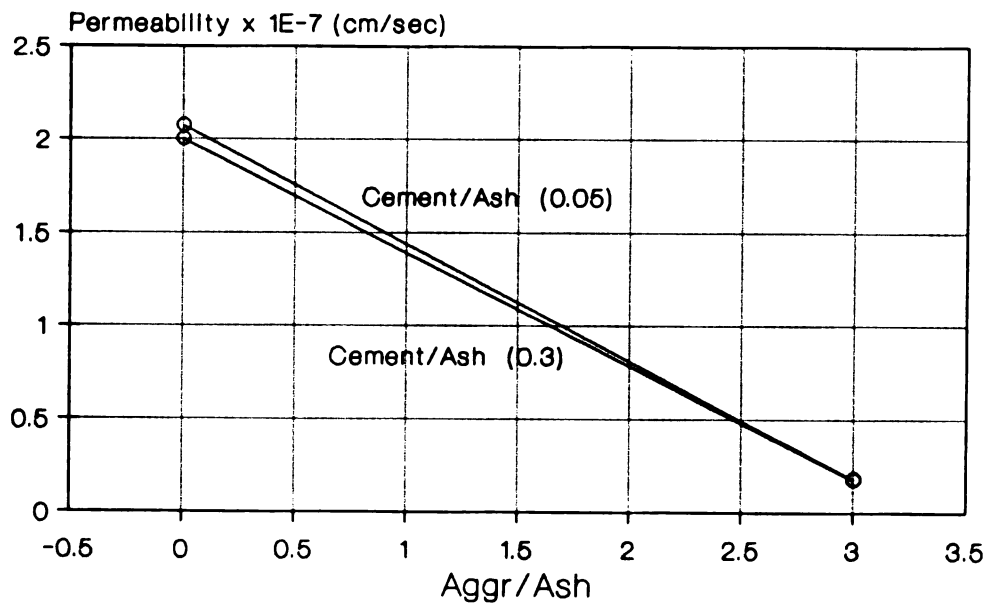
It is interesting to note that increased aggregate/ash ratio leads to reduced compressive strength but increased impermeability of flowable fill. This further highlights the differences in structure of flowable fill and conventional concrete. Mechanisms leading to increased strength of flowable fill are not necessarily those causing reduction in permeability of the material.

Figure 5.3. Trends in Variable Effects on Water permeability



(b) Effects of Aggregate Ash/Ratio and Interaction with Water/Ash Ratio

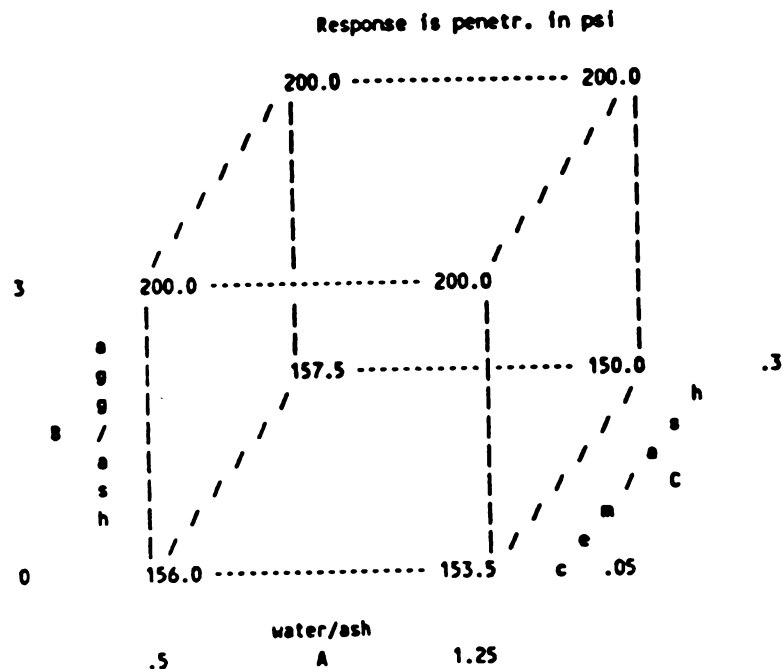
Figure 5.3. Trends in Variable Effects on Water permeability



(c) Effects of Aggregate/Ash Ratio and Interaction with Cement/Ash Ratio

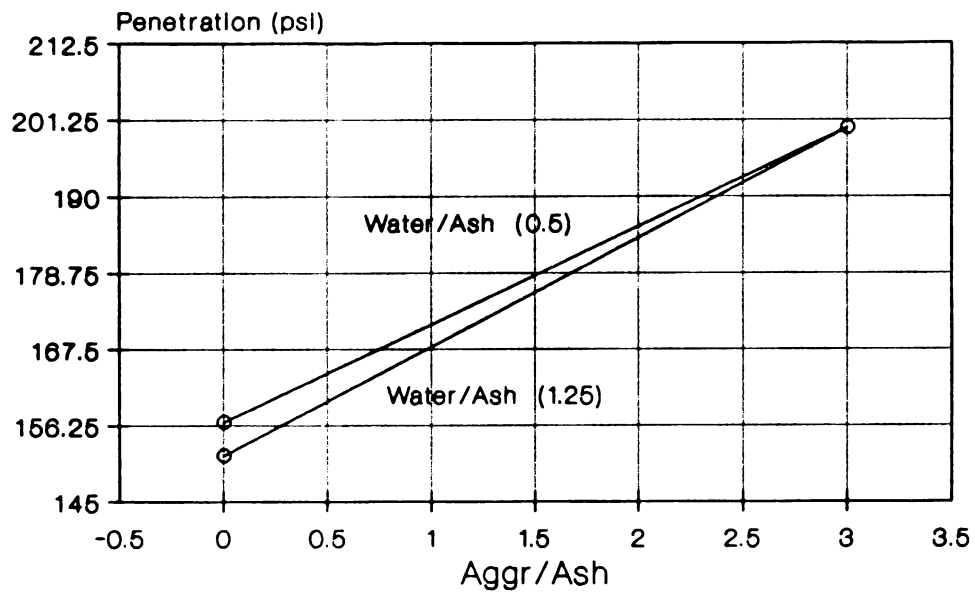
Figure 5.3. Trends in Variable Effects on Water permeability

