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PERFORMANCE STUDIES OF SYNTHETIC AGGREGATE CONCRETE

in cooperation with the Department of Transportation Federal Highway Administration Bureau of Public Roads

RESEARCH REPORT 81-6 STUDY 2-8-65-81 SYNTHETIC AGGREGATE RESEARCH

PERFORMANCE STUDIES OF SYNTHETIC AGGREGATE CONCRETE

by

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PREFACE

The primary objective of the synthetic aggregate research being conducted by the Texas Transportation Institute is to develop recommended acceptance criteria for synthetic aggregates for use in all phases of highway construction.

This is the sixth report issued under Research Study 2-8-65-81, one of the synthetic aggregate research studies being conducted at the Texas Transportation Institute in the cooperative research program with the Texas Highway Department and U. S. Bureau of Public Roads. The first five reports are:

"Correlation Studies of Fundamental Aggregate Properties With Freeze-Thaw Durability of Structural Lightweight Concrete," by W. B. Ledbetter, Research Report 81-1, Texas Transportation Institute, August 1965.

"Effect of Degree of Synthetic Lightweight Aggregate Pre-Wetting on the Freeze-Thaw Durability of Lightweight Concrete," by C. N. Kanabar and W. B. Ledbetter, Research Report 81-2, Texas Transportation Institute, December 1966.

"Aggregate Absorption Factor as an Indicator of the Freeze-Thaw Durability of Structural Lightweight Concrete," by W. B. Ledbetter and Eugene Buth, Research Report 81-3, Texas Transportation Institute, February 1967.

"Flexural Fatigue Durability of Selected Unreinforced Structural Lightweight Concretes," by J. C. Chakabarti and W. B. Ledbetter, Research Report 81-4, Texas Transportation Institute, July 1967.

"Suitability of Synthetic Aggregates Made From Clay-Type Soils for Use in Flexible Base," by W. M. Moore, Richard S. Van Pelt, F. H. Scrivner, and George W. Kunze, Research Report 81-5, Texas Transportation Institute, February 1968.

The authors wish to thank all members of the Institute who assisted in this research, especially Prof. C. E. Sandstedt, whose advice and counsel were instrumental in the proper conduct of this research. They would like to express special appreciation to District 15 of the Texas Highway Department which permitted the gathering of pavement samples for investigation.

The authors wish to acknowledge the guidance and assistance given by the advisory committee for this study. The members are as follows: (a) Texas Highway Department Personnel-Mr. Kenneth D. Hankins, Study Contact Representative; Mr. Ivan K. Mays, Research Area Representative; Mr. H. A. Sandberg, Jr., Materials and Tests Division Representative; and Mr. H. D. Butler, Bridge Division Representative; (b) Bureau of Public Roads Personnel-Mr. Edward V. Kristaponis, Division Representative, and Mr. W. J. Lindsay, Regional Representative.

The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the Bureau of Public Roads.

ABSTRACT

This paper reports the results of three experimental programs conducted on synthetic aggregates; physical durability, chemical durability, and mechanical durability.

In the physical durability program, five selected commercially produced and eight TTI kiln-produced lightweight coarse aggregates were investigated. Absorption characteristics and porosity values were determined for each of the aggregates. A total of 69 batches of concrete using these coarse aggregates and natural sand were mixed and tested in accordance with ASTM C290. Various degrees of saturation of the coarse aggregates at the time of mixing were obtained by immersing them in water for periods ranging from 6 hours to 180 days. The resistance of structural lightweight concrete to freezing and thawing as measured by ASTM C290 was greatly influenced by the degree of saturation of the coarse aggregate at the time of mixing. For the concretes investigated, the critical degree of saturation of the coarse aggregate at the time of mixing was found to be about 0.25.

Experiments on chemical durability centered upon the detection and possible effects of deleterious substances likely to be present, such as "underburned" clay minerals, lumps of lime and of anhydrite, and decomposable sulfides. Underburned clay minerals can sometimes be detected by petrographic and DTA methods, more definitely by X-ray diffraction. Simpler tests, such as boiling in a pressure cooker, are also reported.

It was found that lumps of lime disintegrate during mixing of concrete, but lumps of anhydrite hydrate more slowly and expand, causing swelling and distortion of concrete test bars.

All raw clays containing pyrite or marcasite yielded aggregates giving positive tests for small amounts of decomposable sulfides. Aggregates prepared with artificially high concentrations of FeS oxidized and corroded steel on repeated wetting and drying, even if their initial environment was alkaline. On the other hand, these aggregates cast in a block of concrete did not cause appreciable disintegration in the duration of the experiment. It seems probable that oxidation of the sulfide may do no harm as long as the surrounding concrete remains sufficiently alkaline to neutralize the acid formed. However, the long-term effects are still undetermined, and testing of synthetic aggregates for sulfide is recommended.

The mechanical durability program was designed as a pilot study of the abrasion resistance characteristics of synthetic aggregates. The test used in the investigation was ASTM C418-67T, "Method of Test for Abrasion Resistance of Concrete (Tentative)." This test measures abrasion in terms of the volume of material eroded by a stream of standard size sand particles, blasted onto a specified surface area. Laboratory tests were performed on specimens made with four commercially produced synthetic aggregates and one commercial natural gravel aggregate. All laboratory tests were performed on sawn surfaces to expose the aggregate and therefore amplify the effects on the aggregate. Reproducibility of the method in terms of coefficient of variation was found to vary with moisture content of the specimens. The results obtained on specimens soaked 24 hr. prior to testing (as specified in ASTM C418-67T) were more consistent than those obtained on air dry specimens. There was no significant difference in the performance of the synthetic aggregate concretes tested. However, they all exhibited higher losses than the natural gravel concrete. The cement factor of the concrete did not significantly affect the results obtained.

Core specimens were obtained from three locations on limestone aggregate concrete pavements which exhibited satisfactory and unsatisfactory in-service performance. At each location cores were taken in the wheel path and at the pavement edge where the surfaces at the edge were undisturbed by traffic. Results indicate significantly more wear on specimens from the pavement edge at location 1 (an area exhibiting excessive wear in the wheel path), than on all other cored specimens. These results indicate that the test is capable of distinguishing between a concrete surface that will perform well under traffic and one that will not.

Loss measured on the original surface of the unsatisfactory pavement was of the same order of magnitude as the loss measured on the synthetic aggregate concrete. It follows from these results that if synthetic aggregate is exposed in a concrete pavement surface (as it was in these laboratory tests) it may abrade readily under traffic wear. The test results indicate that the test could be useful in evaluating surface quality in terms of wear under traffic.

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1.1 Purpose

The over-all objective of this phase of the study is the development of a recommended synthetic aggregate classification system and performance standards for synthetic aggregate portland cement concrete. Essentially, what this objective entails is the answer to the following question:

What acceptance criteria and quality control measures must be taken to insure that synthetic aggregate concrete, when used in a highway pavement or structure system, will perform satisfactorily throughout the system's service life?

The purpose of this report is to present a summary of the investigations to date, together with the conclusions and recommendations from these investigations.

1.2 Scope

The investigation of the performance of synthetic aggregate concrete is a major undertaking. Characterization of the behavior of materials and systems becomes complex when the effects of time are introduced, as they must when performance is the primary objective. In an attempt to simplify this complex area of performance, or durability, of portland cement concrete, the area has been divided into four subareas for investigation—physical durability, chemical durability, mechanical durability, and volume change durability.

Physical durability is defined herein as the resistance of concrete to the action of repeated internal stressing from cycles of freezing and thawing or external fatigue loading.

Test procedure ASTM C290 was employed in this study to evaluate the physical durability of concrete.

Chemical durability is defined herein as the resistance of concrete to the various chemical reactions occurring on, or in, the concrete during its service life.

Mechanical durability is defined herein as the resistance of concrete to surface abrasion and wear.

Volume change durability is defined as the resistance of concrete to the detrimental effects of internal volume changes due to shrinkage and creep.

It should be emphasized that (1) the performance of concrete involves other aspects of durability and (2)the four aforementioned aspects are *not* independent of each other. But, in order to conduct a logical research program, each of the four aspects has been treated separately. Results of the investigations on physical, chemical and mechanical durability aspects are reported in the following chapters. Volume change durability will be reported later.

1.3 Conclusions and Recommendations

Conclusions and recommendations from each phase of the research are given in the following three chapters of this report. In addition, these same conclusions are repeated here for the convenience of the reader.

PHYSICAL DURABILITY INVESTIGATION

Conclusions

Theories concerning the freeze-thaw durability of structural concrete as a function of the degree of aggregate saturation have been developed and reported. Application of these theories to structural lightweight concrete has resulted in experimental agreement.

The following specific conclusions have been drawn from the data presented:

1. The rates at which the lightweight coarse aggregates studied in this investigation became saturated when given free access to water were greatly different for different aggregates (Figures 2-6 through 2-13).

2. The resistance of structural lightweight concrete to freezing and thawing as measured by ASTM C290 was greatly influenced by the degree of saturation of the coarse aggregate at the time of mixing.

3. For the concretes investigated, the critical degree of saturation of the coarse aggregate (i.e., the degree of saturation beyond which the resistance of the concrete to freezing and thawing decreased rapidly) at the time of mixing was found to be about 0.25.

4. The length of immersion time required to produce a degree of saturation of 0.25 (by method 2) ranged from about 30 minutes for aggregate D to about 3 days for aggregate R.

Practical Implication of the Physical Durability Test Results

Durability in a given environment such as ASTM C290 can be reasonably predicted. However, the infinite number of environments encountered in the field make accurate predictions an impossibility. The best one can do is assume the most severe environment expected and design accordingly.

A lightweight concrete may be made potentially nondurable (when tested by ASTM C290 after 14 days moist curing) if the coarse aggregate is more than about 0.25 saturated at the time of mixing. As always, it is very difficult to translate the meaning of these results to field use. The quantitative effect of a drying period prior to freezing temperatures has not been determined. If drying will reduce the degree of saturation of the aggregate sufficiently below 0.25 prior to the occurrence of freezing temperatures, the concrete would be expected to be durable. However, if the aggregate is less than 0.25 saturated at the time of mixing, it would probably not reach a higher degree of saturation after being cast in a bridge deck or any such location where free drainage is provided.

If one agrees that concrete should not be mixed and placed with the aggregate above critical saturation (a degree of saturation of about 0.25 for the aggregates studied), a field test method for determining the degree of saturation must be developed. Consideration should be given to the expected environment and the effect of variable drying periods in establishing any limits on the degree of saturation. Additional field information is needed on this aspect.

CHEMICAL DURABILITY INVESTIGATION

Clay Minerals in "Underburned" Aggregates

The pressure cooker test for detecting clay minerals in underburned aggregates has appeal because of its simplicity. Research being conducted on Phase II of this study is showing that this test is promising for base materials where relatively large amounts of clay minerals may exist. However, the sensitivity of this test for amounts of clay minerals that may be deleterious in concrete aggregates is hampered by the existence of dust on the rotary-kiln-produced aggregate particles. This dust is difficult to remove by dry sieving. On the other hand, washing in water may cause the rehydration of the clay minerals to an adherent film that can be dispersed only by mechanical agitation. The one underburned concrete aggregate so far tested, shown by X-ray to contain clay minerals, lost nearly as much fine material by prolonged rubbing under cold water as it did by pressure cooking and rubbing; cooking without agitation caused little loss. It seems that for concrete aggregates, the agitation is at least as important as the temperature and pressure of the pressure cooker test.

A means of detecting clay minerals in concrete aggregates needs to be developed.

Lumps of Lime and Anhydrite

As pointed out above, lumps of lime, unless very large, react so rapidly with water that they are unlikely to pass through the concrete mixer without disintegrating. Lumps of anhydrite react much more slowly and are more likely to cause subsequent popouts. But as lime and anhydrite cannot readily be distinguished by inspection, the following examination is tentatively recommended at this time.

