

**EFFECT OF SYNTHETIC AGGREGATE THERMAL
TRANSFORMATION ON PERFORMANCE OF CONCRETE**

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Research Report 81-10

Synthetic Aggregate Research

Research Study 2-8-65-81

sponsored by

The Texas Highway Department
in cooperation with
U. S. Department of Transportation
Federal Highway Administration
Bureau of Public Roads

October, 1969

Texas Transportation Institute
Texas A&M University
College Station, Texas

PREFACE

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

This is the tenth 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 the U. S. Bureau of Public Roads. The first nine 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.

"Performance Studies of Synthetic Aggregate Concrete," by C. E. Buth, H. R. Blank, and R. G. McKeen, *Research Report 81-6*, Texas Transportation Institute, March, 1969.

"Fundamental Factors Involved in the Use of Synthetic Aggregate Portland Cement Concrete," by W. B. Ledbetter, C. E. Sandstedt and A. H. Meyer, *Research Report 81-7*, Texas Transportation Institute, November, 1969.

"A Sandblast Abrasion Test for Synthetic Aggregate Evaluation," by James T. Houston and W. B. Ledbetter, *Research Report 81-8*, Texas Transportation Institute, October, 1969.

"Studies of the Thermal Transformation of Synthetic Aggregates Produced in a Rotary Kiln," by James T. Houston, H. R. Blank, and G. W. Kunze, *Research Report 81-9*, Texas Transportation Institute, November, 1969.

In addition, a special report has been published under this research study. The report is:

"A Recommended Synthetic Coarse Aggregate Classification System (Revised August, 1969)," by W. B. Ledbetter, B. M. Callaway, W. M. Moore, and Eugene Buth, *Special Report*, Texas Transportation Institute, August, 1969.

The authors wish to thank all members of the Institute who assisted in this research.

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 and Research Area Representative; Mr. H. A. Sandberg, Jr., Materials and Tests Division Representative; and Mr. Louis White, 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

The investigation involved twelve different synthetic aggregates produced in the TTI rotary kiln, one commercially produced synthetic lightweight aggregate, and one regular weight aggregate. Each aggregate was used in concrete which was subjected to a variety of physical durability investigations including autoclave expansion, long-time reactivity, freeze-thaw, and shrinkage. From the results of the study it was found that (a) incompletely burned synthetic aggregates resulted in poor performance in the standard ASTM C-290 freeze-thaw test for concrete, (b) potentially destructive autoclave expansion occurred in some concretes, (c) long-time reactivity curing produced no significant volume changes following initial autoclave treatment, and (d) shrinkage studies showed no relationship to degree of transformation of the synthetic aggregates.

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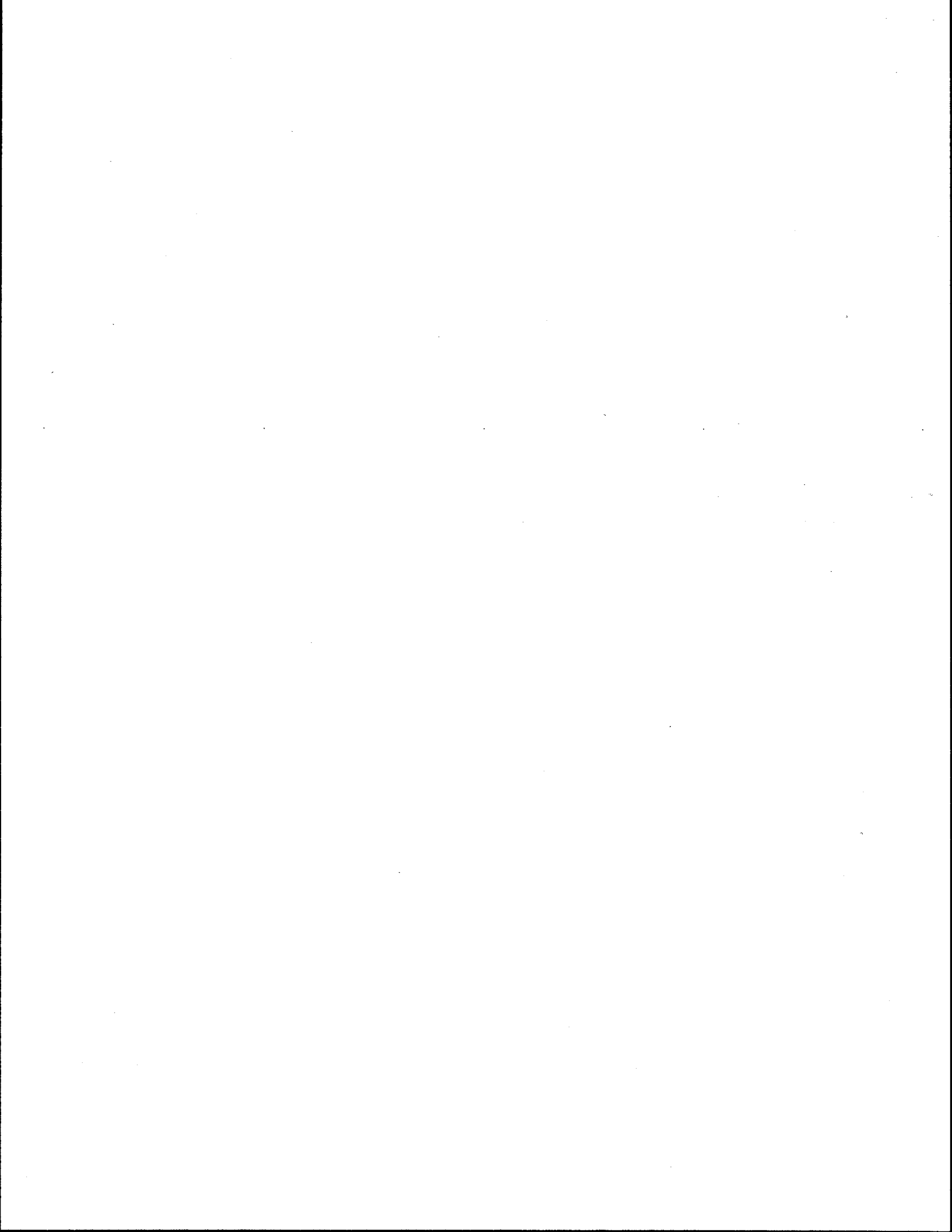
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1. Introduction

1.1 Purpose

The purpose of this report is to present the findings concerning the physical durability of portland cement concrete as a function of the degree of thermal transformation of selected synthetic aggregates used in this concrete. This is a continuation of the phase of the investigation reported in *Research Report 81-6 (1)*.*

1.2 Scope

The phase of the over-all research program reported herein involves twelve different synthetic aggregates produced in the TTI rotary kiln, one commercially produced synthetic lightweight aggregate, and one regular weight aggregate, or a total of fourteen different aggregates. Each aggregate was used in concrete which was subjected to a variety of physical durability investigations. Results of these studies were analyzed and are reported herein.

1.3 Conclusions

Based on the results of the investigation reported herein, the following conclusions can be drawn:

1. A general but tentative trend was found for the concrete strength as a function of aggregate porosity. For concretes having similar cement factors, the higher the aggregate porosity, the lower the concrete compressive strength.

2. An autoclave expansion test was used to measure a concrete's tendency to harmfully expand due to incomplete thermal transformation of the aggregate. Destructive expansions attributed to certain aggregate components were detected by this autoclave test on concrete specimens. However, the mechanism of expansion could not be definitely determined.