(4) Penetration Resistance: At 99% level of confidence, only aggregate/ash ratio had statistically significant effect on 28-day penetration resistance. As shown in the cube plot of Figure 5.4 a and interaction/effects plot of Figures 5.4 b and 5.4 c, increasing aggregate/ash ratio from 0 to 3 leads to an approximately 30% increase in penetration resistance. This conclusion further confirms that factors such as cement/ash ratio or water/ash ratio, which could be critical if the structure of flowable fill are similar to that of concrete, play a secondary role in flowable fill when compared with aggregate-ash ratio (or total binder content). It is also interesting to note that increased aggregate content (i.e., reduced binder content) led to reduced compressive strength but increased penetration resistance.



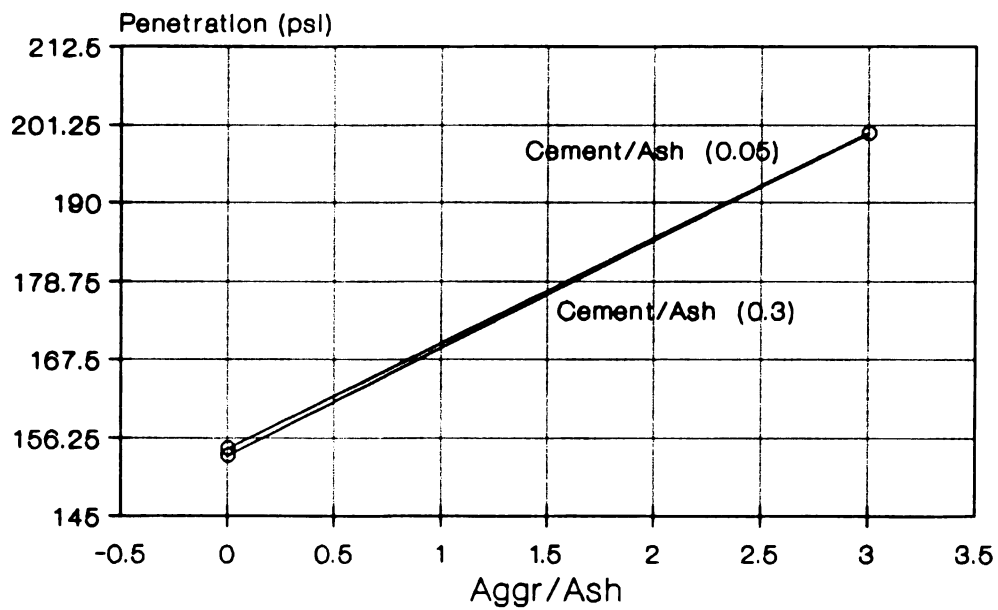
(a) Three-Factor Interaction Cube Plot

Figure 5.4. Trends and Variable Effects on 28-Day Penetration Resistance.



(b) Effect of Aggregate/Ash Ratio and Interaction with Water/Ash Ratio.

Figure 5.4. Trends and Variable Effects on 28-Day Penetration Resistance.



(c) Effect of Aggregate/Ash Ratio and Interaction with Cement/Ash Ratio.

Figure 5.4. Trends and Variable Effects on 28-Day Penetration Resistance.

(5) Slump: None of the variables considered in this investigation (water/ash ratio, aggregate/ash ratio, and cement/ash ratio) had statistically significant effects on slump within the variable ranges considered, (see cube plot of Figure 5.5).

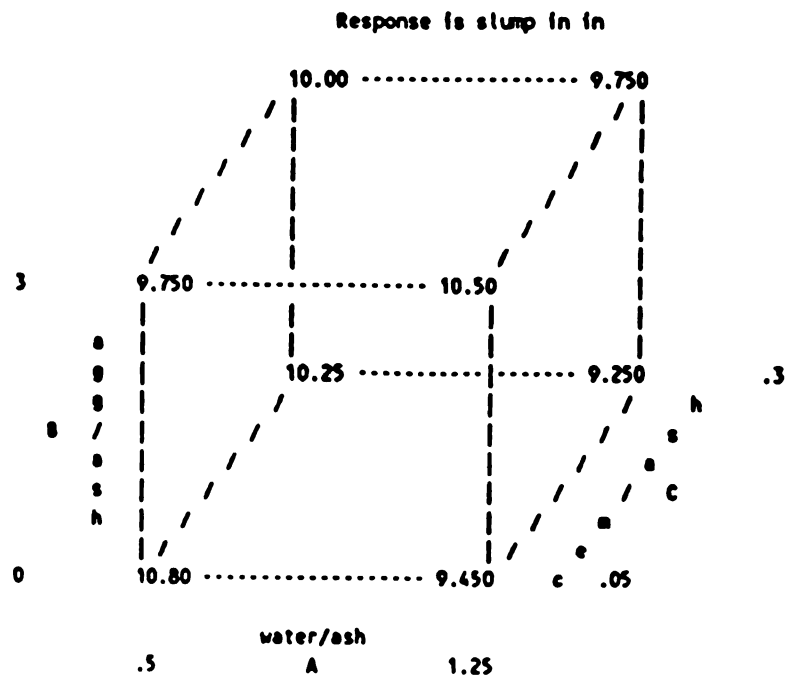


Figure 5.5. Three-Factor Interaction Cube Plot Representing Variable Effects On Slump.