Spread a representative sample of at least one pound (or 500 grams) of the aggregate in a thin layer on a sheet of paper or cloth or on a table top, preferably black or dark colored. Examine the aggregate visually for white lumps $\frac{1}{8}$ inch or more in diameter. If white lumps are present, the test for popout materials (autoclave test) should be run in accordance with ASTM C330.^{3.2}

Sulfides

Experiments have confirmed the expectable chemical behavior of iron sulfide and have shown that when included in a clay-ceramic aggregate it will oxidize in a wide range of pH environments and that its oxidation will accelerate the corrosion of steel with which it is in contact. This is in accord with the restrictions cited by Lea,^{3.4} in effect in Great Britain concerning the use of coal ashes, and with both the British and ASTM specifications on the use of slag containing sulfides.

How then are these observations to be reconciled with the conclusions reached by Everett & Gutt,^{3.8} Josephson, Sillers & Runner,^{3.11} and others, that blast-furnace slag containing sulfides gives satisfactory performance as aggregate in concrete and has no corrosive effect on the reinforcing steel?

Perhaps the conclusions of all the investigators, inconsistent as they are, are correct as far as they go.

The explanation may be that although the high pH of concrete does not prevent the oxidation of the sulfide, the sulfuric acid formed is immediately neutralized by the excess alkali in the concrete, and thus is prevented from either disintegrating the concrete or corroding the steel.

If this is the explanation, then sulfide in an aggregate will do no harm provided that there is at all times sufficient excess alkali in the immediately surrounding cement paste to neutralize all the sulfuric acid formed. Then perhaps the placing of an upper limit (as in ASTM and British standards) on the permissible content of sulfur compounds is the desirable solution to the problem.

It is obvious that the less the permeability of the concrete to air and water, the less the carbonation and oxidation that can occur. It should be remembered, however, that cracks, whether caused by shrinkage or by stress, offer ready access of air and water to the interior of the concrete mass, and that they remain damp much longer than the surface. Thus they offer ideal conditions for the local lowering of the pH and for the oxidation of any sulfide in the aggregate exposed along them, with accompanying attack on the concrete and on the exposed reinforcing steel.

Therefore, it is suggested that the qualitative test for decomposable sulfides, described in a TTI Special Report.^{3.12} be required on all synthetic aggregates considered for use in concrete structures so that the engineer may be aware of the possibilities of the material with which he has to deal.

MECHANICAL DURABILITY INVESTIGATION

Conclusions

The results presented were based on a limited number of tests intended to be used in determining if this test method is worth further consideration for use in evaluating the abrasion resistance characteristics of concrete.

On the basis of the results presented in this report the following tentative conclusions can be made:

(a) The abrasion coefficient is repeatable for concrete specimens of the type tested.

(b) Aggregate D concrete may be significantly less durable than aggregate R concrete, based on the soaked condition. Beyond this, no conclusion can be drawn.

(c) When the coarse aggregate is exposed, synthetic aggregate concrete abrades significantly more than natural gravel concrete, due to the low abrasion resistance of the synthetic coarse aggregate particles.

(d) When the coarse aggregate is exposed, the cement factor apparently does not significantly influence test results.

(e) On the basis of the results obtained from the three locations of existing concrete pavement, it appears that the test can be used to distinguish between a concrete that has performed satisfactorily and one that has not.

(f) The abrasion losses of laboratory specimens of

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synthetic aggregate concrete are high when compared to those of siliceous aggregate concrete and in-service limestone aggregate concrete specimens.

(g) The amount of moisture present in the specimen at time of test significantly affects test results and should be controlled. (h) The test may be of value in evaluating construction practices, such as surface finishing.

(i) The test results presented herein indicate that the test method is worth further consideration for use in evaluating abrasion resistance characteristics of concrete.

2. Physical Durability Investigation

by

Eugene Buth

2.1 Introduction

The objective of the investigation reported herein was to provide information on the resistance of structural lightweight concrete to freezing and thawing. This constitutes a portion of the over-all objectives of the study.

Five selected commercially produced and eight TTI kiln*-produced lightweight coarse aggregates were used in the program. Absorption characteristics and porosity values were determined for each of the aggregates. A total of 69 batches of concrete using these coarse aggregates and natural sand were mixed and tested in accordance with ASTM C290.^{2.1**} Various degrees of saturation of the coarse aggregates at the time of mixing were obtained by immersing them in water for periods ranging from 6 hours to 180 days.

2.2 State of the Art

Many researchers have devoted much effort to the study of the behavior of concrete subjected to freezing and thawing environments. The subject is extremely complex but not totally insurmountable. Lightweight concrete, being relatively new, has not been studied to the extent that regular weight concrete has, and less experimental information concerning its behavior is available. This is not to say there are any problems unique with its behavior. The basic problem of freezethaw durability remains the same with lightweight as with regular weight concrete; only the magnitudes of some of the parameters involved are different.

Deterioration caused by freezing and thawing is a result of excessive stresses created by the expansion of the water being frozen in the void system of the concrete. The factors influencing the amount of stress created include porosity, permeability, void size distribution, degree of saturation, freezing rate, and the ability of the paste to accommodate water being expelled from the aggregate.

The effects of the more influential parameters involved in the durability of concrete may be briefly summarized as follows. For an aggregate to be potentially deleterious, it must have sufficiently large porosity and must have a degree of saturation that is greater than some critical value. If these conditions exist, the expansion of the water during freezing will create excessive stresses causing deterioration of the concrete. Theoretically, the critical degree of saturation is 0.917. However experiments have failed to verify this value.^{2.2} Reasons for the lack of experimental verification given by Powers are:^{2.2}

- (1) non-uniform distribution of water in particle
- (2) pore size distribution (macropores that protect saturated capillaries)
- (3) non-homogenuity of particles

A lower limit of porosity exists below which excessive stresses will not be created. Instead, the expansion will be accommodated by elastic dilation of the concrete.

Two types of deterioration may be caused by nondurable aggregates. Combinations of pore size distribution or permeability and freezing rate exist for which excessive stresses may be created if the aggregate were frozen even if it were unconfined. The other possibility that exists in this respect is that of a high permeability that will allow the expanding water to escape the aggregate particle during freezing. In this instance excessive stresses may be created in the surrounding cement paste if its air content is such that it cannot accommodate the excess water.^{2.3} As Dolch^{2.4} has summarized:

Therefore, while the type of failure one gets depends on the permeability, whether or not one gets failure will depend on the several factors that determine the degree of saturation of the aggregate.

An aggregate may be saturated beyond the critical value at the time it is mixed and placed or may become critically saturated in the hardened concrete if placed where free drainage is not provided. The two major factors that appear to influence the time required for an aggregate to become critically saturated in concrete after being placed are the absorption characteristics of the aggregate and the permeability and thickness of the paste cover separating the aggregate from the external supply of water.^{2.3} In any case the important point is whether or not the aggregate is critically saturated at the time freezing occurs.

Researchers appear to be in general agreement that the subject is far too complex to ever expect to be able to predict the life of concrete in service solely on the

^{*}The TTI kiln is a 25 ft. long by 2 ft. inside diameter research rotary kiln for the closely controlled processing of synthetic aggregates in conjunction with this study.

^{**}References are contained in Section 2.7 of this chapter of the report.

basis of laboratory tests. To be able to do so would be a desirable refinement of the art. However, a satisfactory refinement would be the ability to design and produce concrete with the desired life including consideration for its environment but without undue conservatism.

2.3 Experimental Program

2.3.1 General

Structural lightweight concrete coarse aggregates from five commercial sources and eight aggregates produced with the TTI kiln were investigated in this program. Tests were conducted to determine the moisture absorption properties and porosity values of these aggregates. These data were used to determine the degree of saturation of the coarse aggregate at the time of mixing. Additional data for these aggregates are given in references 2.5 and 2.6. The behavior of concretes made from these aggregates when subjected to freezing and thawing was determined by ASTM C290.^{2.1}

2.3.2 Aggregates

Aggregate C5A was produced in the 25 ft. long, 2 ft. inside diameter, TTI research rotary kiln. The raw material was the same as that used to produce the commercial aggregate C. Aggregate C5A was fired in the research kiln under conditions which would yie'd, as closely as possible, the same physical properties as aggregate C, but with a relatively smooth, fused particle surface.

Aggregates designated by M were produced in the same research kiln from a shale obtained for investigation. Aggregate is not being commercially produced from this material.

Aggregates W6D and W7B were also produced in this same research kiln. The raw material was the same as that used to produce the commercial aggregate W. Unit weight and specific gravity values for the various aggregates are given in Table 2-1 (subsection 2.7.2).

2.3.3 Aggregate Absorption Tests

Absorption-time relationships for each of the various coarse aggregates were determined by the Bryant Method^{2.5} (as first reported by $Saxer^{2.7}$).

2.3.4 Aggregate Porosity Tests

The porosity of each of the aggregates was determined by each of the following three methods:

- Method 1: dry bulk specific gravity and powdered grain density,
- Method 2: dry bulk specific gravity and apparent specific gravity using the pressure pycnometer and

Method 3: absorption of CCl₄.

Method 1: The dry bulk specific gravity obtained from the Bryant absorption and specific gravity test was used in this determination. The powdered (or absolute) grain density was determined by grinding the sample to pass a No. 80 sieve and placing the powdered sample in a pycnometer. Mercury was used as the filling medium in this pycnometer. The porosity was then determined by the equation:

$$P_1 = 1 - \frac{G_B}{G_A}$$

where:

 $G_{\rm B} = dry$ bulk specific gravity,

 G_A = specific gravity of powdered sample and

 $P_1 = porosity.$

Method 2: The dry bulk specific gravity used to determine the porosity by this method is that obtained

TABLE 2-1. PROPERTIES OF LIGHTWEIGHT COARSE AGGREGATES

	Unit	Drv Bulk	Abs	solute Specific Gravit	y
Aggregate	Wt.	Sp. Gr.	Method 1	Method 2	Method 3
R1 R2	$\begin{array}{c} 46.8\\ 48.1\end{array}$	$1.39\\1.43$	2.50	2.11 2.17	2.42
C1 C2 C3 C5A	38.1 38.5 38.1 37.4	$1.36 \\ 1.40 \\ 1.27 \\ 1.19$	$2.21 \\ 2.34 \\ 2.15 \\ 1.99$	2.02 1.99 1.98	$2.37 \\ 2.43 \\ 2.41 \\ 2.39$
S3	42.0	1.41	2.45	2.32	2.47
E4 E6 E7	$\begin{array}{c} 45.3 \\ 44.9 \\ 44.8 \end{array}$	$1.36 \\ 1.40 \\ 1.38$	2.31	$2.11 \\ 2.06 \\ 2.14$	2.05
D2 D3	$\begin{array}{c} 35.8\\ 39.4\end{array}$	1.18 1.24	$\begin{array}{c} 2.22\\ 2.17\end{array}$	$\begin{array}{c} 2.20\\ 2.16\end{array}$	$2.29 \\ 2.32$
W3 W6D W7B	40.8 40.6 47.8	1.48 1.11 1.31		$2.24 \\ 2.14 \\ 1.74$	
S2M2B S2M2C S2M3C S2M3D	42.5 37.6 33.9 37.9	$1.20 \\ 1.12 \\ 1.09 \\ 1.21$		2.07 2.07 2.02 2.04	

from the Bryant absorption and specific gravity test. The apparent specific gravity was determined using a pressure pycnometer which is capable of rapidly saturating the aggregate under 1200 psi pressure. The porosity was determined by the equation:

$$P_2 = 1 - \frac{G_B}{Gpp}$$

where:

 $G_{B} = dry$ bulk specific gravity,

- Gpp = apparent specific gravity (using pressure pycnometer) and
- $P_2 = porosity.$

Method 3: The sample was saturated with CCl_4 under atmospheric pressure. The surface of particles were then wiped dry. The amount of CCl_4 absorbed by the sample and the volume of pores was determined. The bulk volume of the sample was determined by weighing the saturated sample in air and immersed in CCl_4 . The porosity of the aggregate was then determined by dividing the pore volume by the bulk volume of the sample.