*Numbers in parentheses refer to references contained in Section 4.7.

3. Longtime reactivity curing tests of approximately one year produced no additional concrete volume changes of significance after initial autoclave treatment.

4. Incompletely burned aggregates resulted in poor performance in the standard ASTM C-290 freeze-thaw test for concrete.

5. The shrinkage behavior of synthetic aggregate concrete was not a reliable indicator of incomplete thermal transformation of the aggregate.

1.4 Recommendations

1. In freeze-thaw durability studies the degree of saturation of aggregates just prior to batching should be closely controlled for meaningful results. The influence of degree of saturation is worthy of continued study.

2. It is suggested that high alkali cements be used in reactivity studies in order to accelerate any alkali related reactions.

3. In the absence of proven field performance, an aggregate should not be accepted for structural use if any autoclaved concrete specimen shows expansive strain in excess of 1500×10^{-6} in./in.

4. The subject of hydration shrinkage should be explored to determine the parameters influencing shrinkage of synthetic aggregate concrete and how much shrinkage is, in fact, detrimental to the performance of the concrete.

1.5 Implementation Statement

Based on the results of this investigation, it is suggested that recommendation 3 of Section 1.4 be evaluated by the Materials and Tests Division of the Department and, if acceptable, placed in tentative special provisions to the specifications as a *source* qualification test.

The above statement represents the combined opinions of the study contact representative and the authors and should not be construed as departmental policy.

2. Background

2.1 Aggregate Rehydration

Perhaps the most frequently asked question with respect to the nature of synthetic aggregates made from clays or shales is: Under what processing conditions does a burned aggregate cease to be characterized by the behavior of the raw material and begin to exhibit the desirable properties of strength and durability? To answer this question it is necessary to consider the changes which occur in the clays as they are heated.

It is generally recognized that water is connected with the physical structure of clays in several ways. This water is classified by the manner in which it is bonded to the clay structure and the ease with which it is driven off by heating (2). The water which is readily removed by heating at temperatures of 100 to 150°C is of secondary importance and will not be considered here.

On the other hand, the type of water referred to as hydroxyl water is an important factor in the transformation process.

Hydroxyl water (O-H) is an actual structural component of the atomic lattice of the clay and consists of an oxygen and a hydrogen atom bonded to other atoms of the various clay minerals. The thermal energy required to remove the hydroxyl water from the clay structure, that is, the characteristic dehydroxylation temperature as indicated by endothermic DTA peaks, varies with the nature of the clay, but is generally in excess of 400°C. It has become somewhat convenient to refer to the first high temperature endotherm ($\approx 500^{\circ}$ C) as the reaction at which the clay mineral is destroyed and after which an inert stable material exists. Since the great majority of the clays have characteristic endothermic peaks at temperatures below 800°C (1422°F),

it is tempting to over-simplify the problem by assuming that any clay heated in excess of, say, 1500°F will be an inert, stable aggregate suitable for use in concrete.

A more thorough analysis of this behavior reveals several interesting facts necessary for a better understanding of the dehydroxylation process. First, the so-called characteristic dehydroxylation temperature by no means represents the maximum temperature required to completely dehydrate the various clays (2, 3)*. Second, although the dehydroxylation process produces a progressive disruption of the lattice structure, some degree of structural order can persist even beyond the characteristic dehydroxylation temperatures (2, 3, 5). Finally, even though the dehydroxylation temperature may be reached and held for as long as four hours, the rehydration of the clay mineral is possible under certain conditions (2, 6).

A logical question at this point concerns the physical stability of a clay that has undergone some degree of rehydration. Very little if any research of this nature has been conducted in determining the physical properties resulting from the rehydration of rotary-kiln-produced aggregates. Although the ceramists have conducted a limited amount of reliable research on this subject, conclusions of any detail cannot be given at this time. However, it seems reasonable to state that, in general, rehydration of a burned clay will result in some degree of expansion and perhaps some softening of the aggregate particles. The magnitude of this effect, of course, will depend upon the extent to which the dehydration had taken place during the heating process. In addition, it is probable that a specific burning temperature exists beyond which little or no rehydration will occur, and this temperature varies depending upon the clay minerals present in the raw material. Unfortunately, the magnitudes of these temperatures have not been established and much work remains to be done in this area.

On the basis of the preceding discussion, a test for the identification of aggregates susceptible to rehydration seems to be desirable. In choosing a method for the testing of this behavior, the advantage of producing an accelerated rehydration of the test specimen is obvious. A technique which has previously been used for this purpose involves a steaming treatment at above atmospheric pressure (3, 6, 7). This type of treatment is also applicable to many other types of reactions which may occur in aggregate-cement combinations as will be discussed later in this section.

In devising a method for the detection of the rehydration of clay minerals, the accelerating technique discussed in the preceding paragraph played an integral role. However, a similar analysis must be considered for other types of detrimental reactions which may occur with the use of synthetic aggregates in concrete. In this way, a more simplified test procedure may be developed which will apply to a wide variety of undesirable reactions involving the aggregate and its environment.

2.2 Concrete Reactivity

The need for a definitive reactivity evaluation procedure for structural aggregates is especially true for the

*See also the EGA data presented in Reference 4 and in *Research Report 81-9*.

thermally processed aggregates (1, 4). New deposits and/or processing plants are continually resulting in new materials on the market. Since field performance records do not exist in these cases, some orderly and dependable method is needed for the quality classification of these materials. Even with products having proven service records, systematic and continuing evaluation procedures should be used. This is advisable since even a minor modification in the processing technique can result in a significant change in the quality of the synthetic aggregate product.

Alkali-Silica Reactivity—Evidence of an expansive reaction involving certain aggregates used in concrete was noted in the states of Nebraska and Kansas around 1930. Since that time, considerable research has been conducted to identify the exact cause of this disruptive reaction (8, 9, 10, 11). As a result, it is generally concluded that the cause of these concrete failures was a chemical reaction between the alkalis in cement and certain forms of silica in a limited variety of rocks. Besides siliceous rocks such as opaline cherts, chalcidonic cherts and siliceous limestones, a number of volcanic rocks have shown some degree of reactivity (10, 11). Examples of the reactive components of the volcanic rocks are volcanic glass, devitrified glass and tridymite (10).

With respect to synthetic aggregates, certain physical similarities exist between volcanic rocks and thermally processed clay minerals. That is, they both consist of amorphous and crystalline siliceous materials which have been subjected to high temperature environments. This similarity does not automatically place synthetic aggregates into the category of reactive materials, although it does furnish impetus to specify some method of testing which will preclude the use of any aggregate which may be reactive.

In selecting a reactivity test procedure, it is helpful to examine the techniques developed for the study of naturally occurring reactive rock materials. In the early research efforts on this subject, the methods most often used as an indicator of reactivity involved the practice of subjecting concrete prisms to warm, moist curing conditions (10, 12). In these techniques the increase in the length of the prism and their physical appearance were used as an indication of reactivity. As a result of these efforts, in 1950 a tentative method of test for alkali-aggregate reactivity was issued as ASTM C227 (13). This test, referred to as the mortar bar method, essentially involves placing concrete prisms in a humid atmosphere at a temperature of approximately 100°F and taking length measurements at periodic intervals.