(6) Flow: In the case of flow also, no statistically significant effects of any of the variables was observed (see cube plot of Figure 5.6).

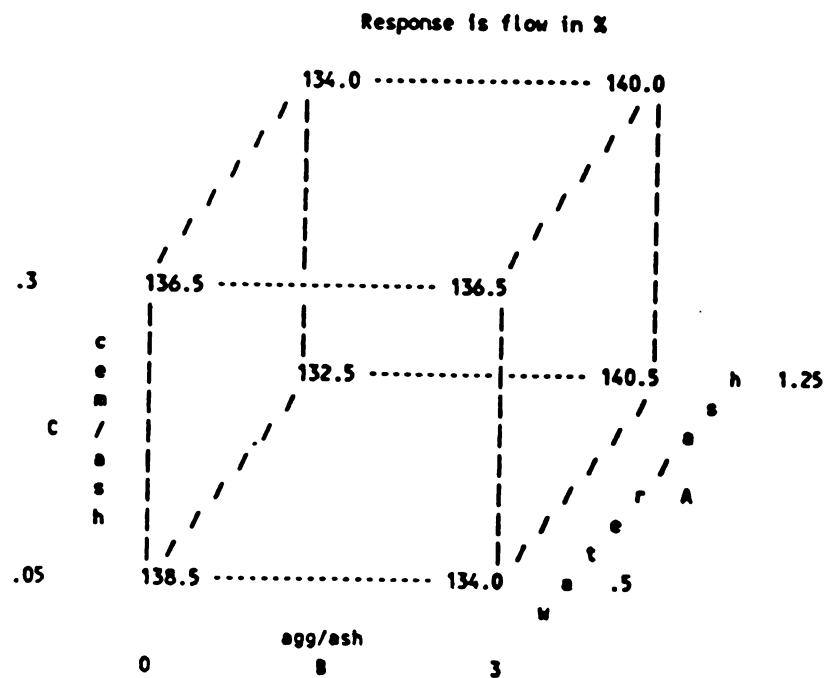
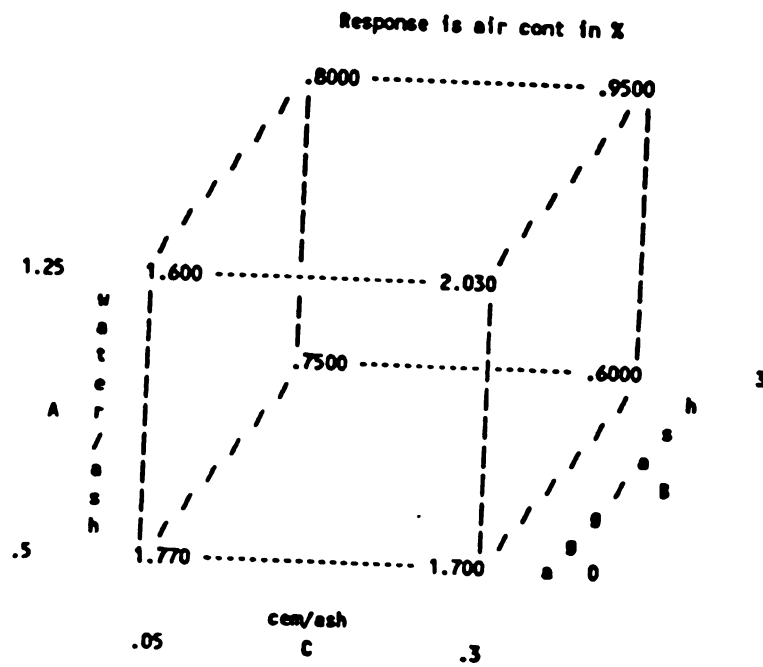


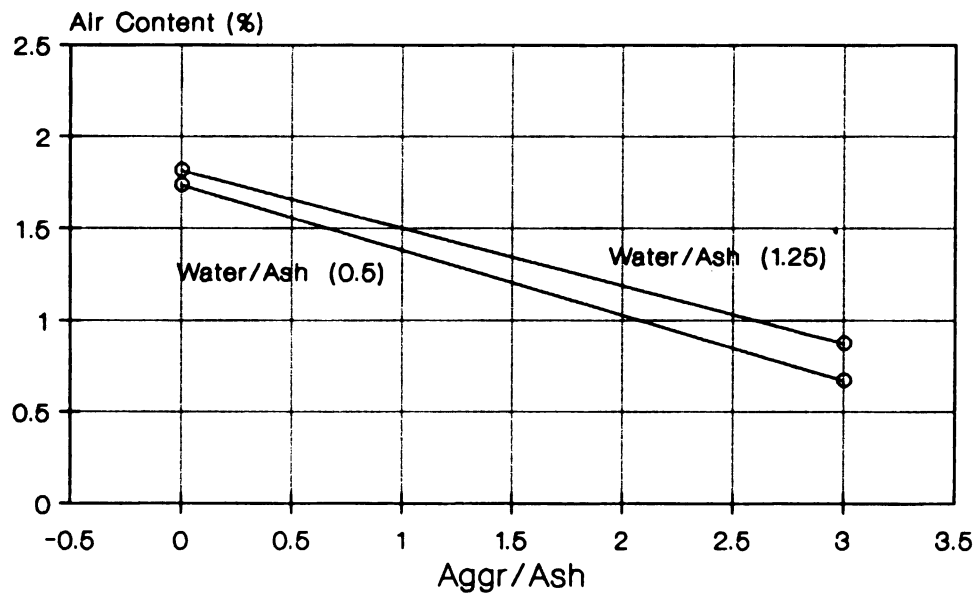
Figure 5.6. Three-Factor Interaction Cube Plot Representing Variable Effects on Flow.