2.3.5 Concrete Freezing and Thawing Tests

A total of 69 batches of concrete were mixed and cast from five selected lightweight coarse aggregates produced commercially and eight produced in a research rotary kiln. Various degrees of prewetting of the coarse aggregates were obtained by immersing the aggregates in water for periods ranging from six hours to 180 days prior to mixing. All concrete mixtures had a nominal cement factor of five sacks per cu. yd., three to four in. slump, and five to six percent air content. A natural sand was used in all mixtures. The proportions of coarse and fine aggregate were held constant. The various coarse aggregates and the amount of prewetting they received were the only parameters varied. Mixture proportions and properties of the concretes are given in Table 2-2 (subsection 2.7.2). Three 3 in. \times 3 in. \times 16



Figure 2-1. Absorption-time relationships for commercially produced coarse aggregates for first 24 hours.

in. specimens for freeze-thaw testing were cast from each batch. These specimens were moist cured until 14 days of age at which time they were subjected to cycles of freezing and thawing according to ASTM C290. Standard cylinders were cast and moist cured for compressive strength determinations at 14 and 28 days of age (strength values are given in Table 2-3 in subsection 2.7.2).

2.4 Results and Discussion

2.4.1 Aggregate Experimental Program

Absorption-time curves for the aggregates are shown in Figures 2-1 through 2-4. For the details of the test

	Days	Lot	Per	Cent Abs	olute Volu	ime	Air	Slump	Initial Unit Woight	
Batch	Immersed	No.	Cement	Water	F. A.	C. A.	(%)	(in.)	pcf	Nf*
3FTR 4FTR 5FTR	14 14 14	$1 \\ 1 \\ 2$	8.2 8.7 8.7	$20.9 \\ 19.0 \\ 18.7$	34.9 33.1 33.0	$31.7 \\ 33.8 \\ 33.6$	$4.3 \\ 5.4 \\ 6.0$	$4.0 \\ 3.0 \\ 3.8$	$114.4 \\ 113.2 \\ 115.1$	$100+\\100+\\100+$
14R 24R	21 21	$\frac{2}{2}$	8.7 8.2	$\begin{array}{c} 15.8\\ 18.3 \end{array}$	$\begin{array}{c} 38.2\\ 36.6\end{array}$	$\begin{array}{c} 33.3\\ 31.4 \end{array}$	$\begin{array}{c} 4.0 \\ 5.5 \end{array}$	$\begin{array}{c} 3.0\\ 3.5\end{array}$	$115.6 \\ 115.6$	300 + 265
2-25R	28	2	8.3	17.6	36.7	31.9	5.5	3.0	114.8	300 +
16R 26R	$\begin{array}{c} 42\\ 42\end{array}$	$2 \\ 2$	8.8 8.5	$\begin{array}{c} 15.4 \\ 16.4 \end{array}$	$\begin{array}{c} 36.6\\ 37.2 \end{array}$	$\begin{array}{c} 33.7\\ 32.4\end{array}$	5.5 5.5	$\begin{array}{c} 5.0\\ 3.0\end{array}$	$\begin{array}{c} 116.8\\ 116.4\end{array}$	$\begin{array}{c} 130 \\ 125 \end{array}$
18R 2-28R	85 85	$2 \\ 2$	8.8 8.8	$\begin{array}{c} 17.2\\ 17.2\end{array}$	$\begin{array}{c} 33.7\\ 33.4\end{array}$	$\begin{array}{c} 34.3\\ 34.1\end{array}$	$\begin{array}{c} 6.0 \\ 6.5 \end{array}$	$\begin{array}{c} 4.5\\ 3.5\end{array}$	115.6	$\begin{array}{c} 217 \\ 86 \end{array}$
7FTR	180	2	8.7	19.5	33.1	33.7	5.0	5.0		21
$0.5\mathrm{D}$	1/4	3	8.6	21.4	31.8	32.7	5.5	4.0	113.2	300 +
$1.5\mathrm{D}$	1/2	3	8.9	18.7	32.7	33.7	6.0	3.5	113.6	300+
10D 20D	1 1	3 3	8.7 9.1	$\begin{array}{c} 20.4 \\ 17.9 \end{array}$	32.0 33.9	$\begin{array}{c} 32.9\\ 34.6\end{array}$	$\begin{array}{c} 6.0\\ 4.5\end{array}$	$\begin{array}{c} 3.0\\ 4.0\end{array}$	$113.2 \\ 113.6$	84 54

TABLE 2-2. CONCRETE MIX DATA

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Table 2-2 Concrete Mix Data

	Days A corregate	, Lot	Per Cent Absolute Volume		Air	Initial Slump Unit Weight				
Batch	Immersed	No.	Cement	Water	F. A.	С. А.	(%)	(in.)	(pcf)	Nf*
11D 21D	3 3	3	8.9 8.8	$\begin{array}{c} 18.9 \\ 20.0 \end{array}$	$\begin{array}{c} 32.9\\ 32.6\end{array}$	33.8 33.3	$\begin{array}{c} 5.5\\ 5.3\end{array}$	$\begin{array}{c} 3.2\\ 3.0\end{array}$	$\begin{array}{c} 110.0\\ 114.0 \end{array}$	$\begin{array}{c} 34 \\ 45 \end{array}$
12D	7	3 .	8.9	19.4	32.8	33.7	5.2	3.0	115.2	8
13D 5FTD	14 14	$\frac{3}{2}$	$9.0\\8.9$	$\begin{array}{c} 20.3\\ 17.8 \end{array}$	$\begin{array}{c} 29.9\\ 36.7 \end{array}$	$\begin{array}{c} 34.3\\ 31.6\end{array}$	$\substack{6.5\\5.0}$	$\begin{array}{c} 3.2\\ 3.2\end{array}$	116.1	8 27
3FTE 4FTE 5FTE	14 14 14	4 4 6	8.3 8.3 8.2	$17.6 \\ 17.1 \\ 16.6$	$36.0 \\ 37.4 \\ 37.1$	$32.2 \\ 32.2 \\ 32.1$	5.9 5.0 6.0	$4.5 \\ 3.0 \\ 3.5$	$125.2 \\ 120.8 \\ 117.1$	$44 \\ 100 + \\ 100 +$
$14 \mathrm{E}$ $24 \mathrm{E}$	$\begin{array}{c} 21\\ 21 \end{array}$	6 6	8.9 8.3	14.4 17.1	$\begin{array}{c} 36.6\\ 36.7\end{array}$	$\begin{array}{c} 33.6\\ 31.9\end{array}$	$\begin{array}{c} 6.5\\ 6.0\end{array}$	$\begin{array}{c} 3.2\\ 3.0\end{array}$	$115.2 \\ 118.0$	$300 + \\ 300 +$
16E 26E	$\begin{array}{c} 42\\ 42\end{array}$	6 6	8.7 8.8	$\begin{array}{c} 16.8\\ 16.7\end{array}$	$\begin{array}{c} 36.2\\ 36.2 \end{array}$	$\begin{array}{c} 33.3\\ 33.3\end{array}$	$\begin{array}{c} 5.0 \\ 5.0 \end{array}$	$\begin{array}{c} 3.0\\ 3.5\end{array}$	$\begin{array}{c} 117.6\\ 118.0\end{array}$	$300 + \\ 195$
17E 27E	60 60	6 6	8.5 8.6	$\begin{array}{c} 17.9 \\ 17.2 \end{array}$	$\begin{array}{c} 35.3\\ 35.5\end{array}$	$\substack{32.5\\32.7}$	$\begin{array}{c} 5.8 \\ 6.0 \end{array}$	$\begin{array}{c} 3.0\\ 3.5\end{array}$	$\begin{array}{c} 117.6\\ 116.4 \end{array}$	$\begin{array}{c} 149 \\ 183 \end{array}$
18E 28E	85 85	$\begin{array}{c} 6 \\ 6 \end{array}$	$\begin{array}{c} 8.4\\ 8.8\end{array}$	$\begin{array}{c} 18.9 \\ 16.3 \end{array}$	$\begin{array}{c} 35.0\\ 35.4\end{array}$	$\begin{array}{c} 32.2\\ 33.5\end{array}$	$\begin{array}{c} 5.5 \\ 6.0 \end{array}$	$\begin{array}{c} \textbf{3.0} \\ \textbf{4.5} \end{array}$	$\begin{array}{c} 118.4\\ 117.6\end{array}$	81 59
0.5S 0.6S	1/4 1/4	3	$\begin{array}{c} 8.6 \\ 8.6 \end{array}$	$\begin{array}{c} 21.5\\ 21.2 \end{array}$	$\begin{array}{c} 31.6\\ 31.5\end{array}$	$\begin{array}{c} 32.8\\ 32.7\end{array}$	$\begin{array}{c} 5.5 \\ 6.0 \end{array}$	$\begin{array}{c} 3.0\\ 3.0\end{array}$	$\begin{array}{c} 112.8\\ 112.8\end{array}$	300 + 300
1.5S	.//2	3	8.7	21.7	31.7	32.9	5.0	3.0		300
10S 203	1 1	$\frac{3}{3}$	8.5 8.8	$\begin{array}{c} 21.1 \\ \underline{21.3} \end{array}$	$\begin{array}{c} 31.1\\ 32.3\end{array}$	$\begin{array}{c} 32.3\\ 33.5\end{array}$	$\begin{array}{c} 7.0 \\ 4.1 \end{array}$	$3.5 \\ 3.8$	$\begin{array}{c} 116.0\\ 116.0\end{array}$	$\frac{188}{252}$
11S 21S 31S	3 3 3	3 3 3	8.8 9.0 8.7	$21.2 \\ 19.7 \\ 19.9$	$32.1 \\ 33.3 \\ 32.4$	$33.3 \\ 34.0 \\ 33.0$	$\begin{array}{c} 4.6\\ 4.0\\ 6.0\end{array}$	$5.5 \\ 3.0 \\ 3.5$	$112.8 \\ 117.2 \\ 115.6$	$78 \\ 95 \\ 179$
$\begin{array}{c} 128\\ 228\end{array}$	7 7	33	8.7 8.8	$\begin{array}{c} 21.3 \\ 19.6 \end{array}$	$\begin{array}{c} 32.4\\ 33.4\end{array}$	$\begin{array}{c} 33.1\\ 33.4\end{array}$	$\begin{array}{c} 4.5 \\ 4.8 \end{array}$	$3.5 \\ 3.25$	$\begin{array}{c} 118.4\\ 116.8\end{array}$	$\begin{array}{c} 46 \\ 52 \end{array}$
5FTS	14	3	8.8	20.3	32.6	33.8	4.5	3.8	118.2	11
0.5C	1/4	3	8.9	19.1	32.3	34.0	5.7	4.0	111.6	300 +
1.5C 10C 20C 30C	√₂ 1 1 1	3 2 2 3	8.7 8.3 8.9 9.0	$20.7 \\ 23.2 \\ 21.7 \\ 21.4$	$31.5 \\ 31.4 \\ 30.6 \\ 32.4$	$33.1 \\ 31.6 \\ 33.8 \\ 34.2$	$\begin{array}{c} 6.0 \\ 5.5 \\ 5.0 \\ 3.0 \end{array}$	$3.0 \\ 3.0 \\ 3.0 \\ 4.7$	$110.8 \\ 114.8 \\ 112.4 \\ 116.0$	$300+\ 51\ 102\ 87$
11C 21C 31C	3 3 3	3 3 3	8.8 8.9 8.9	$21.0 \\ 20.7 \\ 19.9$	$31.8 \\ 32.1 \\ 32.0$	33.5 33.8 33.7	$4.9 \\ 4.5 \\ 5.5$	$\begin{array}{c} 3.0\\ 4.0\\ 4.0\end{array}$	$117.2 \\ 116.4 \\ 114.4$	$\begin{array}{c} 83\\52\\237\end{array}$
12C 22C 1-C5A 2-C5A	7 7 7 7	3 3 C5A C5A	8.8 8.8 8.9 8.9	$19.5 \\ 18.9 \\ 19.9 \\ 19.7$	31.7 33.9 32.2 32.3	33.5 33.3 33.9 34.0	$6.5 \\ 5.1 \\ 5.1 \\ 5.1 \\ 5.1$	$3.5 \\ 4.0 \\ 4.0 \\ 3.0$	$116.0 \\ 113.6 \\ 109.6 \\ 108.0$	$45 \\ 54 \\ 300 + \\ 300 + $
2-3FTC 2-4FTC 2-5FTC	14 14 14	$egin{array}{c} 1 \\ 1 \\ 2 \end{array}$	8.3 8.4 8.1	$21.0 \\ 20.2 \\ 21.3$	$34.3 \\ 34.4 \\ 33.3$	$32.1 \\ 32.2 \\ 31.8$	$4.3 \\ 4.8 \\ 5.5$	$\begin{array}{c} 3.5\\ 4.0\\ 3.0\end{array}$	$117.6 \\ 115.2 \\ 115.6$	$95 \\ 65 \\ 46$
1W 2W 3W 4W 5W 6W	14 14 14 14 14 14	3 3 W6D W7B W7B W6D	8.8 8.7 9.2 9.1 9.0 9.0	$20.1 \\ 20.1 \\ 18.3 \\ 18.6 \\ 18.4 \\ 17.8$	33.8 33.2 34.8 33.9 33.5 33.7	$33.1 \\ 32.5 \\ 31.7 \\ 33.9 \\ 33.6 \\ 33.5$	$\begin{array}{c} 4.2 \\ 5.5 \\ 6.0 \\ 4.5 \\ 5.5 \\ 6.0 \end{array}$	$3.0 \\ 3.0 \\ 4.2 \\ 3.0 \\ 4.0 \\ 3.0$	$117.2 \\ 116.2 \\ 109.2 \\ 114.8 \\ 115.2 \\ 116.8 \\$	$27 \\ 46 \\ 200 + \\ 140 \\ 200 + \\ 200 + $
3M 4M 5M 6M 7M 8M	14 14 14 14 14 14	S2M2B S2M2B S2M2C S2M2C S2M3C S2M3D	8.7 8.5 8.7 8.7 8.8 8.7	$16.8 \\ 17.2 \\ 18.1 \\ 18.8 \\ 17.2 \\ 17.3 \\$	$\begin{array}{c} 34.9\\ 34.0\\ 34.9\\ 34.6\\ 35.0\\ 35.0\\ 35.0\end{array}$	33.1 32.3 32.8 32.6 33.2 33.0	6.5 8.0 5.5 5.3 5.8 6.0	3.0 3.2 3.0 3.5 3.2 3.2	$112.4 \\110.8 \\113.2 \\111.6 \\110.0 \\111.2$	$300 + 250 \\ 300 + 300 $