Alkali-dolomitic Carbonate Reactivity—In a recent study by Hadley (14), certain of the dolomitic rocks have been shown to be reactive in concrete as indicated by expansive behavior or the presence of reaction products. Those dolomitic rocks which have shown reactivity are fine grained, argillaceous materials. In this respect it is of interest to note that these material characteristics are similar to those of raw materials used for the production of synthetic aggregates. Here again, an analogous reactive behavior of synthetic aggregates cannot be assumed, although a means of determining reactivity is desirable.

In the study of the carbonate reactivity, the use of concrete prisms has again proved valuable. Significant

expansions have occurred for concretes made with this type of reactive aggregates when placed above water at an elevated temperature in sealed containers. In addition, the use of high alkali cements has provided an accelerated reaction and therefore results in a shortened testing period.

For this type of reactivity as well as that previously discussed, the desirability of a moist, warm atmosphere for the promotion of various types of chemical reactions in concrete is seen. As a result, it appears that a properly designed procedure may be developed which will provide acceptability criteria for a broad variety of possible, detrimental chemical reactions of thermally processed concrete aggregates.

Sulfate Reactivity—The presence of small amounts of gypsum in cement has resulted in a like amount of calcium sulfoaluminate as a result of a reaction with the tricalcium aluminate component of the cement (10, 15). The reaction product which can produce a destructive expansion in concrete is the high-sulfate form of calcium sulfoaluminate (ettringite, $C_3A \cdot 3CaSO_4 \cdot 32H_2O$). Physical evidence of this reaction is seen not only in an expansive behavior but also in the formation of fibrous or subspherulitic groups of crystals within fractures and voids (10). In studies of this type of reactivity, as was the case for the other types of reactivity discussed, concrete prism test specimens were cured in warm moist conditions for the purpose of accelerating the reaction.

This type of reaction is, perhaps, particularly applicable to the evaluation of synthetic aggregates. This is because many raw materials contain gypsum as well as other forms of sulfur bearing minerals. The availability of these minerals for reaction is determined by the extent of thermal processing and the manner in which they are held within the aggregate matrix. Gypsum ($CaSO_4 \cdot 2H_2O$) is converted to an hydrite, $CaSO_4$, at relatively low temperatures and remains in the anhydrite form until it either reacts with silicates at temperatures in excess of $1000^\circ C$ or decomposes upon melting at $1350^\circ C$ (16). Thus, it appears that in thermal processing $CaSO_4$ is difficult to either decompose or react and may well be available for reaction with the cement in concretes.

Lime Popouts—Disruptive reactions resulting from the rehydration of anhydrous lime have been recognized as particularly troublesome with respect to synthetic aggregates. This is because many raw clay materials contain calcium bearing minerals which may decompose upon heating to form anhydrous lime. If this lime rehydrates in concrete after the final set, a disruptive expansion is likely to result. The standard test for lime popouts is an autoclave test on concrete prisms (ASTM C330 and C151).

Other Reactions—It is apparent from the preceding discussions that chemical reactions, in general, are promoted by the warm, moist curing of concrete. It therefore seems probable that other types of reactions not mentioned here will also be accelerated if the proper compounds are present. In addition, it is likely that these reactions will manifest themselves by either causing a volume change in a concrete specimen or will occur in the form of a deposit or discoloration which can be detected by visual examination.

2.3 Concrete Freeze-Thaw Durability

Theories on Freeze-Thaw Failure Mechanisms—A great deal of research has been conducted in the field of concrete freeze-thaw durability, and several very good summary reports have been published in the past few years (1, 17, 18, 19). Most of these summary reports concur in the enumeration of the aggregate properties which control the freeze-thaw behavior of concrete. These important aggregate properties are the degree of saturation, porosity, permeability, elastic accommodation, and size of the aggregates. All of these properties are related in some way to the volume and movement of absorbed water, its expansion upon freezing, and the forces exerted by the water and ice within and outside the aggregates. Synthetic aggregates are particularly influenced by these parameters since they, in general, are characterized by high porosity and permeability (1).

There is general agreement on the failure mechanisms involved in the freeze-thaw behavior of concrete (17, 19). These mechanisms can most easily be described by following the action of freezing water within the pores of aggregates within concrete. First, the presence of water inside the aggregates is due to presaturation, absorption of mix water or the ingress of water into the hardened concrete by way of cracks or capillaries. As the concrete freezes, the water inside the aggregates begins to freeze; generally the water in the smaller pores freeze first (19). Since all the water in the aggregate particles does not freeze simultaneously, hydraulic pressures are built up by the action of the expanding ice forcing the unfrozen water along paths of least resistance ahead of the moving ice front.

The forces set up by the hydraulic pressures and the expanding ice can be accommodated in several ways. First, if sufficient porosity and open pathways exist inside the aggregate, all of the water may freeze with no damage to the aggregate. Second, the stress exerted by confined and expanding ice may be elastically accommodated by the aggregates. Third, the hydraulic pressure built up within the aggregates due to the restricted movement of the freezing water may be accommodated by the expulsion of water out of the aggregate and into the surrounding paste. Of course, the accommodation of the hydraulic pressure within the paste is subject to the same aspects of stress relief just discussed with respect to the aggregates. It can therefore be seen that the absence of one or more of the stress relieving mechanisms just mentioned can result in disruptive forces in the aggregates or paste during the freezing process.

2.4 Concrete Shrinkage Behavior

Because of the many harmful effects caused by large shrinkage strains in concretes, this aspect of concrete behavior has received much attention (20). This is particularly true for synthetic aggregates because their porous nature and relatively low modulus of elasticity contribute significantly to high shrinkage strains. For this reason the ASTM specifications for structural lightweight concrete limit the allowable shrinkage strain to 1000×10^{-6} in./in. in 100 days of drying (ASTM C330-64T) (13). In view of the apparent importance of this aggregate related property, a shrinkage study was conducted in an effort to examine the effect of the degree of thermal transformation of the aggregates on concrete shrinkage.

3. Experimental Program

3.1 Experimental Procedure

Twelve coarse synthetic aggregates were produced in the Texas Transportation Institute (TTI) rotary kiln at different temperatures and retention times—six from each of two sources of raw material (coded R and Z).^{*} Raw material R is a gray shale from the Pennsylvanian system of the Paleozoic era, and from which a commercial lightweight aggregate is produced. Raw material Z is a gray, medium to soft calcareous clay, from the Eagle Ford group of the Cretaceous system of the Mesozoic era. Unit weights of approximately 35 and 50 pcf (dry loose) were achieved through variations in firing temperature coupled with retention times of around 20, 35, and 50 min. Processing parameters for aggregates R and Z are given in Table 3-1. Note that the odd and even numbered aggregates are classed as light (around 35 pcf) and heavy (around 50 pcf), respectively. In each weight category the progression of the numbering system follows a corresponding increase in retention time (values of approximately 20, 35, and 48 min.).

For control purposes one regular weight, siliceous, river-run coarse aggregate (Hearne) was also studied.

3.2 Aggregate Properties

The aggregate properties of specific gravity, absorption, saturation, and porosity were determined for all coarse synthetic aggregates used and are given in Section 4.1. The fine aggregate used was a siliceous, regular weight, river-run sand with a specific gravity of 2.60 (see Table 4.5).