(7) Air Content: Aggregate/ash ratio was the only factor with statistically significant-effect (at 99% level of confidence) on the air content of flowable fill. No interaction between the effects of different factors was observed. Aggregate effects on air content could be simply illustrated by the fact that increased aggregate content reduces the binder content when air resides. As shown in the cube plot of Figure 5.7, increasing the aggregate/ash ratio from 0 to 3 reduced air content by about half. The interaction/effect plots of Figures 5.7 b and 5.7 c further show the trends in aggregate-ash ratio effects on air content.



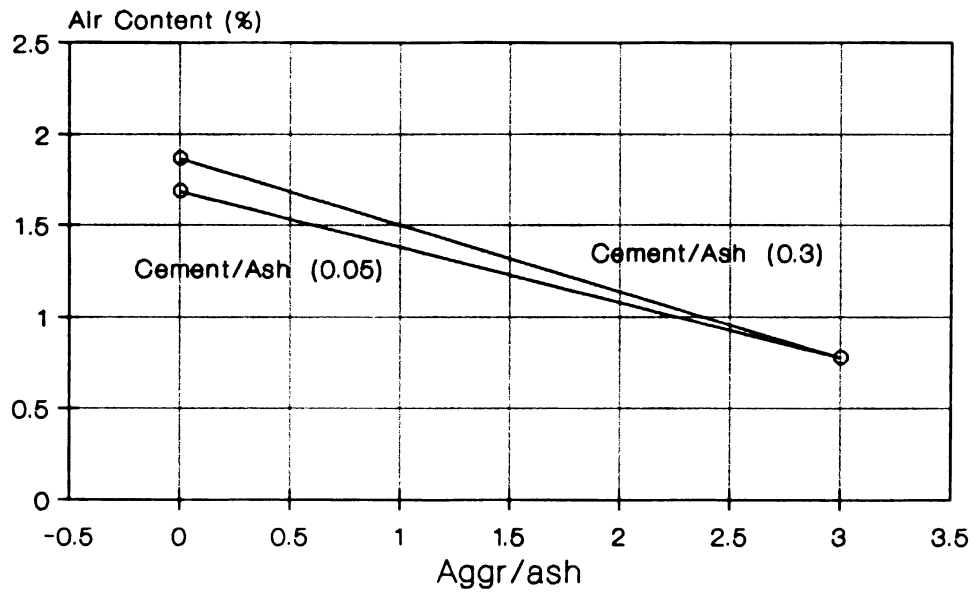
(a) Three-Factor Interaction Cube Plot Representing Variable Effects on Air Content

Figure 5.7. Trends in Variable Effects on Air Content.



(b) Effect of Aggregate/Ash Ratio and Interaction with Water/Ash Ratio

Figure 5.7. Trends in Variable Effects on Air Content.



(c) Effect of Aggregate/Ash Ratio and Interaction with Cement/Ash Ratio.

Figure 5.7. Trends in Variable Effects on Air Content.

(8) Unit Weight: None of the factors considered in this investigation (cement-ash ratio, aggregate-ash ratio) had any statistical significant effect on fresh mix unit weight (see Cube plot of figure 5.8).

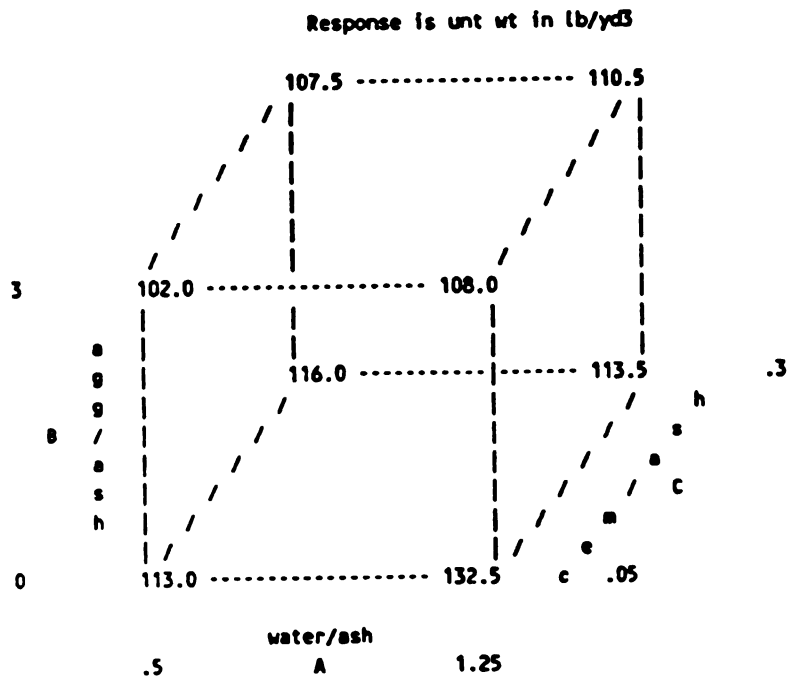
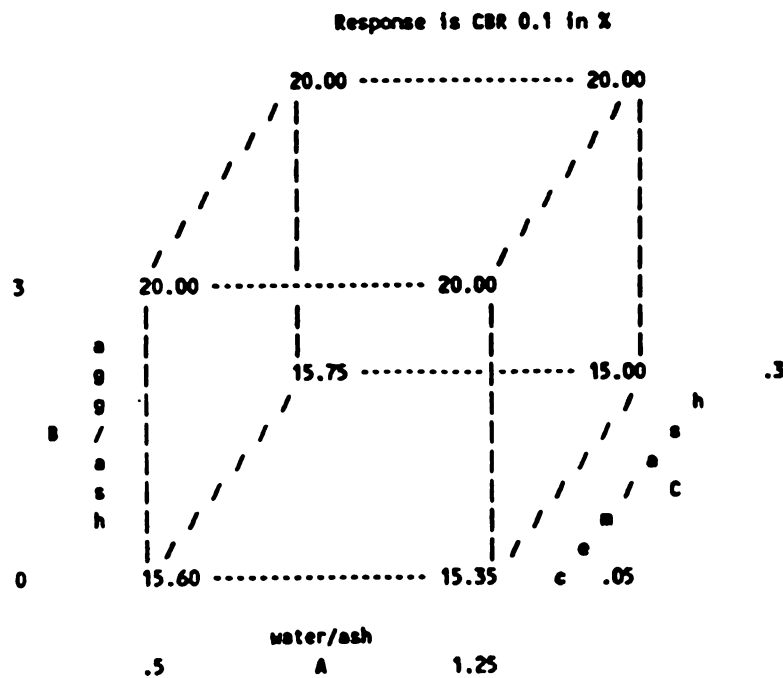