*Number of cycles to failure by ASTM C290

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Figure 2-2. Absorption-time relationships for research kiln produced coarse aggregates for first 24 hours.



Batch No.	14-day Cmp. Str.	28-day Cmp, Str.
10S	3270	3820
20S	3420	3610
11S	3270	3730
21S	3830	4060
12S	3950	4460
22S	4120	
3FTS	3180	3740
4FTS	3490	3900
5FTS	3490	3930
10C	3090	3420
$\overline{20C}$	2660	2870
30C	3020	3550
ĨĨČ	3330	3620
$\tilde{21C}$	3190	3570
12Č	3250	3510
22C	3360	
3FTC	3390	3780
AFTC	3060	3540
SFTC	2710	3240
100	3740	4000
2010	4200	4690
11D	4020	4440
91D	3400	3990
1210	3660	3870
12D	4110	0010
1017	3360	3810
	2420	3980
4F I D 5 E T D	2670	- 4260
	2000	2660
1415	4110	3640
23E	9990	4960
101	2620	4303
2615	3030	4200
SF LEA	3100	9090
4.FTE	3400	0900 9570
5FTE	3200	0010 4040
14K	3910	4240
24R	3460	3320
16R	3870	4080
26K	3420	3880
3FTR	3200	3650
4FTR	3710	4330
5FTR	3820	4180
1FTW	3150	3620
2FTW	2820	3520
3FTW	2750	. 3160
4FTW	2860	2980
5FTW	3150	3590
6F"TW		3620
,		



Figure 2-3. Absorption-time relationships for commercially produced coarse aggregates.

method used to develop these absorption-time curves, the reader is referred to Reference 2.5. The wide range in absorption values is one of the first and most obvious differences noticed among lightweight aggregates. However, the important difference among the aggregates is not the value of the absorption, but the rate at which the aggregates become saturated. The slope of the absorption-time curve, a value which changes with time or degree of saturation, is a measure of the rate at which the aggregate is "soaking up" water. This is an important property of the aggregate, since it is one of the parameters that influences the ease with which an aggregate may become critically saturated.

Determination of the degree of saturation of a coarse aggregate requires the determination of its porosity and the amount of the absorbed water. Three different techniques were used to determine porosity, each yielding different values for a particular aggregate.



Figure 2-4. Absorption-time relationships for research kiln produced coarse aggregates.

These values of porosity are compared in Figures 2-5a and 2-5b. P_1 , P_2 , and P_3 represent the porosity obtained by methods 1, 2, and 3, respectively. For each aggregate, P_1 is greater than P_2 indicating that powdering the sample is probably exposing more aggregate voids than are being filled in the pressure pycnometer at 1200 psi pres-The relative magnitude of P3 is not consistent sure. when compared to either of the other two values. This is, in all probability, a result of variations in attempting to physically obtain a saturated-surface dry condition of the aggregate. The porosity values of the aggregates tested are very definitely large enough to accommodate sufficient water to cause disruption when frozen. However, some of the voids contained in these aggregates are isolated and not subject to saturation under atmospheric pressure.

The degree of saturation of the coarse aggregates after various lengths of immersion time was determined by using the porosity values determined by the three methods discussed earlier. Curves illustrating these values and the close agreement of the three methods are given in Figures 2-6 through 2-13. Obviously, the degree of saturation obtained after a given immersion time is much greater for some aggregates than for others. This is in agreement with Powers^{2,2} statement that,

Various observations . . . suggest that a principal difference among different kinds of rock particles is the rate at which they become saturated when given free access to water.

The immersion time required for the degree of saturation by method 2 to reach 0.25 is illustrated for the various aggregates in Figure 2-14. Approximately the same discrimination among the aggregates can be obtained by comparison of their aggregate absorption factor (the difference between percent absorption at 1000 min. and 100 min. of immersion time)^{2.5} as indicated in Figure 2-15. Values of 24-hour absorption by the Bryant Method^{2.5} are presented in Figure 2-16 for ready reference.

2.4.2 Concrete Freezing and Thawing Program

A relationship was found to exist between the number of cycles to failure of the concrete, N_{f} , by ASTM







Figure 2-5b. Porosity values of the coarse aggregates.



Figure 2-6. Relatonship between degree of saturation by method 1 and immersion time for commercially produced coarse aggregates for first 24 hours.



Figure 2-7. Relationship between degree of saturation by method 1 and immersion time for commercially produced coarse aggregates.



Figure 2-8. Relationship between degree of saturation by method 2 and immersion time for commercially produced coarse aggregates for first 24 hours.



Figure 2-9. Relationship between degree of saturation and immersion time for research kiln produced coarse aggregates for first 24 hours.



Figure 2-10. Relationship between degree of saturation by method 2 and immersion time for commercially produced coarse aggregates.



Figure 2-11. Relationship between degree of saturation and immersion time for research kiln produced coarse aggregates.



Figure 2-12. Relationship between degree of saturation by method 3 and immersion time for commercially produced coarse aggregates for first 24 hours.



Figure 2-13. Relationship between degree of saturation by method 3 and immersion time for commercially produced coarse aggregates.

C290,^{2.1} and days of immersion of the coarse aggregate prior to mixing (Figues 2-17 through 2-21). The number of cycles to failure of the concrete is the number of cycles at which the square of the fundamental transverse frequency of vibration of the specimen decreased to 60 percent of its original value (ASTM C215^{2.1}). The maximum allowable length of immersion time of the coarse aggregate prior to mixing for a given level of concrete durability is considerably different for different coarse aggregates. This is a result of the different saturation rates of the aggregates. Some of the aggregates were more highly saturated than others at the time of mixing even though they were immersed in water for the same length of time.

The data points with attached arrows plotted at 100 cycles were obtained from an earlier series of tests, which were terminated at 100 cycles. There is no reason to believe the specimens could not have withstood 300 cycles. There is one unexplained failure for aggregate E concrete at 44 cycles. This point is not felt to be representative and thus is ignored in the analysis. Data

for aggregate C5A concrete are included in Figure 2-18 with the data for aggregate C concrete. The source of raw material for the commercially produced aggregate C was used to make aggregate C5A. Aggregate C5A was burned in the research kiln under conditions which would yield, as closely as possible, the same physical properties as aggregate C. A comparison of these properties can be made in Table 2-1. The primary difference in the appearance of the two aggregates is that a particle of aggregate C5A is a single mass with a relatively smooth surface, whereas a particle of aggregate C appears to be made up of several smaller particles that have been partially fused together. The surface texture of aggregate C is rather rough and it does *not* have a fused outer surface. For this reason, it is believed that the values of porosity and dry bulk specific gravity obtained for aggregate C may not be valid.

2.4.3 Relationships Developed

The resistance of structural lightweight concrete to freezing and thawing as measured by ASTM $C290^{2.1}$ is greatly influenced by the degree of saturation of the



Figure 2-14. Immersion time required for degree of saturation of the coarse aggregate as determined by method 2 to reach 0.25.

coarse aggregate at the time of mixing as indicated by Figures 2-22 through 2-24. An arbitrary limiting curve has been drawn on each of the three figures. With the exception of some data points for aggregate C concrete, all data points are to the right and above (on the safe side) the limiting curve. The aggregate C concretes present a problem in that the measured degree of saturation indicates a durability much greater than actually obtained. This may not void the degree of saturation concept; rather the parameters determining the degree of saturation may be improperly evaluated for this one aggregate by the test methods employed. Although only one value of degree of saturation was used for aggregate C5A, these data fit the general trend much better than those of aggregate C concrete.

If the problem with aggregate C concretes is neglected, a degree of saturation of 0.25 to 0.3 (determined by method 2) may be chosen as the value distinguishing non-durable from durable concrete (by ASTM C290^{2.1}). If 0.25 is used with Figures 2-8 and 2-10, it is found that this degree of saturation is reached at only 30 minutes of immersion for aggregate D but is not reached until after 3 days of immersion for aggregate R. This serves to accent the primary difference among the different aggregates i.e., the rate at which they become saturated, given free access to water.

The relatively close agreement of degree of saturation and the relationships between number of cycles to failure (N_f) and degree of saturation indicate that any of the methods employed will result in about the same relationship.

No attempt was made to determine if failures of the concretes were caused by excessive stresses inside the aggregate particles or by excessive stresses in the paste caused by water being expelled from the aggregate particle during freezing. If the latter were true, a higher entrained air content would be beneficial in increasing the durability of the concrete.

2.5 Summary and Conclusions

Theories concerning the freeze-thaw durability of structural concrete as a function of the degree of aggregate saturation have been developed and reported. Application of these theories to structural lightweight concrete have resulted in experimental agreement.

The following specific conclusions have been drawn from the data presented:

1. The rate at which the lightweight coarse aggregates studied in this investigation become saturated when given free access to water was greatly different for different aggregates (Figures 2-6 through 2-13).