Several techniques were used to study the thermal transformation characteristic of the aggregates of this

^{*}These aggregates are more fully described in Reference 4 and *Research Report 81-9*.

study. These techniques include the use of X-ray diffraction, differential thermal analysis (DTA), effluent gas analysis (EGA), and gas chromatography (GC). The details of these techniques, together with complete data are given in *Research Report 81-9*. However, a summary of the thermal transformation results utilizing the above techniques is given below. Each aggregate is identified by its code designation, its retention time in the kiln, and its firing temperature.

R raw	clay minerals—chlorite (7%), illite (62%), kaolinite (31%) other components— α -quartz, organic matter, trace of calcium carbonate and gypsum bloating gases—EGA indicates gradual release of H ₂ O and CO ₂
R-2 21 min. 2020° F	X-ray—no clay minerals detected; spinel, α -quartz present DTA, EGA, GC—no high temperature phase transition detected in DTA burn, slight indication of the presence of organic matter
R-4 36 min. 1980° F	X-ray—no clay minerals detected; spinel, α -quartz, α -cristobalite, feldspar present DTA, EGA, GC—same as R-2
R-6 48 min. 2005° F	X-ray—no clay minerals detected; spinel, α -quartz, hematite present DTA, EGA, GC—same as R-2

TABLE 3-1. PROCESSING PARAMETERS FOR AGGREGATES R AND Z

Processing Parameters	Aggregates R						Aggregates Z						
	Comm.	1	3	5	2	4	6	1	3	5	2	4	6
% Moisture* Raw Material	—				3.8%						11.5%		
Grading Raw Material	—			1 in. — #4							1 in. — #4		
Feed Rate (CFM)	—	.145	.090	.062	.145	.090	.062	.145	.090	.062	.145	.090	.062
Kiln Slope (in./ft)	—	.730	.433	.306	.730	.433	.306	.730	.433	.306	.730	.433	.306
Kiln Speed (rpm)	—	1.6	1.5	1.5	1.6	1.5	1.5	1.6	1.5	1.5	1.6	1.5	1.5
Retention Time R. T. (mins.)	—	21	36	48	21	36	48	20	34	48	20	34	48
Loading Factor (%)	—	3.9	4.1	3.9	3.9	4.1	3.9	3.8	3.9	3.9	3.8	3.9	3.9
Max. Temp.** T _m (°F)	≈2000	2115	2090	2115	2020	1980	2005	2009	2035	2040	1712	1685	1850
Flame	—			oxidizing							oxidizing		
Unit Wt.*** U.W. (pcf)	48.1	37.1	36.4	35.8	53.0	58.5	52.1	33.4	34.2	32.3	46.5	50.4	42.5

*% Moisture determined by oven drying at 200° F for minimum of 24 hrs.

**Obtained by optical pyrometer readings.

***Tested and Graded according to midpoints of grading limits set for ¾ in. — No. 4 aggregates of ASTM C29 and C330.²

R-1 X-ray—no clay minerals detected; spinel, α -quartz present
21 min.
2115°F

DTA, EGA, GC—same as R-2

R-3 X-ray—no clay minerals detected; spinel, α -quartz, β -quartz, feldspar present
36 min.
2090°F

DTA, EGA, GC—same as R-2

R-5 X-ray—no clay minerals detected; spinel, cristobalite, α -quartz, β -quartz, feldspar present
48 min.
2115°F

DTA, EGA, GC—same as R-2

R-comm. X-ray—no clay minerals detected; spinel, cristobalite, mullite, feldspar, α -quartz present

DTA, EGA, GC—slightly more organic matter than other six aggregates R

Z-raw clay minerals—montmorillonite (29%), illite (44%), kaolinite (27%)

other components— α -quartz, pyrite, organic matter, much calcium carbonate, and small amounts of gypsum

bloating gases—EGA indicates large quantities of rapid evolutions of CO_2 , SO_2 , and H_2O

Z-2 X-ray—no clay minerals detected; spinel, dolomite, α -quartz, feldspar present
20 min.
1712°F

DTA, EGA, GC—incomplete formation of high temperature phases, probable presence of partially hydrated clay minerals, sizeable quantity of organic matter, and carbonate

Z-4 X-ray—no clay minerals detected; spinel, pyrite, α -quartz, feldspar present
34 min.
1685°F

DTA, EGA, GC—incomplete formation of high temperature phases, probable presence of partially dehydrated clay minerals, sizeable amount of organic matter, slight indication of carbonate

Z-6 X-ray—no clay minerals detected; spinel, cristobalite, α -quartz, feldspar present
48 min.
1850°F

DTA, EGA, GC—slight indication of incomplete high temperature transformation, slight indication of partially dehydrated clay minerals, appreciable organic matter

Z-1 X-ray—no clay minerals detected; spinel, cristobalite, mullite, α -quartz, feldspar present
20 min.
2009°F

DTA, EGA, GC—small amount of organic matter, slight indication of incomplete high temperature transformations

Z-3 X-ray—no clay minerals detected; spinel, cristobalite, mullite, α -quartz, feldspar present
34 min.
2035°F

DTA, EGA, GC—small amount of organic matter, very slight indication of incomplete high temperature transformations

Z-5 X-ray—no clay minerals detected; spinel, cristobalite, mullite, α -quartz, feldspar present
48 min.
2040°F

DTA, EGA, GC—small amount of organic matter, very slight indication of incomplete high temperature transformations

3.3 Concrete Strength

The batch properties for both the freeze-thaw concretes and reactivity concretes given in Tables 4-6 through 4-9 of the Appendix show a wide variation in the compressive strengths for concretes having similar mix proportions. These variations are shown in a plot of the compressive strengths as a function of aggregate porosity given in Figure 3-1. Although the magnitudes of the variations shown might not generally be expected, consideration of the cement factors, air contents and porosities serves to partially explain the deviations noted.

First, note in Figure 3-1 that the least thermally transformed aggregates, Z-2 and Z-4, apparently produce no unfavorable effect on 28-day compressive strengths of concrete. This is a significant finding since compressive strengths are perhaps the most widely used indicators of concrete quality, and based upon the results of this study, may be very misleading in conjunction with certain types of aggregates (i.e., Z-2 and Z-4).

In Figure 3-1 note that in 8 of 13 batches of the reactivity series, concrete strengths are higher than those of the freeze-thaw concretes even though the water-cement ratios for the reactivity batches are higher. This somewhat contradictory behavior can be partially explained by the values of air content and cement factor. The data of Tables 4-6 through 4-9 show that the air contents of the freeze-thaw concretes were approximately twice that of the reactivity concretes. As a result, one would expect the strengths of the air entrained freeze-thaw concretes to be decreased with respect to similar batches of non-air entrained concretes.

In addition note that while the cement factors given in Figure 3-1 are approximately the same for both concrete series (4.9 sk/cu. yd.), the approximate water-cement ratios are significantly different (7.4 and 9.5 gal/sk.). Here, it is important to remember that the calculation of the water-cement ratio is subject to error because of the uncertainty involved in estimating how

much mixing water is actually available for the hydration of the cement and how much is absorbed by the aggregates during and after mixing. This uncertainty tends to support the use of the cement factor at equal consistency in lieu of the water-cement ratio as a gage of the concrete's strength. If the cement factor is an important parameter, then the strengths of the two series of batches should be somewhat similar.

When the parameters of air content and cement factor are considered jointly, the relationships of the strengths of the two different concrete series seem much more plausible than when only the water-cement ratios (questionable as they may be) are considered.