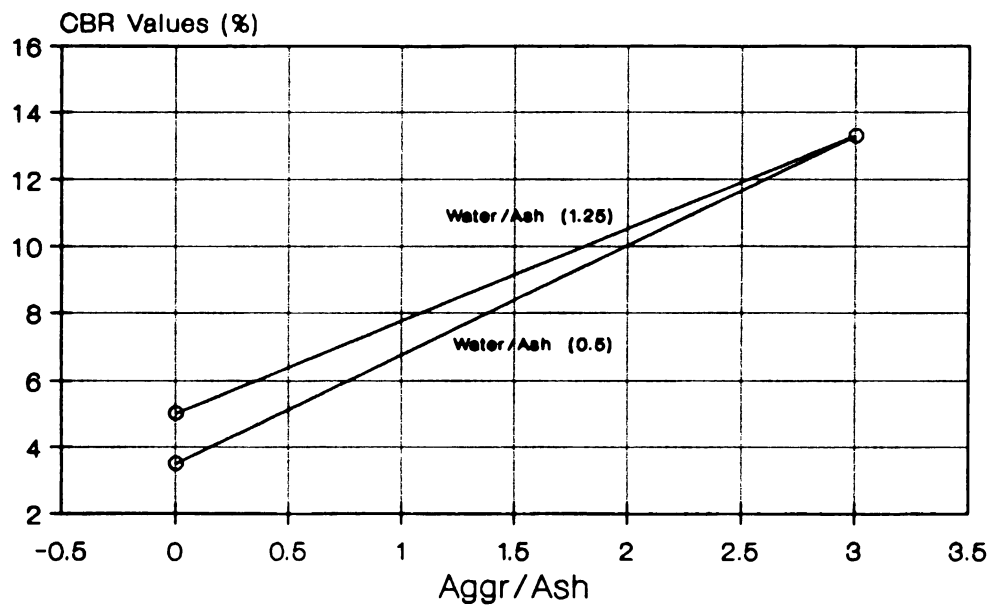
Figure 5.8. Three-Factor Interaction Cube Plot Representing Variable Effects on Unit Weight.

(9) California Bearing Ratio (CBR): Aggregate/ash ratio is the only factor with statistically significant effect, at 99% level of confidence, on the California Bearing Ratio for flowable fill (see Figures 5.9 and 5.10). Increasing the aggregate-ash ratio from 0 to 3 led to approximately 30% increase in California Bearing Ratio at all water-ash and cement-ash ratios.



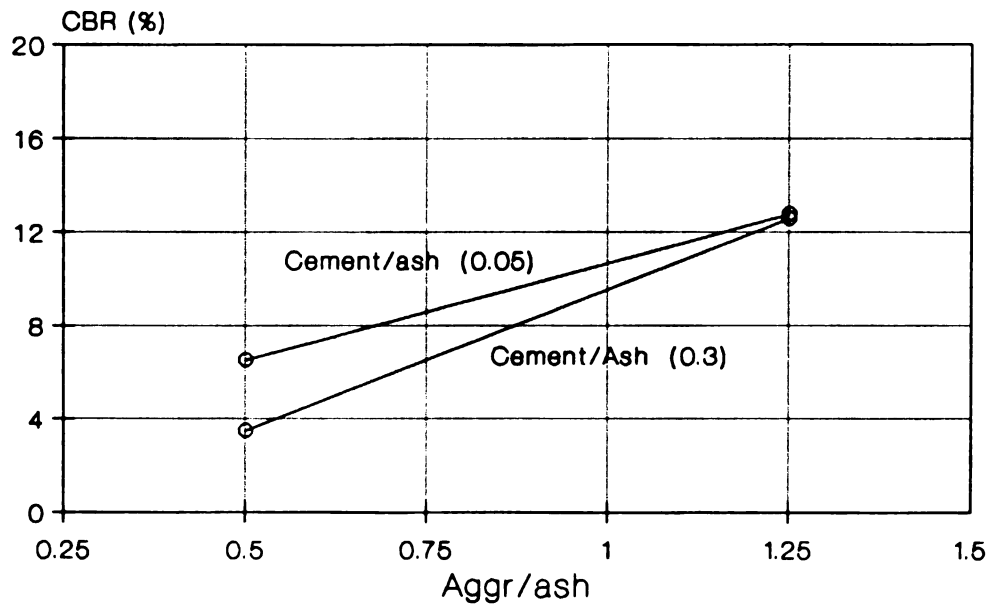
(a) Three-Factor Interaction Cube Plot (0.1 in., Penetration)

Figure 5.9. Trends in Variable Effects on California Bearing Ratio (CBR, 0.1 in.)