2. The resistance of structural lightweight concrete to freezing and thawing as measured by ASTM C290 was greatly influenced by the degree of saturation



Figure 2-15. Aggregate absorption factor, the difference between percent absorption at 1000 min. and 100 min. of immersion time, for the coarse aggregate.

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Figure 2-16. Twenty-four-hour absorption values for the coarse aggregates by the Bryant Method.

of the coarse aggregate at the time of mixing (Figures 2-22 through 2-24).

3. For the concretes investigated the critical degree of saturation of the coarse aggregate (i.e., the degree of saturation beyond which the resistance of the concrete to freezing and thawing decreased rapidly) at the time of mixing was found to be about 0.25.

4. The length of immersion time required to produce a degree of saturation of 0.25 (by method 2) ranged



Figure 2-17. Effect of coarse aggregate immersion time prior to mixing on number of cycles to failure by ASTM C290 for aggregate R concrete.

from about 30 minutes for aggregate D to about 3 days for aggregate R.

2.6 Practical Implication of Test Results

Durability in a given environment such as ASTM C290 can be reasonably predicted. However, the infinite



Figure 2-18. Effect of coarse aggregate immersion time prior to mixing on number of cycles to failure by ASTM C290 for aggregate C concrete.

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Figure 2-19. Effect of coarse aggregate immersion time prior to mixing on number of cycles to failure by ASTM C290 for aggregate S concrete.

number of environments encountered in the field make accurate predictions an impossibility. The best one can do is assume the most severe environment expected and design accordingly.

A lightweight concrete may be made potentially nondurable (when tested by ASTM C290 after 14 days moist curing) if the coarse aggregate is more than about 0.25 saturated at the time of mixing. As always, it is very difficult to interpret the meaning of these results for field use. The quantitative effect of a drying period prior to freezing temperatures has not been determined. If drying will reduce the degree of saturation of the aggregate sufficiently below 0.25 prior to the occurrence of freezing temperatures, the concrete would be expected



Figure 2-20. Effect of coarse aggregate immersion time prior to mixing on number of cycles to failure by ASTM C290 for aggregate D concrete.



Figure 2-21. Effect of coarse aggregate immersion time prior to mixing on number of cycles to failure by ASTM C290 for aggregate E concrete.

to be durable. If the aggregate is less than 0.25 saturated at the time of mixing, it would probably not reach a higher degree of saturation after being cast in a bridge deck or any such location where free drainage is provided.

If one agrees that concrete should not be mixed and placed with the aggregate above critical saturation (a degree of saturation of about 0.25 for the aggregates studied), a field test method for determining the degree of saturation must be developed. Consideration should be given to the expected environment and the effect of drying periods in establishing any limits on the degree of saturation. Additional information is needed on this aspect.



Figure 2-22. Relationship between number of cycles to failure (ASTM C290) and degree of saturation of the coarse aggregate at the time of mixing by method 1.



Figure 2-23. Relationship between number of cycles to failure (ASTM C290) and degree of saturation of the coarse aggregate at the time of mixing by method 2.

2.7 References for Chapter 2

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- 2.2. Powers, T. C., "Basic Considerations Pertaining to Freezing and Thawing Tests," *Proceedings*, ASTM, Vol. 55, 1955, p. 1132.
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Figure 2-24. Relationship between number of cycles to failure (ASTM C290) and degree of saturation of the coarse aggregate at the time of mixing by method 3.

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- 2.6. Ledbetter, W. B., "Correlation Studies of Fundamental Aggregate Properties With Freeze-Thaw Durability of Structural Lightweight Concrete," Research Report 81-1, Texas Transportation Institute, Texas A&M University, August, 1965.
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3. Chemical Durability of Synthetic Aggregates

by

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3.1 Introduction

Research on the chemical durability of synthetic aggregates has centered chiefly upon the detection of the possibly deleterious substances which are likely to be present in aggregates made from clay or shale and on the effects which these substances may have on the durability of the aggregates and on the concrete made from them.

3.2 Possible Deleterious Constituents

3.2,1 General

Many clays and shales contain, in addition to the clay minerals and silica, such minerals as calcite, dolomite, siderite, gypsum, anhydrite, pyrite, and marcasite, as well as organic matter. Any of these constituents, except silica, if incompletely transformed or eliminated in the burning process, can give rise to substances which, if not proven harmful in concrete, at least must be regarded with suspicion.

3.2.2 Underburned Clay Minerals

In a well-burned aggregate the clay minerals should be completely dehydrated and transformed by reaction and fusion into an amorphous glass, which may contain small amounts of new high-temperature minerals of various types. But if this process is incomplete, because of either too low a temperature or too short a time in the kiln, the dried clay minerals can again take up water or possibly participate in other harmful reactions with accompanying expansion and disintegration of the aggregate particle and probable cracking of the adjacent concrete.^{3.1*}

3.2.3 Calcite, Dolomite, and Siderite

At kiln operating temperatures calcite, dolomite, and siderite normally lose carbon dioxide to become calcium, magnesium, and iron oxides. These oxides in turn react with clays to form the fusible silicates of the glass of which the finished aggregate is composed. Thus if these minerals are distributed in fine particles throughout the clay, they help rather than hinder the manufacture of synthetic aggregate. But if they are localized in the raw material as crystals, fossils, or concretions, much of the resulting oxide may not be in direct contact with the clay and will be left in the aggregate as lumps. If the lumps of CaO and MgO do not completely hydrate and disintegrate in the concrete mixer, they may cause expansion and popouts in the concrete as they absorb water and/or carbon dioxide (ASTM C330-64T).^{3.2}

3.2.4 Gypsum and Anhydrite

Gypsum, $CaSO_4 \cdot 2H_2O_3$, is not likely to pass through a kiln without losing its water and becoming anhydrite, CaSO₄. This latter substance should not cause any difficulty if uniformly distributed through the aggregate in fine particles, although if the aggregate is soaked in water and then allowed to dry, the calcium sulfate (now gypsum again) will tend to form an efflorescent coating on the surface of the aggregate particles. But in many clays gypsum occurs as layers, pockets, and clusters of small crystals, which produce lumps of anhydrite in the kiln product. Anhydrite takes up water slowly, but with considerable expansion. Consequently, such lumps are even more likely to cause popouts in concrete than are lumps of lime, because they are less likely to rehydrate and disintegrate in the mixer before the setting of the cement. In fact, considering the rapidity and energy with which burned lime, CaO, reacts with water, it is possible that many of the popouts attributed to lime in concrete are actually due to anhydrite.

3.2.5 Iron Sulfides

Many clays and shales contain widely disseminated microscopic crystals of pyrite or marcasite (both are FeS_2), and in some deposits these minerals are localized as sizable crystals or aggregates of crystals. In addition, clays rich in organic matter often contain finely divided FeS disseminated throughout.

At high temperatures in the presence of abundant air, the sulfur in all of these sulfides should be completely oxidized and driven off as SO_2 or SO_3 . But these ideal conditions for sulfur removal do not exist in many rotary kilns, and if sulfides are abundant in the raw material, some of them tend to remain in the kiln product. With insufficient air the FeS₂ loses only half its sulfur and changes to FeS, which is decomposable by dilute acids. Thus if an aggregate particle containing FeS is exposed to the weather in a reasonably humid climate, the FeS might be expected to be decomposed and oxidized by water, carbonic acid, and oxygen.

The exact course of the oxidation reactions involved probably varies with conditions, but the final products,

*References are contained in Section 3.6 of this chapter of the report. as observed on rock outcrops and masonry buildings, are limonite (ferric hydroxide) and sulfuric acid. The latter can migrate with the water and can attack adjacent concrete.

The oxidation and disintegration of marcasite, with attack on the surrounding material by the sulfuric acid formed, have been observed to occur in a few months where specimens have been stored under damp atmospheric conditions. The less common mineral pyrrhotite (essentially FeS) likewise oxidizes fairly rapidly on mine dumps. With most pyrite the reactions apparently proceed more slowly, although Midgley^{3.3} states that this mineral occurs in both a reactive and a nonreactive form.

From the above behaviors it seems apparent that sulfides, especially those like FeS which are decomposable by dilute acids, may be deleterious in aggregates. Nevertheless there seems to be considerable difference of opinion as to their effects, if any, in concrete.

Hydrogen sulfide gas is commonly evolved in sewers from the bacterial decomposition of sulfur compounds in the sewage.^{3.4, 3.5} This gas dissolves in the moisture clinging to the walls and roof of the sewer above the water line, where it oxidizes to sulfuric acid and attacks the concrete. Rates of attack of one-quarter to one-half inch per year have been recorded.^{3.4}

Lea^{3.4} also states:

It used to be considered that the sulfur compounds in clinker (coal ashes, generally called cinders in the U. S.) aggregates were responsible for the troubles experienced with them, but it has been shown that in plain concrete they are rarely a source of trouble provided the maximum content is limited. In reinforced concrete they do, however, in combination with other factors, cause accelerated corrosion of the reinforcement. . . . clinker aggregates are banned in Great Britain for reinforced concrete, and their use in any position in contact with steelwork is also prohibited.

Considerable research has been done on the durability of concrete made from blast-furnace slag, which commonly contains sulfides of calcium, iron, and/or manganese, ranging up to 2.5% sulfur. Lea^{3.4} says:

The presence of calcium sulfide . . . is not a source of risk unless its content becomes excessive. . . The British standard limits the content of acid-soluble SO_3 to 0.7%, and that of total sulfur to 2 percent S.

The American Society for Testing and Materials does not mention sulfides in specifications for lightweight aggregates, including slag and cinders. They do, however, limit the sulfide sulfur in slag cement to 2.0% S. (ASTM C358-66T).^{3.6}

Gutt, Kinniburgh, and Newman^{3.7} prepared prisms of concrete with slag aggregate containing 0.89 to 2.40% decomposable sulfide and exposed them to the weather in England for two years. They concluded that the "volume stability" of the slag concrete was satisfactory and was similar to that of gravel concrete. Everett & Gutt,^{3.8} reporting on the effect of two years of exposure of reinforced concrete prisms made with the same slag aggregates, concluded that steel embedded in concrete made with slag aggregate will undergo no more severe corrosion than concrete made with the more usual dense gravel aggregate. In a brief review of the sulfide problem these authors cite Parker^{3.9} as attributing the expansion of certain concrete to oxidation of sulfide in slag aggregate, followed by the reaction of the sulfate produced with hydrated calcium aluminate to form calcium sulfo-aluminate, which resulted in an increase in volume and a disintegrative effect. Midgley^{3.3} offers a similar explanation for the effects of the oxidation of reactive pyrite.

Steinour^{3.10} attributes the usual absence of corrosion of the reinforcing steel in normal concrete to the high pH caused by the hydroxides of calcium and alkalies in the cement. The high pH preserves a thin film of oxide over the metal and renders it passive to any further attack. Similarly, Josephson, Sillers, & Runner,^{3.11} in discussing blast-furnace slag, say:

. . . Slag is alkaline, and alkalinity generally prevents corrosion. Studies of steel removed from slag concrete after long years of service have shown that slag has no corrosive effect on steel.

Despite these reassuring statements it appears that some users of concrete still regard the presence of sulfides in aggregate or cement with suspicion, as is indicated by the restrictions on the total sulfur content of blast-furnace slag in both British and American standards. Consequently, most of the effort in the study of the chemical durability of synthetic aggregates has been devoted to this problem.

3.2.6 Organic Matter

Organic matter is said to retard the hardening of a concrete.^{3.4} Although present in considerable amounts in some dark clays and shales, at kiln temperatures with abundant air it should be completely oxidized and burned off. Nevertheless, some commercial aggregates are black, which suggests that organic matter is still present, and this suspicion is confirmed by the shape of some of the DTA curves discussed below. Other dark aggregates, however, show no strong DTA exotherms suggesting oxidizable substances, and in these cases the dark color may be due to magnetite or to manganese oxides.