Finally, a general trend for the concrete strength as a function of aggregate porosity is shown by the dashed lines of Figure 3-1. Note that the very porous aggregates (numbers 1, 3 and 5) generally yielded concretes of lower strength in comparison with the denser-aggregate concretes (2, 4, and 6). This behavior seems reasonable in view of the fact that the greater the porosity, the lower the density and the greater the number of voids within a given cross-section of the concrete.

3.4 Concrete Reactivity Series

General Comments—A discussion of aggregate rehydration and several types of harmful concrete-aggregate reactions was given in Section 2.1 and 2.2. A description of the test procedure for concrete reactivity is given in Section 4.4.

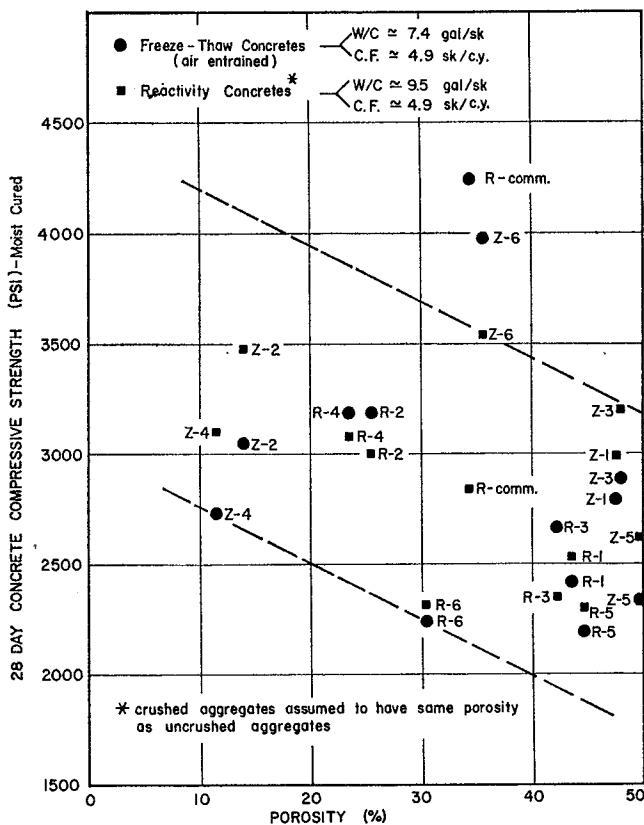


Figure 3-1. Concrete strength as a function of aggregate porosity.

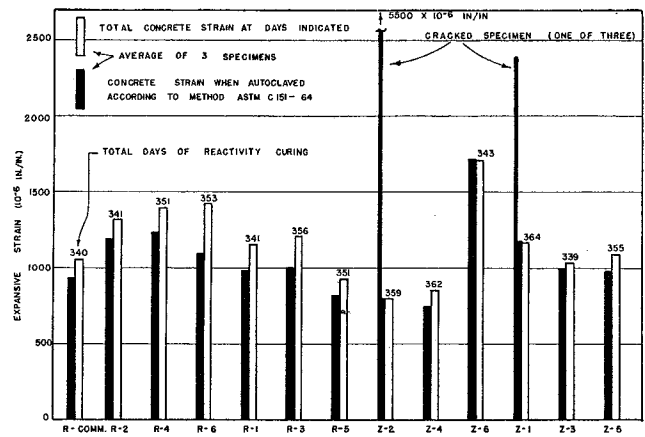


Figure 3-2. Concrete expansive strain due to autoclaving followed by reactivity curing.

In general, all thirteen synthetic aggregates of this study were crushed, combined with a specified amount of natural siliceous sand and mixed with water and cement so that the resulting concretes were characterized by similar water-cement ratios and cement factors. After an appropriate curing period, three concrete prisms from each batch were autoclaved. Length measurements before and after autoclaving gave the expansive strain caused by the autoclave process.

Following the autoclave test, the concrete specimens were placed over a water bath at $104^{\circ} \pm 2^{\circ}\text{F}$, and periodic length measurements were made. Test procedures consisted of strain measurements and visual observations for cracks and reaction deposits.

Concrete Autoclave Test Results—The expansive strains resulting from the autoclave test are shown by the solid blocks of Figure 3-2. Note that concretes made with aggregates Z-2 and Z-1 show very high expansive strains resulting from one of each group of three specimens having cracked during autoclaving. A photograph showing the cracked specimens is given in Figure 3-3. The exact cause of this behavior is not known, although the failure of only one of three specimens tends to indicate that a single or closely spaced group of reactive aggregates was responsible. Possible causes are the hydration of anhydrous lime and CaSO_4 . A general rehydration of clay minerals is not indicated since aggregate Z-4 showed relatively low expansions as did two of three specimens in each of Z-2 and Z-1.

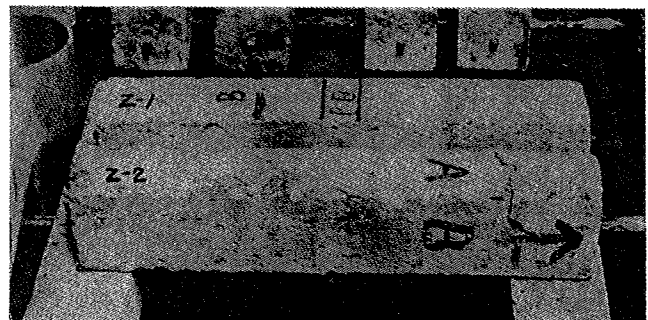


Figure 3-3. Concrete specimens Z-1 and Z-2 after autoclave testing.

Concrete specimens made with aggregate Z-6 showed a different type of behavior when subjected to the autoclave test. In this case all three specimens expanded uniformly to about 1700×10^{-6} in./in. strain, and no cracking was noted. Although aggregate Z-6 is classed as the third least transformed material of this study, no conclusive statement can be made concerning the cause of the expansion in view of the rather low expansion for Z-4 which was even less thermally transformed.

Autoclave tests performed on concretes made with aggregates R produced relatively low expansions of a uniform nature, and no cracking was observed.

Suggested Failure Criteria—Even though it was not possible to determine the exact causes of expansion in the three concretes Z-1, Z-2, and Z-6, the fact that relatively large expansive strains were indicated by the test procedure is significant. Noting that expansive strains of 1000×10^{-6} in./in. at 6 months are considered destructive in the alkali reactivity test (ASTM C227-67) (13), it seems probable that structural concretes made with aggregates Z-1, Z-2 and Z-6 will also suffer destructive volume changes on aging under certain conditions.

To make a firm statement of acceptance criteria for autoclaved concretes is not justified at this time because of the lack of sufficient data. However, a tentative suggested criterion based upon the limited data of this study may be helpful. With these qualifications, it is suggested that an aggregate, not proven by field performance, should be considered as unacceptable for structural use if any one of three specimens tested as described in Section 4.4 exceeds an expansive strain of 1500×10^{-6} in./in. during autoclaving.

Long Time Reactivity Curing Test Results—After the concrete specimens were autoclaved, they were placed in a humid atmosphere at $104^\circ \pm 2^\circ\text{F}$ for extended periods of time (up to one year). Periodic length measurements were taken in order to maintain a continuous record of any volume change activity. The results of this long-time reactivity curing are also given in Figure 3-2 (the outlined blocks). Here the outlined blocks give the magnitude of the total strain, including autoclave strain, at the number of curing days indicated. In this way any difference between the solid and outlined blocks of Figure 3-2 represents either an expansion or contraction during the long-time reactivity curing.