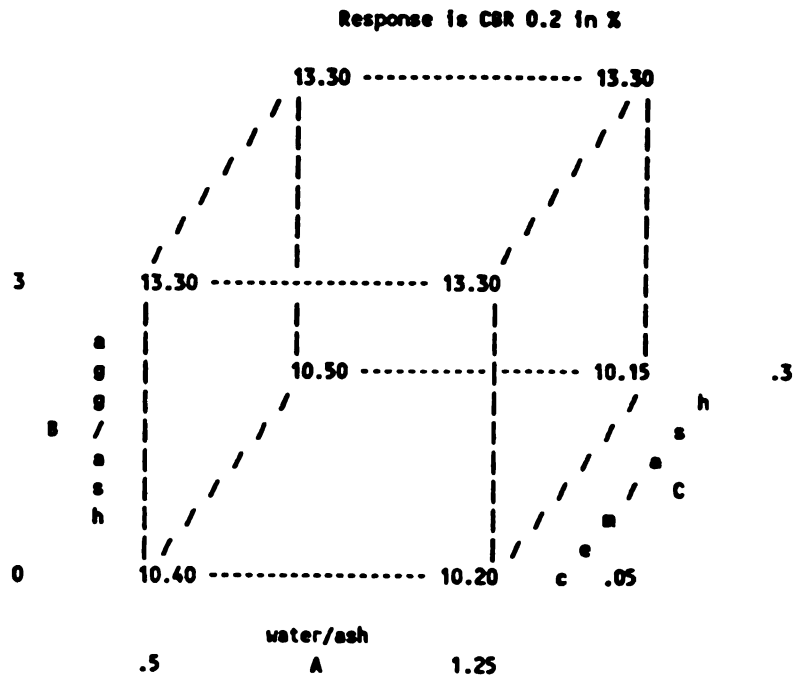
(b) Effect of Aggregate/Ash Ratio and interaction with Water/Ash Ratio (0.1 in.)

Figure 5.9. Trends in Variable Effects on California Bearing Ratio (CBR, 0.1 in.)



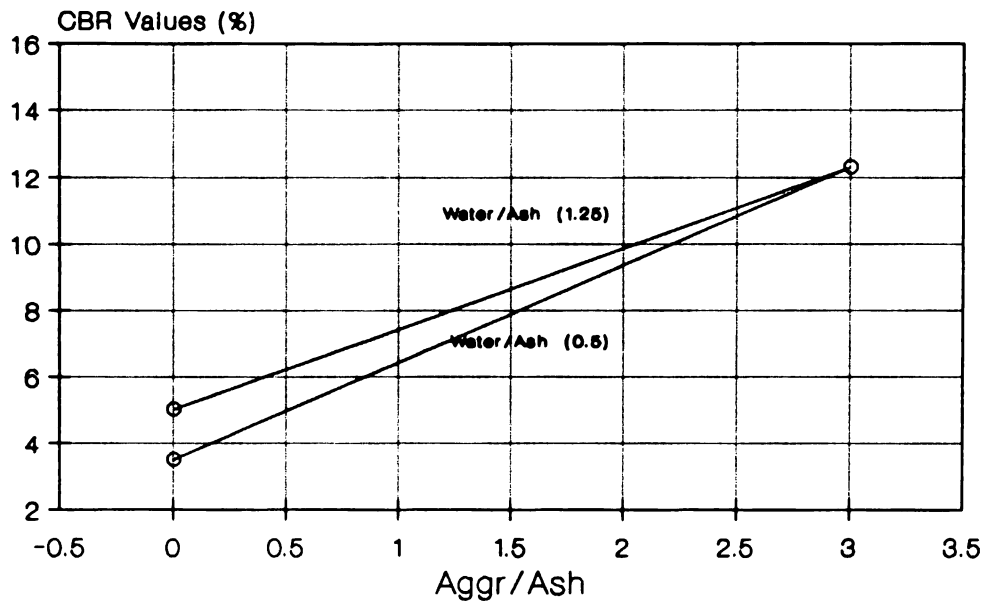
(c) Effects of Aggregate/Ash Ratio and interaction with Cement/Ash Ratio (0.1 in.)

Figure 5.9. Trends in Variable Effects on California Bearing Ratio (CBR, 0.1 in.)



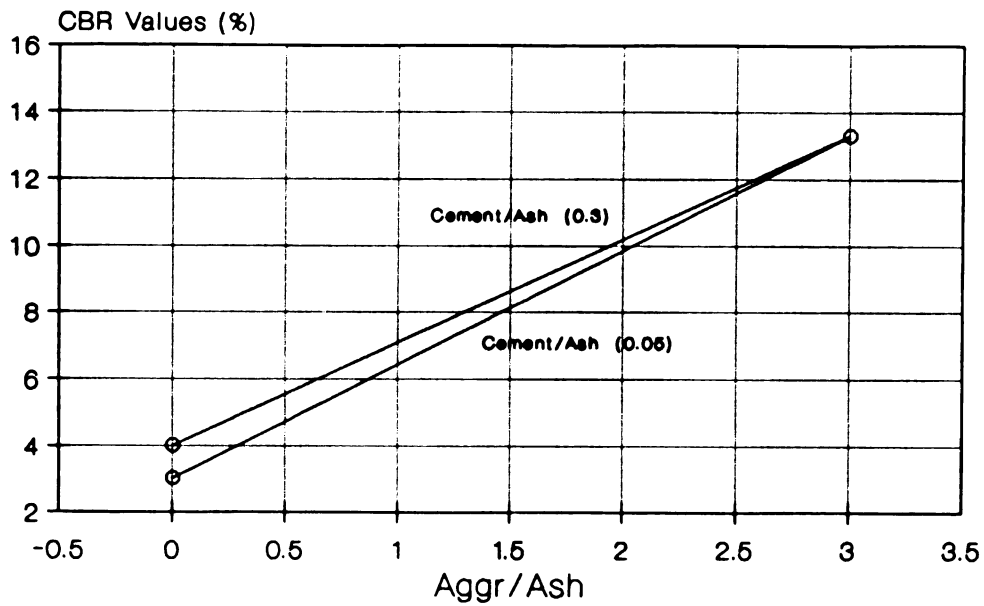
(a) Three-Factor Interaction Cube Plot (0.2 in., Penetration)

Figure 5.10. Trends in Variable Effects on California Bearing Ratio (CBR, 0.2 in.)