3.3 Investigations on Aggregates

3.3.1 Petrographic Studies

Synthetic aggregates may be studied under the petrographic microscope either as crushed fragments in immersion oils or as thin sections mounted on microscope slides. The latter method gives by far the most information but suffers from the inherent disadvantage that it deals with only one piece of aggregate at a time, a piece which may or may not be representative.

As synthetic lightweight aggregates made from bloated clays consist of a sponge of glass, they require impregnation with a suitable isotropic material before a thin section can be ground. Satisfactory thin sections must be very thin; otherwise, much of the glass may appear dark and nearly opaque because of its content of extremely small bubbles.

The thin-section method permits the study of the aggregate, of concrete made from it, and of some of its

constituents, all in their normal relations. Between crossed polarizing prisms the glass and the bubbles are both dark but can be distinguished by the introduction of a little reflected light. Silt grains in the aggregate, being anisotropic, stand out sharply. Clay minerals in an aggregate particle, whose individual grains may be too small to see, commonly are oriented and can be detected by their "mass extinction."

3.3.2 Differential Thermal Analysis Investigations

Differential thermal analysis (DTA) curves were obtained on a number of aggregates, including some commercial ones as well as some produced in the TTI kiln. However, the curves showed such great variation between different runs on the same material that it seemed that the method could not be relied upon unless carried out under specific and closely controlled conditions. The nature and condition of the sample and of the comparison material, the rate of heating, and even idiosyncrasies of the particular furnace, all affected the shape of the curve obtained. Due to the complexity of this problem, DTA studies were carried out in a separate phase of this study and will be reported in a later technical report.

It did seem, however, that the DTA gave useful information about the presence of oxidizable constituents, such as organic matter and sulfides, in the sample, for these constituents produced exotherms considerably larger than the exotherms caused by differences in the method of operation. The exotherms caused by oxidation were particularly well shown when parallel runs were made in air and in helium on the same material.

3.3.3 X-Ray Diffraction Studies

As with the DTA, X-ray diffraction patterns were run on a few commercial aggregates as well as on several aggregates produced in the TTI kiln. In general the patterns, although difficult to interpret in detail, furnished useful information.

The bulk of the material in a synthetic aggregate, being noncrystalline glass, produced no peaks on an Xray pattern, but the included silt showed prominent peaks of quartz. Only one of the aggregates tested, purposely made at a low temperature $(1550^{\circ}F)$ in the TTI kiln, showed definite evidence of clay minerals on its X-ray graph, although three others showed suggestions of their presence. A series of kiln runs on the first material at different temperatures indicated that most of the clay minerals were destroyed by a temperature of 1870°F and that new minerals began to form around that temperature and were well defined at 1950°F. However, it was not established that these temperatures would be the same for material from different sources. A commercial aggregate, probably made from a clay rich in gypsum, showed strong lines of anhydrite, which was afterward confirmed by chemical tests.

Study of both the DTA and the X-ray patterns raised some doubt in the mind of this writer whether the X-ray alone is able to detect small percentages of clay minerals unless they are first fractionated away from the bulk of the material by a rather tedious procedure. However, if present in quantity, they apparently can be detected either by X-ray or DTA techniques. As mentioned earlier, this is a complex problem and is being studied in detail. As results are obtained they will be reported.

3.3.4 Pressure Cooker Experiments

Moore, Van Pelt, Scrivner, and Kunze^{3.1} outlined a procedure by which aggregates for base materials containing "underburned" clay minerals could be detected by their slaking after heating with water in a pressure cooker. The rehydration of the clay minerals produced an increase in the proportion of fine material in the sample and therefore a decrease in the percentage of the sample retained on a #40 sieve.

In applying this test to concrete aggregates it was found that some aggregates produced in the TTI kiln contained a great deal of fine dust, on the surface of the coarser particles, which had to be first washed off. On the other hand, an underburned concrete aggregate, known to contain clay minerals, when soaked in water acquired a clay-like coating or film on the particles which did not slake off and could not be removed without vigorous mechanical agitation during washing, even after pressure cooking in water at 15 psi. More research is needed on this matter before a widely applicable procedure can be worked out.

3.3.5 Lumps of Lime and Anhydrite

Two commercial aggregates, and several aggregates produced experimentally in the TTI kiln, contained white lumps, some of which were friable and some fairly hard. In every case these lumps were shown by qualitative tests to be either lime (CaO) or anhydrite (CaSO₄). Attempts were made to incorporate some of the white lumps in concrete to test their effect in causing popouts, but the lime lumps reacted so quickly with water that they did not survive the mixing of the concrete. Fragments of hard commercial quicklime were tried with the same result. However, some of the white lumps of anhydrite were successfully incorporated in concrete prisms, which were then autoclaved according to the procedure of ASTM C330-64T.^{3.2} One or two of the prisms showed swelling and distortion of the surface, indicating that the expansion had occurred.

3.3.6 Sulfide Studies

Tests for Decomposable Sulfides: A qualitative test for decomposable sulfides in synthetic aggregates has been described in Appendix 3 of a special report on a recommended synthetic aggregate classification system.^{3.12} This test depends upon the detection of the hydrogen sulfide gas evolved when the aggregate powder is warmed in dilute hydrochloric acid.

A procedure was devised for making this test quantitative by evolving the gas in a current of nitrogen (to prevent oxidation), collecting it in an ammoniacal zinc solution, then acidifying the solution and adding an excess of standard iodine to react with the H_2S . The excess of unreacted iodine is then titrated with standard thiosulfate. Considerable difficulty has been experienced in obtaining consistent results by this method when small amounts of sulfide are present, and precautions, such as tight apparatus, cooling during acidification, etc., must be taken to prevent the escape of any hydrogen sulfide.

Although one commercial aggregate and several aggregates made in the TTI kiln gave strong qualitative tests for sulfide, the amount of sulfide necessary to produce a strong test proved to be quite small. The largest amount found in any of the aggregates analyzed was 0.138% sulfide, equivalent to 0.38% FeS.

All raw clays in which pyrite or marcasite was observed under the microscope yielded aggregates giving positive tests for decomposable sulfide. Thus under the usual operating conditions of the rotary kiln complete oxidation throughout the aggregate particles apparently does not occur.

Experiments on Oxidation of Sulfides: Any deleterious effects in concrete caused by the oxidation of sulfides in the aggregate would be expected to manifest themselves only after the repeated wetting and drying resulting from exposure to the weather during several years. Therefore, in order to study the possible effects in the laboratory it was felt necessary not only to accelerate the wetting and drying, but also to work with higher concentrations of sulfide in order to intensify the effects. Accordingly, a synthetic aggregate was prepared by grinding together equal weights of a sulfidefree clay and Technical Grade FeS and firing this mixture at 2000°F in a muffle furnace. This resulted in a ceramic called 50% FeS ceramic in the experiments described below. Analyses showed that after firing it actually contained 39% FeS.

Experiments: Approximately 0.5 gram portions of the 50% FeS ceramic, ground to minus 100 mesh, were placed in 34 different solutions of measured pH ranging from pH 4.5 to pH 12.4. The solutions were made up from mixtures of chemicals having buffering behaviors so that their pH should remain fairly constant despite changes in the concentration of the solution.

All beakers containing the samples and solutions were allowed to stand open in the laboratory for $2\frac{1}{2}$ to 3 months, during which interval they evaporated to dryness seven times. Each time they were dry distilled water was added to restore their volume. The chemicals used were not volatile, but crystallized out and then went back into solution.

All beakers were examined visually and their appearance recorded at intervals of one week or less.

In most of the beakers rusty oxidation products $(Fe(OH)_3)$ were observed to be forming after the first week, and in some, notably those containing calcium hydroxide, within the first day. The solutions containing sodium hydroxide and sodium carbonate oxidized more slowly, and in those containing sodium hydroxide and sodium phosphate there is some doubt as to whether any oxidation occurred at all.

For comparison, technical grade FeS alone, both powdered and granular but without any admixture of clay, was allowed to stand in similar solutions, with very similar results.

Significant changes, mostly a decrease, in the pH of the solutions occurred during the experiment. Probably this was due chiefly to the sulfuric acid formed by oxidation and to a lesser extent by absorption of carbon dioxide from the air. The buffering action of the solutions kept the final pH of most of them between 7 and 9.

The experiments showed that FeS in a clay-ceramic aggregate, when exposed to repeated wetting and drying in air, will oxidize in a wide range of pH environments, even in one as alkaline as calcium hydroxide (pH 12.4).

Experiments on Effects of Sulfide on Corrosion of Steel: In view of the reports in the literature^{3.8, 3.10, 3.11}

of the absence of corrosion of reinforcing steel in concrete made with sulfide-bearing blast-furnace slag, another series of experiments was carried out in which the 50% sulfide ceramic was exposed to wetting and drying in solutions of measured pH in the presence of pieces of steel bar.

In these experiments 16 solutions of pH ranging from 7.0 to 12.5 were used, in each of which a weighed piece of steel bar was placed and 0.5 gm. of the 50% FeS ceramic added. A parallel set of 16 contained the steel but no sulfide-bearing material.

All beakers were allowed to stand in the laboratory for $6\frac{1}{2}$ weeks, during which time they evaporated to dryness five times and were rewetted with distilled water each time.

Coatings of iron hydroxide and sulfide appeared on the steel in nearly all the solutions to varying degrees, in some very promptly, in others very slowly. At the end of the experiment the bars were cleaned to bright metal and weighed again. In every instance the percentage loss in weight of the steel was greater in the solution containing the 50% FeS ceramic than in the corresponding solution without sulfide. It is of interest that while all the bars were attacked to some degree in the presence of sulfide, the bar in calcium hydroxide, pH 12.4, without sulfide showed corrosion within three days, but the bar in sodium hydroxide, pH 12.0, without sulfide remained bright throughout the duration of the experiment.

As in the other experiments with the FeS ceramic, drastic reductions in the pH of some of the solutions took place during the experiment, probably because of the formation of more sulfuric acid by oxidation than could be overcome by the buffering action of the solution.

3.4 Investigations on Concrete

3.4.1 Steaming Tests on Concrete Blocks Containing Sulfide Ceramic

As another means of simulating and accelerating possible weathering effects, small blocks of the 50% FeS ceramic were embedded in concrete prisms, which were then sawn into sections to expose the ceramic surface. These sections were then supported over heated water in such a way that they were subjected daily to a mixture of steam and air, and that the condensate dripping from them could be collected in a glass dish. They were also weighed daily before the water was heated. The expectation was that the concrete surrounding the ceramic blocks would show signs of disintegration and that the condensate would become acid.

After 20 cycles of heating, the blocks and the condensate were examined. Contrary to expectations, the 50% FeS ceramic showed no change other than a few minute rusty spots. The condensate was alkaline, pH 9.8, and etched the glass plate. It reacted for carbonate and sulfate, but both of these could have come from the cement. Evidently any sulfuric acid extracted from the ceramic was more than neutralized by the alkalis extracted from the cement.

Control blocks of concrete containing no sulfide, subjected to the same treatment, gave a condensate with similar reactions, except that a trace of iron was found in the condensate from the ceramic and not in that from the control blocks.

Weights of all the blocks first showed a gain and then fluctuated erratically during the experiment, probably because of changing moisture content.

3.4.2 Investigation of Changes in pH of Concrete with Time

Because of the possible influence of the pH of concrete on the oxidation of sulfides in the aggregate, and because the absorption of CO_2 from the air and from rain water is known to lower the pH of at least the surface of concrete structures, a small-scale investigation was made of the extent of these changes on concrete of different ages.

Concrete from the following sources was studied:

- Lot #1: Bryan Field, apron along hangars. Placed in 1942. Low spot, overgrown with about 1 inch of soil and grass. Probably wet much of the time.
- Lot #2: Bryan Field, taxiway. Placed in 1942. About 3 inches above ground surface. Well drained and probably dry most of the time.
- Lot #3: Kyle Field Stadium, College Station. Placed in 1927. Probably seldom if ever exposed to rain.