Note that ten out of the thirteen concretes tested continued to expand a small amount and that the other three remained essentially dimensionally stable. If the hypothesis is accepted that harmful reactions occur if long time expansions are observed, then a conclusion might be drawn that some harmful reactions did occur. However, the extent of the expansions is slight (less than 200×10^{-6} in./in. additional expansion beyond autoclaving for all concretes except R-6, which exhibited an additional 320×10^{-6} in./in. expansion). Thus, these slight expansions were not considered significant and it is concluded that no harmful reactivity was indicated by warm, moist curing for the approximately one year time periods shown when the specimens were initially autoclaved.

Visual Observations—Although no noteworthy evidence of chemical reactivity was observed on the surfaces of any concrete specimen, an interesting find resulted when the two cracked specimens (Z-1 and Z-2)

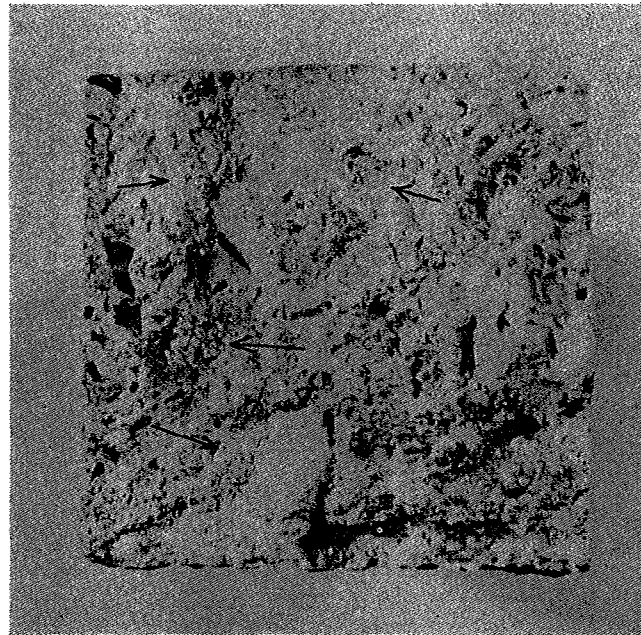


Figure 3-4. Cross-sectional view of cracked area of concrete made with aggregate Z-2 (arrows point to reaction deposits).

were broken open after curing periods of 106 and 101 days, respectively. A photograph of the cross-section at the cracked area of concrete Z-2 is shown in Figure 3-4. The arrows in Figure 3-4 point to white deposits which seem to be clustered about certain of the coarse aggregate particles.

Observation of these areas with the aid of a microscope revealed that the deposits were present in two different forms. Photomicrographs showing the two deposit forms in concrete Z-2 are given in Figure 3-5. Similar deposits were also found in the concrete made with the aggregate Z-1.

Even though the aggregates were suspected as the source of these deposits, the role that cement may have played in their formation is unknown. Therefore, samples of each of the thirteen aggregates were boiled in distilled water for 5 to 10 minutes, and then left half submerged at room temperature for several days. After the water had evaporated, white deposits were noted in a number of pieces of aggregates Z, but none were seen on any of aggregates R.

The deposits found on aggregates Z were of two forms and are shown in Figure 3-6. Note that even though differences exist, the deposits in the concretes (Figure 3-5) and those found on the aggregates (Figure 3-6) show definite similarities. When considering that it is common for many crystals to exhibit different forms, it seems very likely that the deposits of Figures 3-5 and 3-6 are of similar composition. However, the possibility that a reaction involving cement may have resulted in the minor differences between the deposits cannot be precluded.

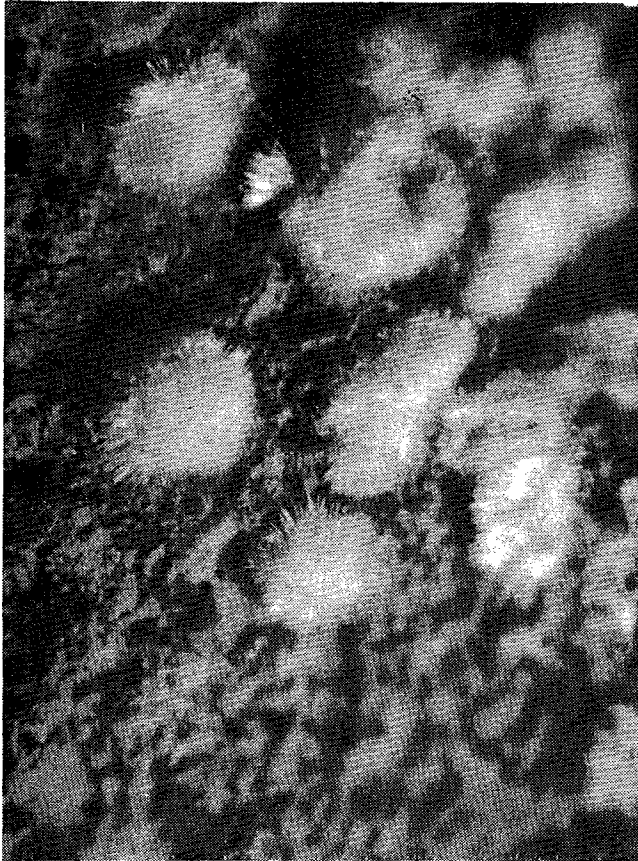
Identification of Deposits—Scrapings from the aggregate deposits were subjected to qualitative chemical tests in an effort to determine their identity. An acid

treatment showed no effervescing action, and therefore calcium carbonate was ruled out as the source of deposits.

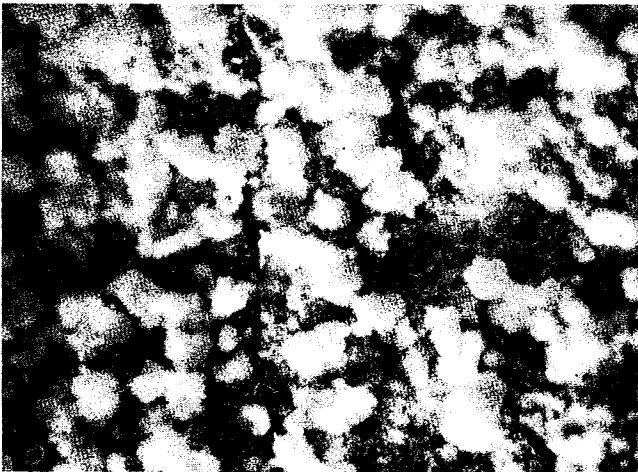
A second treatment involved dissolving the deposits in about $\frac{1}{2}$ cc of distilled water and two drops of dilute hydrochloric acid. The formation of a white precipitate upon addition of four drops of barium chloride indicated that a sulfate such as CaSO_4 or Na_2SO_4 was present. Considering the facts that gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) is

reported to sometimes occur in a fibrous structure and that some indication of anhydrite was noted in the transformation study of aggregates Z, it is very likely that calcium sulfate was a component of the deposits in question.