(b) Effect of Aggregate/Ash Ratio and interaction with Water/Ash Ratio (0.2 in.)

Figure 5.10. Trends in Variable Effects on California Bearing Ratio (CBR, 0.2 in.)



(c) Effect of Aggregate/Ash Ratio and interaction with Cement/Ash Ratio (0.2 in.)

Figure 5.10. Trends in Variable Effects on California Bearing Ratio (CBR, 0.2 in.)

CHAPTER 6

CONCLUSIONS

A comprehensive investigation was undertaken in order to derive statistically reliable conclusions regarding material properties of flowable fill as influenced by various mix proportions. The correlations between various aspects of the performance characteristics of flowable fill were also investigated. The results indicated that:

1. The development of the structure of flowable fill is different from that of concrete; physical factors such as the presence of sand greatly influence structural development in flowable fill. The three different coal ashes with different qualities and various combinations of fly ash and bottom ash produced flowable fills with comparable performance characteristics.
2. It is difficult to develop a brief set of experiments to make a comprehensive evaluation of flowable fill qualities. For example, as binder content increases (and aggregate content decreases), flowable fill tends to gain additional strength but lose penetration resistance and impermeability qualities. Hence, strict reliance on strength as the sole representative of flowable fill quality may produce misleading conclusions in relation to penetration resistance and impermeability (and vice versa).

3. The California Bearing Ratio correlates well with 28-day penetration resistance, compressive strength and water permeability of flowable fill.
4. Early age compressive strength of flowable fill may not fully represent the strength characteristics of the material at later ages.
5. Slump and flow of flowable fill are not strongly correlated.
6. The effects of water/ash ratio and cement/ash ratio (critical variables for concrete performance) on flowable fill quality tend to be overshadowed by the corresponding effects of aggregate content (or total binder content).
7. Reduced sand content, which is accompanied with increased binder content, leads to increased compressive strength but reduced penetration resistance and California Bearing Ratio. When a combination of mixes with and without sand are considered, binder content is the only factor with statistically significant effects on compressive strength; cement/ash ratio and water/ash ratio were not as influential in this case.

8. Set time of flowable fill is not much affected by mix proportioning variables such as the water/ash ratio, cement/ash ratio or aggregate/ash ratio.
9. Aggregate/ash ratio is the only mix proportioning variables with statistically significant effects on flowable fill permeability (when mixes with and without sand are considered together); increased aggregate/ash ratio tends to reduce water permeability (while damaging compressive strength).
10. The penetration resistance and California Bearing Ratio of flowable fill tend to be improved with increased aggregate content (while compressive strength tends to be damaged when aggregates are present).

It is our overall conclusion that, given specific raw materials, the relevant mix proportions and specific job requirements, one may not be able to use mix proportioning methods or correlations between different qualities of conventional mortar and concrete to apply the existing information to a specific flowable fill job condition. The best approach is to subject the flowable fill developed with relevant materials and mix proportions to specific tests representing the job requirements in order to make a judgement on the merits of the material.

REFERENCES

1. Nailk, T., Ramme, B., and Kolbeck, H., " Filling Abandoned Underground Facilities with CLSm Fly Ash Slurry", Concrete International, July 1990, pp. 19-25.
2. Larsen, R.L., "Sound use of CLSMs in the Environment," Concrete International, July 1990, pp. 26-29
3. Larsen, R.L., "Use of Controlled Low-Strength Materials in Iowa," Concrete International, Vol. 10, No. 7, July 1988, pp.22-23.
4. Buss, W.E., "Iowa Flowable Mortar Saves Bridges and Culverts" Transportation Research Record 1234, pp. 30-34.
5. Fox, T.A., "Use of Course Aggregate in Controlled Low-Strength Materials", Transportation Research Record 1234, pp.35-38
6. Krell, W.C., "Flowable Fly Ash" Transportation Research Record 1234, pp. 8-12
7. Swaffer, K.M., and Price, H.R., "Tunnel Saved by Fly Ash," Civil Engineering, Sept. 1987, pp. 68-70.
8. Funston, J.J., Krell, W.C., and Zimmer, F.V., "Flowable Fly Ash: A New Cement Stabilized Backfill" Civil Engineering/ASCE, pp. 48-51
9. Narang, R.P., "Innovations Cut Cost of a Power Plant's Water System", Civil Engineering/ASCE, July 1983, pp.46-49
10. American Electric Power, "Using Coal Ashes to Pave the Way," Concrete International, pp. 90.
11. Fritz-Flow # 1, "Anti-Gelling Dispersant, Class C Fly Ash Stabilizing Admixture", Product Bulletin # 12, 1990, pp. 1-2
12. Ontario Research Foundation, "Coal Ash Technology" pp. 1-10
13. Soroushian, P. and Okwuegbu A., "Classification of MSU Power Plant Fly Ash for Application to Concrete," Report MSU-Engr-89-012, June 1989.