Samples were selected from the above three lots, ground, shaken with previously boiled distilled water, and let stand 40 minutes. The pH of the water above each sample was then determined, with the following results.

Sample No.	Lot No.	Year Placed	Distance from Other Nearest Identifying Surface Data	pH
1	2	1942	At least 3/4"	11.35
2	2	1942	Slight Yellowed areas	10.25
· 3	2	1942	Interior pieces	11.35
4	3	1927	$\begin{array}{c} \text{Dark gray} \\ \text{Less than } \frac{1}{2} \\ \text{Very white} \end{array}$	10.5
5	3	1927	Unknown areas	9.9
Ğ	1	1942	At least 1"	11.3
7	ī	1942	Less than 1/4"	10.05

It may be concluded that exposure to air and water does lower the pH of concrete as the free hydroxides in it absorb CO_2 and change to carbonates, and that this carbonation increases with time.

The depth of penetration of the carbonation appears to be influenced by the porosity of the concrete and whether it remains dry, is alternately wet and dry, or remains water-soaked.

3.5 Conclusions and Recommendations

3.5.1 Clay Minerals in "Underburned" Aggregates

The pressure cooker test for detecting clay minerals in underburned aggregates has appeal because of its simplicity. Research being conducted on Phase II of this study is showing that this test is promising for base materials where relatively large amounts of clay minerals may exist. However, the ability to detect amounts with this test that may be deleterious in concrete aggregates is hampered by the existence of dust on the rotarykiln produced aggregate particles. This dust is difficult to remove by dry sieving.

On the other hand, washing in water may cause the rehydration of the clay minerals to an adherent film that can be dispersed only by mechanical agitation. The one underburned concrete aggregate tested, shown by X-ray to contain clay minerals, lost nearly as much fine material by prolonged rubbing under cold water as it did by pressure cooking and rubbing. Cooking without agitation caused little loss. It seems that for concrete aggregates the agitation is at least as important as the temperature and pressure of the pressure cooker test.

A means of detecting small amounts of clay minerals in concrete aggregates needs to be developed.

3.5.2 Lumps of Lime and Anhydrite

As pointed out above, lumps of lime, unless very large, react so rapidly with water that they are unlikely to pass through the concrete mixer without disintegrating. Lumps of anhydrite react much more slowly and are more likely to cause subsequent popouts. But as lime and anhydrite cannot readily be distinguished by inspection, the following examination is recommended:

Spread a representative sample of at least one pound (or 500 grams) of the aggregate in a thin layer on a sheet of paper or cloth or on a table top, preferably black or dark colored. Examine the aggregate visually for white lumps $\frac{1}{8}$ inch or more in diameter. If white lumps are present the test for popout materials (autoclave test) should be run in accordance with ASTM C330.^{3.2}

3.5.3 Sulfides

Experiments have confirmed the expectable chemical behavior of iron sulfide and have shown that when included in a clay-ceramic aggregate it will oxidize in a wide range of pH environments, and that its oxidation will accelerate the corrosion of steel with which it is in contact. This is in accord with the restrictions cited by Lea^{3.4} as in effect in Great Britain on the use of coal ashes, and with both the British and the ASTM specifications on the use of slag containing sulfides.

How then are these observations to be reconciled with the conclusions reached by Everett & Gutt,^{3.8} Josephson, Sillers and Runner,^{3.11} and others, that blast-furnace slag containing sulfides gives satisfactory performance as aggregate in concrete and has no corrosive effect on the reinforcing steel?

Perhaps the conclusions of all the investigators, inconsistent as they are, are correct as far as they go. The explanation may be that although the high pH of concrete does not prevent the oxidation of the sulfide, the sulfuric acid formed is immediately neutralized by the excess alkali in the concrete, and thus is prevented from either disintegrating the concrete or corroding the steel.

If this is the explanation, then sulfide in an aggregate will do no harm provided that there is at all times sufficient excess alkali in the immediately surrounding cement paste to neutralize all the sulfuric acid formed. Then perhaps the placing of an upper limit (as in ASTM and British standards) on the permissible content of sulfur compounds is the desirable solution of the problem.

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It is obvious that the less the permeability of the concrete to air and water the less the carbonation and oxidation that can occur. It should be remembered, however, that cracks, whether caused by shrinkage or by stress, offer ready access of air and water to the interior of the concrete mass, and that they remain damp much longer than the surface. Thus they offer ideal conditions for the local lowering of the pH and for the oxidation of any sulfide in the aggregate exposed along them, with accompanying attack on the concrete and on the exposed reinforcing steel.

Therefore, it is suggested that the qualitative test for decomposable sulfides, described in a TTI Special Report,^{3.12} be required on all synthetic aggregate considered for use on concrete structures, so that the engineer may be aware of the possibilities of the material with which he has to deal.

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by

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4.1 Introduction

4.1.1 General

Durability of concrete, as currently defined by ACI committee 201,^{4.1*} is:

For present purposes, durability of concrete is defined as its resistance to deteriorating influences which may through inadvertence or ignorance reside in the concrete itself, or are inherent in the environment to which it is exposed.

It follows then that the purpose of a durability test on concrete and concrete making materials is to separate them into classes, grades or groups in the order of their ability to resist some type of deterioration. In this report the type of deterioration being considered is wear of concrete surfaces due to traffic passing over them. Distress usually takes the form of rutting in the wheel path with subsequent loss of crown and surface drainage. Since little, if any, long-time performance data are available on concrete pavements made with synthetic aggregates,** it is desirable to develop some procedure for evaluation of these materials in terms of their resistance to rutting in the wheel path before it is used extensively in highway pavement construction.

4.1.2 Background

Researchers have realized the importance of the resistance of concrete to wear for many years. This is evidenced by the early development of testing procedures for measuring the resistance of aggregates and concrete to abrasion of various types. Over the years many different approaches have been made to the problem of abrasion or wear testing. Some of the methods will be discussed in the following paragraphs.

Several revolving disc methods have been developed in which prepared cores of aggregate material are pressed against a wheel in motion. Other methods provide for the specimen to be in motion, and some require both to move. The abrasive action is usually provided by a silica sand, quartz, or grit which is supplied to the wheel and specimen. Samples of the aggregate are normally cylindrical and may become quite expensive or time consuming to prepare from conventional aggregates used in highway construction. Some methods of the type described above are the Shuman and Tucker Machine,^{4.3} Kessler Machine,^{4.4} and Master Builders Rotating Disc Machine.^{4.5} The Spellman and Ames Vibrating Machine is another means of measuring abrasion resistance.^{4.6} In this test a four-inch diameter sample is placed in a cylinder. Steel balls and water are added and the machine vibrates vertically.

The Davis Steel Ball Test consists of a one-thousand pound load applied to a 12 in. diameter, rubber-covered, rotating head which is in contact with the specimen surface.^{4.7} Both the specimen and the steel balls are submerged in circulating water to remove material eroded during the test. It has been found that this test requires modifications to produce appreciable abrasive loss.

Another type of resistance may be studied with the Dressing Wheel Abrasion Test, where 32 rotating dressing wheels under an 18-pound load supply the abrasive forces.^{4.7} The driving head is rotated for a specified number of revolutions for each test. Abrasive material is applied to the wheels at a constant rate throughout the test.

A stream of steel shot provides the abrasive forces in the Ruemelin Shot Blast Test.^{4.7} Two-thousand, No. 20, broken steel shot are ejected through a ¹/₄-in. nozzle under 90 psi air pressure against a specimen surface four in. away. The shot are discharged at the rate of 500 gm./min. This treatment is applied to eight different locations on each specimen. There are several other tests of this nature.

The American Society for Testing and Materials has accepted three abrasion tests for concrete and concrete aggregates. The Deval Machine may be used for graded coarse aggregates or rock. The Los Angeles Machine is used for graded coarse aggregates. These two tests involve placing a sample and a number of steel balls in a metal cylinder and rotating the cylinder. The procedures are described fully in the ASTM Standards.^{4.8} The third test accepted by the ASTM involves the resistance of a concrete surface to the abrasive force of a stream of high velocity sand particles. In this test eight areas of one square inch each are subjected to the sand blast for one minute, at the rate of 600 gm./min.

There are many means of eroding away the surface of materials in service and this has created many different abrasion tests. Each test is believed to simulate some type of service wear. It is up to the researcher to select the test which seems most appropriate for the situation being investigated.

4.1.3 Purpose

This investigation was planned and conducted as a pilot study with somewhat limited objectives. The purpose of this study was to develop preliminary information on the abrasion resistance characteristics of concrete.

A step-by-step approach was followed throughout this investigation. It was necessary to select a test method to employ in the investigation and determine whether

^{*}References for this chapter are contained in section 4.5. **For purposes of this report synthetic aggregates are defined as structural quality aggregates produced by fusing raw shales and clays in a rotary kiln under intense heat into predominantly amorphous silicates. These aggregates can be broadly divided into two categories: (a) fused and bloated aggregates (less than 55 pcf dry unit weight), generally termed structural lightweight aggregates and (b) fused but not bloated aggregates (greater than 55 pcf dry unit weight).⁴⁻²

or not the test was repeatable on the concrete being studied. Next, tests were set up to determine the effects of aggregate, cement factor, and moisture conditions. Once these aspects of the procedure had been tentatively confirmed the next step was to obtain specimens of concrete in service which exhibited satisfactory and unsatisfactory behavior in terms of traffic wear and compare test results with laboratory concrete specimens.

4.1.4 Scope and Limitations

This pilot study was conducted with the above specific objectives in mind. From the start it was intended to answer the following questions:

(a) Does the selected test method yield repeatable results?

(b) Is there a significant difference among the abrasion coefficients of various synthetic aggregates?

(c) Is there a significant difference between test values for synthetic aggregate concrete and a natural gravel concrete?

(d) Does the cement factor of the concrete mix significantly affect the test results?

(e) Can the test be used to distinguish between a concrete that has performed satisfactorily and one that has not?

(f) How do the abrasion coefficients of synthetic aggregate concrete specimens compare with specimens of in-service concrete?

The results presented later in this report are intended to provide answers to these questions. They should be interpreted as preliminary results until confirmed by more extensive testing.

4.2 Experimental Program

4.2.1 General

The test selected was the sand blast abrasion test, ASTM C418-67T, "Method of Test for Abrasion Resistance of Concrete (Tentative)."^{4.8} This test was the most advantageous for several reasons:

(a) Equipment was readily available.

(b) Adequate supply of specimens was available.

(c) Only a short time was required for testing.

(d) The test was relatively inexpensive and easy to perform.

(e) The test has been recognized by the ASTM.

4.2.2 Sand Blast Abrasion Test (ASTM C418-67T)

The test consists of sandblasting eight areas of one square inch each on the surface of a concrete specimen. The abrasive used is natural silica sand from Ottawa, Illinois, graded to pass a No. 20 sieve and be retained on a No. 30 sieve. The abrasive is discharged from a $\frac{1}{4}$ in. nozzle at a rate of 600 gm./min. under 60 psi air pressure. The specimen weight loss is determined by weighing before and after the test in a 24-hour saturatedsurface-dry (SSD) condition. The specimen is saturated



Specimen Number Figure 4-1. Reproducibility of concrete abrasion test.

24 hours prior to testing, and bulk specific gravity is determined by weighing in water and in air. The specimen is again soaked for two hours after testing before weighing to determine the weight loss. The abrasion coefficient is calculated from bulk specific gravity of the concrete and weight loss. It is expressed as $cm.^3/cm.^2$ (volume of material abraded per unit surface area sand-blasted).

Tests were conducted to determine the reliability of the test method on laboratory concrete made with one of the synthetic aggregates under study. The abrasion loss was computed for two tests on each of five specimens made with aggregate R. Tests were run both with and without soaking the specimens 24 hours prior to testing. This was done in order to evaluate the effect of moisture condition on the test results. The specimens for these tests were cast from one batch of concrete with a cement factor of five sacks per cubic yard. The concrete was approximately 28 days old when tested. The specimens were cast as $3 \times 3 \times 16$ in. prisms which were sawn in half to form $1.5 \times 3 \times 16$ in. specimens for testing. The tests were run on the sawn surface in order to amplify the effect of the aggregate used.