A Possible Cement-Gypsum Reaction—The presence of anhydrite in aggregate Z prompts the consideration of a cement-gypsum reaction as the cause of the deposits in the concretes of Figure 3-5. It has been reported that

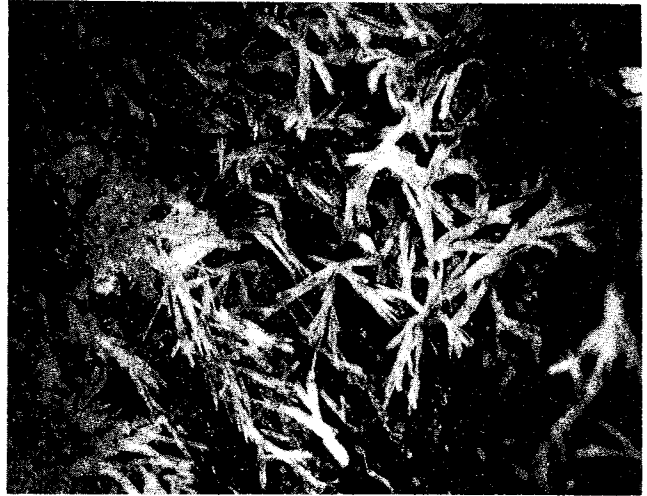


(35X)



(35X)

Figure 3-5. Photomicrographs of deposits found in cracked areas of concrete Z-2 following reactivity curing.



(35X) Aggregate Z-1



(35X) Aggregate Z-6

Figure 3-6. General type of deposits formed on aggregates Z after boiling followed by evaporation at room temperature.

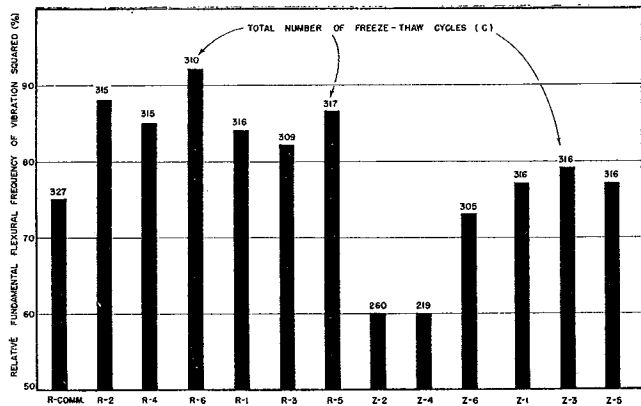


Figure 3-7. Freeze-thaw data for concretes containing aggregates R and Z.

a potentially harmful expansive reaction can occur between gypsum and the hydration products of cement (4, 9). The product of this reaction is calcium sulfoaluminate (ettringite, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$). This product is crystalline in nature and is described as occurring in the voids and cracks of affected concrete (10). Furthermore, this compound is described as being precipitated "as fibrous and subspherulitic groups of crystals." The similarity of both form and occurrence

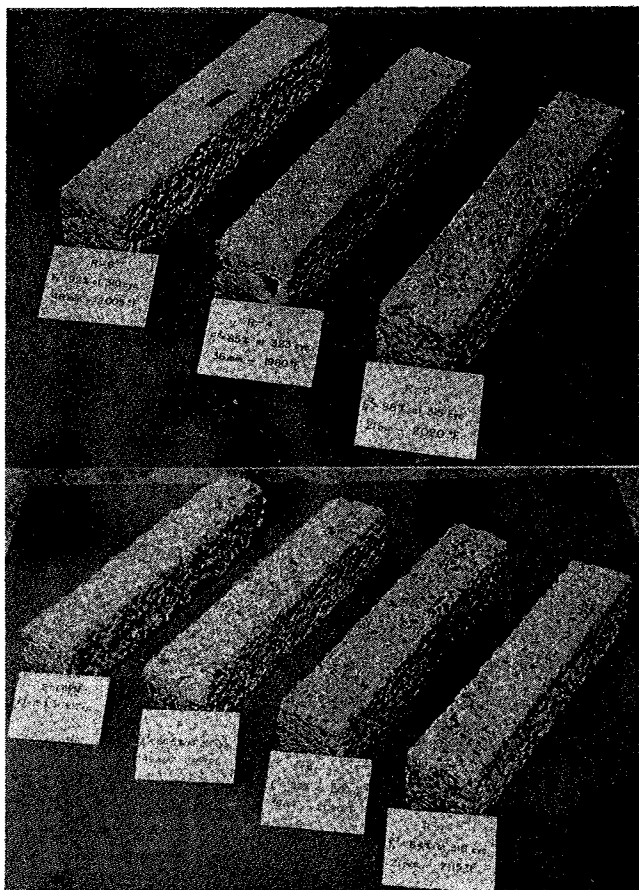


Figure 3-8. Freeze-thaw specimens containing aggregates R.

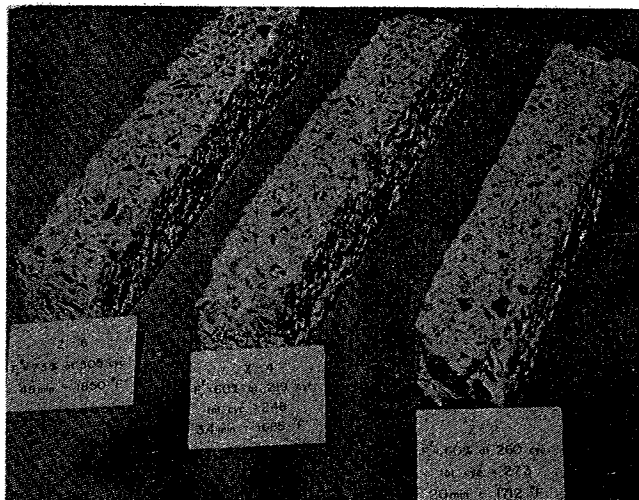


Figure 3-9. Freeze-thaw specimens containing aggregates Z.

of ettringite and the deposits shown in Figure 3-5 is striking. The involvement of anhydrite in the deposits formed on aggregate Z, suggests that the deposits pictured in the upper photograph of Figure 3-5 are very possibly that of ettringite. In any case, there can be little doubt that a potentially harmful sulfate material is present in aggregates Z and that destructive expansions have been exhibited by concretes made with aggregates Z-1, Z-2, and Z-6 when subjected to autoclaving.

3.5 Concrete Freeze-Thaw

The results of the freeze-thaw tests are presented by the bar graph in Figure 3-7. Photographs of specimens after testing are shown in Figures 3-8 and 3-9. If it is assumed that a freeze-thaw failure occurs whenever the relative squared frequency of vibration drops to 60 percent before 300 cycles* of testing are concluded, then the concretes made with aggregates Z-2 and Z-4 are the only failures noted. Also, with the exception of R-comm., the remaining four concretes of aggregates Z show more deterioration than any of the concretes made with aggregates R. The exception noted for aggregate

*The definition of failure for these conditions is in accordance with the general implications of ASTM C290-67.

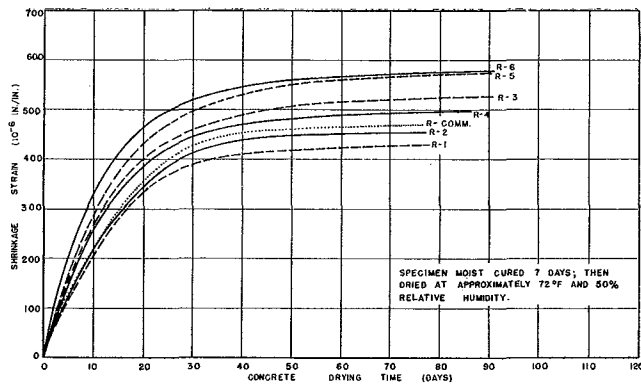


Figure 3-10. Shrinkage strain vs. drying time for concretes made with aggregates R.