14. Meininger, R.C., "Use of Fly Ash in Cement and Concrete-Research of Two Meetings", Concrete International, July 1982, pp. 52-58.
15. Olitt, A., "Courting the Client: Essential Program for Effective Marketing", Civil Engineering/ASCE, June 1976, pp. 65-66.
16. Fritz-Flow # 1, "Anti-Gelling Dispersant, Class C Fly Ash Stabilizing Admixture", Product Bulletin # 112M, pp. 1-2.
17. Hermiz, S.M., and Spence, S., "Belle River Power Plant Fly Ash (Type "C") in Concrete for Structural Application", Design Engineering, Power Generation Organization, Dec. 9, 1988, pp. 1-8
18. Sridharan, A., Rao, A., and Sivapullaiah, P., "Swelling Pressure of Clays", Geotechnical Testing Journal, pp. 24-33.
19. Naik, T.R., Ramme, B.W., and Kolbeck, H.J., "Filling Abandoned Underground Facilities with CLSM Fly Ash Slurry", Concrete International, July 1990, pp. 19-25.
20. EPRI Reports CS-6100, "Laboratory Testing of Fly Ash Slurry", Project 2422-2, Dec. 1988.
21. Shah, S.P., and Young J.F., "A Renaissance in Cement-Based Materials", pp. 301-322.
22. Sawan J.S., and Qasrawi, H.Y., "Early Age Properties of Pozzolanic Mortars Under Hot Weather", pp. 165-172.
23. Woolgar, G., and Oates, D.B., "Fly Ash and the Ready-Mixed Concrete Producer", Concrete International, Nov. 1979, pp. 34-40
24. Lane, D.S., "Testing Fly Ash in Mortars for Air-Entrainment Characteristics", Cement, Concrete, and Aggregates, pp. 25-31.
25. Torrens, I.M., "Cleaning up Coal" Civil Engineering, Oct. 1990, pp. 63-65
26. Joshi, R.C., and Nagaraj, T.S., "Generalization of Flowing Behavior of cement Fly Ash Paste and Mortars" pp. 128-135.

27. Smith, A., "Controlled Low-Strength Material", *Concrete Construction*, May 1991, pp. 391-398.
28. Board of Water and Light, "Flowable Back-Filled Demonstration (Eliminate Compacting)", April 20, 1989, pp. 1-19.
29. Environmental Regulations and Technology, "Fugitive VOC Emissions in the Synthetic Organic Chemicals Manufacturing Industry", Dec. 1984, pp. 1-27.
30. Gebler, S.B., and Klieger, P., "Effect of Fly Ash on the Air-Void Stability of Concrete", *Research and Development Bulletin Ser. No. 1705*.
31. Bernard, R.D., and Tansley, R.S., "A Laboratory Testing Program for Development of a Lean Mix Backfill Specification", *Scientific Service, Inc.*, May 1981.
32. Larsen, R.L., "Environmentally Sound Usages of Controlled Low Strength Materials", Oct. 1989.
33. T.T.Y.C., "The Bridge", *Linkage Transportation Research and Practice*, Vol. 6, No. 4, Summer 1992.
34. Janardhanam, R., Burns, F., and Peindl, R.D., "Mix Design for Flowable Fly-Ash Backfill Material", pp. 252-263.
35. Berry, E.E., and Malhotra, V.M., "Fly Ash in Concrete."
36. Hansen, T.C., "Modified DOE Mix Design Method for High Volume Fly Ash Concretes and Controlled Low Strength Concretes", *Magazine of Concrete Research*, No. 158, March 1992, pp. 39-45.
37. Horner, R.R., "Environmental Monitoring and Evaluation of Calcium Magnesium Acetate (CMA)", *National Cooperative Highway Research Program Report*, April 1988, pp. 1-3.
38. *Concrete Technology Today*, "Durability of Lean Concretes Containing Fly Ash", Vol. 7, No. 1, April 1986, pp. 1-5.
39. Stark, D., "Effect of Length of Freezing Period on Durability of Concrete", *Research and Development Bulletin RD 096.01T*, 1989 pp.1-9.

40. Al-Manaser, A.A., Nasser, K.W., Haug, M.D., "Consistency and Workability of Flowing Concrete", *Concrete Placement*, pp. 14-18.
41. Danial, D.E., "Permeability Test for Unsaturated Soil", *Geotechnical Testing Journal*, Vol. 6, No. 2, June 1983, pp. 81-86.
42. Horiuchi, S., Tadetsukam., Odawara, T., and Kawasaki, H., "Fly-Ash Slurry Island: 1. Theoretical and Experimental Investigations", *Journal of Material in Civil Engineering*, Vol. 4, No. 2, May 1992, pp. 117-133.
43. Kawasaki, H. Horiuchi, S., Akatsuka, M., and Sano, S., "Fly-Ash Slurry Island: 2. Construction in Hankucho Ohashi Project", *Journal of Materials in Civil Engineering*, Vol. 4, No. 2, May 1992, pp. 135-153.
44. Gray, D.H., Tons, E., Razi, M., "Use of Fly Ash in a Highway Shoulder Base Course", *DRDA Project No. 388803*, June 1985, pp. 1-31.
45. Zimmerman, L.G., "Practical Considerations for the Formulation and Use of Flowable Fill Materials", *Midwest Fly Ash and Materials*.
46. EPA, "EPA Guideline for Purchasing Cement and Concrete Containing Fly Ash", *EPA/530-SW-91-086*, January 1992.
47. Thiunalai, K., "Technology Issues for Enhancing Waste Material Utilization in Highway Construction Addressed by SHIRP-Idea Program".

MICHIGAN STATE UNIV. LIBRARIES



31293010468282