The results of the tests are shown in Figure 4-1. The coefficient of variation indicates slightly more consistent results on specimens that are soaked prior to testing (13%) for the dry tests and 8.7% for the soaked tests). It is evident from these results that the test is as reliable as most tests used in the evaluation of properties of concrete. The moisture condition does have an effect on the test results and should be controlled as ASTM C418-67T provides.

4.2.3 Specimens Selected for Testing

Laboratory Test Specimens. The specimens used for the laboratory tests were about three years old when tested. The specimens were $1.5 \times 4 \times 8$ in. prisms with the sawn surface used in order to amplify the effect of the various aggregates. Four commercial synthetic aggregates (R, C, D, E) and one commercial natural gravel aggregate (H) were used. Three specimens having cement factors of four, five, and six and one-half sacks per cubic yard were used for each aggregate involved, for a total of 15 specimens.

In the first series of tests one specimen of each aggregate type was tested first in a 24-hour soaked condition and then in a dry condition. These tests were run on dry specimens to see how the moisture condition affected the relationships between the various aggregates used. The next series of tests were intended to determine how the various cement factors affected the test results. All fifteen specimens were used with one test on each specimen. These tests were all conducted with the specimens in a dry condition. Test results are presented and discussed in Sections 4.3.2 and 4.3.3.

Field Test Specimens. In selecting sites for obtaining pavement cores it was desired to find areas which exhibited both satisfactory and unsatisfactory performance. Three locations were selected for investigation. Location 1 was in an area which showed excessive wear in the wheel path. Four specimens were taken six to nine inches from the pavement edge and four were taken in the outer wheel path. The specimens from the pavement edge still had the original surface while those in the wheel path had been worn off. Location 1 will be



Figure 4-2. Equipment used in obtaining pavement cores.

referred to as the worn area. Location 2 was in an area where the surface was not worn in the wheel path, approximately 30 feet from the worn area (location 1) in the same traffic lane. As before, four specimens were taken at the pavement edge outside of the wheel path and four were taken in the outer wheel path. The original surface was intact on all specimens in this area. Location 2 will be referred to as the unworn area. Location 3 was in a section of pavement several miles from the first two locations. At this location the surface was only slightly worn in the wheel path. Three specimens were taken in the wheel path and three at the pavement edge. Location 3 will be referred to as the slightly worn area. Figure 4.2 shows the equipment used in obtaining the specimens of concrete pavement. All specimens taken were six to ten inches in length and four inches in diameter. Although several cores were separated at the level of the reinforcement and some contained pieces of steel, all core surfaces were preserved in their service condition. The specimens were all made with a limestone aggregate.

Four specimens from each location were selected for testing, two from the pavement edge and two from the wheel path. Due to the limited amount of surface area available on the specimens four areas of one square TABLE 4-1. PORTABLE SKID TESTER RESULTS*

	Location	$\begin{array}{c} \operatorname{Location} \\ 2 \end{array}$	Location 3	
Edge of Pavement	worn	unworn	slightly worn	
dry wet	$\begin{array}{c}103\\47\end{array}$	86 50	$\begin{array}{c} 105\\ 45\end{array}$	
Temperature of Wet Surface	28.5°C	28°C	34.5°C	
In Wheel Path			· · ·	
wet dry	$\begin{array}{c} 30\\ 103 \end{array}$	45 108	41 105	
Temperature of Wet Surface	28.5°C	29.5°C	33°C	

*Values measured are BPN, British Portable (Tester) Number

inch each were tested rather than eight such areas as required by the test method (ASTM C418-67T). All other aspects of the testing met the requirements of the test procedure. The exposed wearing surface was tested on all pavement cores.

4.2.4 Portable Skid Tester

The skid resistance of the concrete pavement in the areas discussed in the previous section was measured at the time the cores were taken. The British Portable Skid Tester shown in operation in Figure 4-3 was used for the measurements.^{4.9} This information was obtained in order to develop a more complete description of the condition of the pavement surface in the areas selected for sampling.* Results of the tests are presented in Table

*These data should not be interpreted to indicate a relationship between wear and surface slipperiness which are not the same. The test does provide an easily obtainable measure of one property of the surface and may or may not be related to abrasion resistance.



Figure 4-3. British portable skid tester.

4-1. The values shown are BPN, British Portable (Tester) Number.

4.2.5 Strength Tests on Pavement Cores

In order to more fully evaluate the properties of the concrete in the various locations, the dynamic modulus of elasticity was computed from the fundamental transverse frequency measured according to the ASTM Method C215-60,4.8 and the uncorrected slope of the static load-deformation curve was determined by means of an Instron testing machine with a machine plot of the loaddeformation curve. The load deformation curve includes deflection of the machine as well as the specimen and therefore should not be interpreted as an absolute value of the static modulus. Compressive strength of the core specimens was measured according to the ASTM Method C42-64^{4.8} with the exclusion of the soaking period. All specimens were treated similarly, and relative values are the important consideration in this case. The results of the strength tests are shown in Table 4-2.

Specimen Length in.	Position Core Taken From	Location*	Dynamic Modulus Elasticity psi $\times 10^{-6}$	Uncorrected Slope of Static Load Defor- mation Curve	Compressive Strength psi
· 6**	edge of pavement	1	3.88	0.158	3790
6	wheel path	1	3.94	0.162	3690
8	edge of pavement	1	3.88	0.215	3010
8	wheel path	1	3.73	0.205	2880
6	edge of pavement	2 .	4.18	0.166	4560
, 6	wheel path	2	4.38	0.163	3700
8	edge of pavement	- 2	4.28	0.211	4010
8	wheel path	2	3.75	0.210	3490

TABLE 4-2. RESULTS OF STRENGTH TESTS ON PAVEMENT CORES

*Location 1 is worn area and location 2 is unworn area.

**Strengths of 6-in. cylinders were corrected for length to diameter ratio. Due to the presence of steel in some specimens and the use of the top two inches for sandblasting on others, only four specimens were available from each location to be used in strength determinations. One six-in. and one eight-in. specimen were used from the wheel path and from the pavement edge at each location.

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4.3 Discussion of Results

4.3.1 Effect of Aggregate

From tests conducted on concrete made with the five different aggregates being studied it was apparent that the abrasion resistance of lightweight aggregate concrete was significantly less than that for a good regular weight gravel aggregate concrete, when the coarse aggregate was exposed. The results of specimens tested dry and in a 24-hour soaked condition are shown in Figure 4-4. The test results clearly separate the synthetic aggregate concretes from the natural gravel concrete. This observation leads to the conclusion that synthetic aggregate concrete may abrade more easily than natural gravel concrete, if the coarse aggregate is exposed.

Concerning the relative difference between various synthetic aggregates, the results are not as clear. Tentatively, it can be said that the aggregate D concrete may be significantly less resistant to abrasion than aggregate R concrete, based on the soaked condition. Beyond this, no conclusions can be drawn.

4.3.3 Effect of Cement Factor

In order to determine how the cement factor of concrete affects abrasion resistance, specimens of the five aggregates under study having cement factors of 4, 5, and 6.5 were tested. Examination of the test specimens revealed that the abrasion of the synthetic aggregate concrete was primarily due to the erosion of coarse aggregate, while for the natural gravel concrete the mortar eroded more readily than the coarse aggregate (Figure 4-5). This would mean that provided the aggregate is exposed, an increase in the cement factor, or strength of the mortar, should not significantly affect the abrasion coefficient of the synthetic aggregate concrete. Test results in Figure 4-6 tend to verify this statement (each value shown represents one test on one specimen).

4.3.4 Concrete Pavement Cores

Four cores of limestone aggregate concrete from each area sampled were tested for abrasion loss. Two were taken from the pavement edge and two in the wheel path at each location. Results are shown in Figure 4-7. The test results on cores from the worn area revealed a significant difference in abrasion coefficient between the pavement edge and the wheel path. The surface in the wheel path had been worn away and the coarse aggregate exposed, while at the pavement edge the original finished



Figure 4-4. Effect of aggregate and moisture condition on concrete abrasion coefficient.



Aggregate H Specimen



Aggregate C Specimen

Figure 4-5. Abrasion test specimens of aggregates C and H.

surface was present. The difference then indicated that the top finished mortar layer abraded much easier than the limestone coarse aggregate. In the unworn area there was no significant difference in abrasion coefficient between the wheel path and pavement edge. This is an indication that the concrete in this area is of uniform quality.

The most significant result of these tests was in comparing the results of specimens from the worn area to those of specimens from the unworn area. There was a very significant difference between abrasion coefficient at the pavement edge for the two locations. The value in the worn area was $0.24 \text{ cm.}^3/\text{cm.}^2$ and in the unworn area was $0.11 \text{ cm.}^3/\text{cm.}^2$.

4.3.5 Comparison of Synthetic Aggregate Concrete and Pavement Cores

The results obtained on synthetic aggregate concrete in the laboratory are also presented in Figure 4-7 for comparison. All of the synthetic aggregate concrete specimens had losses nearly as large or larger than the concrete pavement worn area. From these limited tests



Aggregate Used in Concrete

Figure 4-6. Effect of cement factor on concrete abrasion coefficient.

it can be concluded that if synthetic aggregate particles are exposed to wear in a concrete surface they will abrade appreciably. Since it is not known how extensively the loss would be or how it would affect skid resistance, no conclusions can be made as to the expected service life of synthetic aggregate concrete pavement.

The cores from location 3, where the surface was slightly worn, were tested and results are also presented in Fig. 4-7. There was not a significant difference between cores from the pavement edge and those from the wheel path.

In order to extend the meaning of the results obtained on specimens from the worn and unworn areas, some strength characteristics were determined as presented in Section 4.2.5 of this report. The significance of the information presented is that it appears that the concrete at both locations was of adequate strength. The values shown do indicate differences; however, they are within the limits of normal data scatter for the methods used. These results indicate that, in the worn area, only the surface layer was of inferior quality. Whatever the cause for the failure of the surface to resist wear, it can be tentatively concluded that this test (ASTM C418-67T) can distinguish whether or not a surface will resist traffic wear.

4.4 Conclusions and Recommendations

4.4.1 Conclusions

The results presented were based on a limited number of tests intended to be used in developing preliminary information on the abrasion resistance characteristics of concrete. On the basis of the results presented in this report the following tentative conclusions can be made:

(a) The abrasion coefficient is repeatable for concrete specimens of the type tested.

(b) Aggregate D concrete may be significantly less durable than aggregate R concrete, based on the soaked condition. Beyond this, no conclusions can be drawn.

(c) When the coarse aggregate is exposed, synthetic aggregate concrete abrades significantly more than natural gravel concrete, due to the low abrasion resistance of the synthetic coarse aggregate particles.

(d) When the coarse lightweight aggregate is exposed, the cement factor apparently does not significantly influence test results.

(e) On the basis of the results obtained from the three locations of existing concrete pavement, it appears that the test can be used to distinguish between a concrete that has performed satisfactorily and one that has not.

(f) The abrasion losses of laboratory specimens of synthetic aggregate concrete are high when compared to those of siliceous aggregate concrete and in-service limestone aggregate concrete specimens.

(g) The amount of moisture present in the specimen at time of test significantly affects test results and should be controlled.

(h) The test may be of value in evaluating construction practices, such as surface finishing.

(i) The test results presented herein indicate that the test method is worth further consideration for use in evaluating abrasion resistance characteristics of concrete.



Figure 4-7. Results of abrasion tests on concrete pavement cores.

4.4.2 Recommendations

It is recommended that investigation of the abrasion resistance characteristics of synthetic aggregate concrete be continued.

4.5 References for Chapter 4

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