R-comm. was probably due to the fact that this aggregate was batched with a degree of saturation of 38.2 percent as compared to 20 percent for all others. (See *Research Report 81-6.*)

The failures noted for Z-2 and Z-4 are for concretes made with the least thermally transformed aggregates as determined by transformation tests of all thirteen materials tested (4). In addition, the low durability of concretes made with aggregates Z-6, Z-1, Z-3 and Z-5 in relation to aggregates R is perhaps explained by a comparison of their transformation properties.

Consideration of Intervening Factors—Although the freeze-thaw data show very good agreement with the behavior expected on the basis of thermal transformation properties, the intervening factors of absorption and saturation require some discussion. First, Figures 4-3 and 4-4 (Section 4.1) show that aggregates Z-2 and Z-4 are definitely the most easily saturated aggregates. From the previous discussions of the importance of saturation in affecting freeze-thaw durability, no conclusive statements can be made that thermal transformation factors are solely responsible for the freeze-thaw behavior of these two concretes. A close examination of Figure 3-9 reveals that the deterioration of concretes Z-2 and Z-4 can definitely be attributed to the disintegration of the aggregates and not the mortar. This indicates that these poorly burned aggregates disintegrated due to a combination of differential thermal stresses, hydraulic-

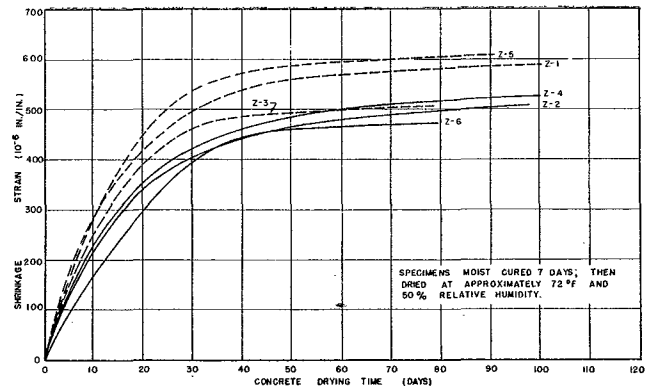


Figure 3-11. Shrinkage strain vs. drying time for concretes made with aggregates Z.

ice pressure, and possibly expansion of rehydrating clay minerals. In contrast, note in Figure 3-8 that practically all of the exposed aggregates of the specimens made with aggregates R remain intact. Also note that the comparatively minor deterioration of these specimens is due to the disruption of the mortar.

When all of these durability related factors are considered, it seems logical to conclude that the primary cause of the freeze-thaw failures of this study is related to the degree of thermal transformation of the aggregates. The degree of saturation is important, but in this particular case, can be considered secondary.

3.6 Concrete Shrinkage

The results of the shrinkage study are shown by the shrinkage strain vs. time relationships of Figures 3-10 and 3-11. It is apparent from these results that none of the concretes of this study have shrinkage strains exceeding the 1000×10^{-6} in./in. limit set by ASTM C330-64T. It is also apparent that no significant relationship exists between the degree of thermal transformation of the concrete aggregates and the shrinkage behavior of the concrete.

On the basis of these results, it may be concluded that the shrinkage behavior of synthetic aggregate concrete is not a reliable indicator of incomplete thermal transformation of the aggregates.

4. Appendix

4.1 Coarse Aggregate Properties

Specific Gravity—The determination of the specific gravity for aggregates sometimes furnishes an important relationship between the basic aggregate properties and the structural use related properties. In addition, the determination of the specific gravity is necessary for such basic calculations as concrete and asphalt mix designs, absorption and saturation properties. For these reasons tests were conducted in order to determine the bulk specific gravity and the absolute specific gravity of all the aggregates of this study. The results of these determinations are given in Table 4-1.

From Table 4-1 it can be seen that the bulk specific gravities of the lighter aggregates (1, 3, 5) and the heavier aggregates (2, 4, 6) follow the general relationship that would be expected from the data on unit weights. It is also interesting to note that the crushed aggregates give higher bulk specific gravities in every case when compared to the uncrushed aggregates. This is perhaps explained by the fact that a considerably greater number of open surface pores in the crushed aggregates are immediately saturated in the pycnometer test method used. The exclusion of these pores from the bulk volume determination would tend to increase the bulk specific gravity.

TABLE 4-1. SPECIFIC GRAVITIES OF COARSE AGGREGATES

Specific Gravity	Aggregate Type													
	comm.	1	Aggregates R						Aggregates Z					
			3	5	2	4	6	1	3	5	2	4	6	
Dry Bulk Specific Gravity ¹ Uncrushed ²	1.43	1.16	1.10	1.03	1.66	1.73	1.53	1.15	1.10	1.03	1.67	1.77	1.44	2.64
Crushed ³	1.52	1.28	1.21	1.18	1.73	1.83	1.61	1.33	1.28	1.16	1.76	1.80	1.52	—
Absolute Specific Gravity ⁴ Uncrushed	2.17	2.05	1.90	1.86	2.23	2.26	2.18	2.19	2.12	2.05	1.94	2.00	2.24	2.66

(All specific gravity values given above represent the average of three tests)

¹Dry Bulk Specific Gravity—The basic method of Bryant⁴⁰ was used for this calculation. The method utilizes a pycnometer technique in which oven dried samples are immersed in water during a timed series of 2, 4, 6, 8, 10, 20, 30, 60, and 120 minutes. Readings of the total weight of the pycnometer, aggregate and maximum water capacity are made at each time interval and the resulting curve from these data is extrapolated back to zero time in order to obtain the volume of water in the pycnometer when no aggregate voids are saturated. Since the volume of the pycnometer is known, the dry bulk volume is easily calculated as well as the dry bulk specific gravity.

²Uncrushed representative sample graded -1 in. to + No. 4 size.

³Crushed aggregate used for reactivity tests graded -1/2 in. to + No. 30 size.

⁴Absolute Specific Gravity—The absolute specific gravity of the aggregates was determined by the use of a pressure pycnometer in which the aggregates were saturated under 1200 psi water pressure.

By using a pressure pycnometer, the aggregates were saturated under 1200 psi to obtain the absolute specific gravity. Although 100 percent saturation was not guaranteed by this technique, the procedures used insured representative results, and the assumption of 100 percent saturation for the purpose of comparison seems justified. The values given in Table 4-1 in every case are significantly higher than the bulk values, and as might be expected, the absolute specific gravities for all of the aggregates are not greatly different.

The utility of both the bulk and absolute specific gravities can be seen in a study of the absorption-saturation characteristics of the aggregates. The following section gives a calculation procedure and the resulting relationships concerning these important properties.

Absorption—In obtaining the absorption properties with respect to time, the samples used for the determination of the bulk specific gravity were continued in the

soaking cycle for 14 days. Periodic readings were taken (Tables 4-2 and 4-3) and the resulting absorption vs. time relationships were plotted as shown in Figures 4-1 and 4-2. Each of these curves represents an average curve drawn through the data obtained from three samples. The absorption values given at 1200 psi water pressure are assumed to be a 100 percent saturated condition. The method of calculation for this value is given in the latter paragraphs of this section.

Several interesting relationships are shown by Figures 4-1 and 4-2. Note in Figure 4-1 that both the lighter (R-1, R-3, R-5) and the heavier (R-2, R-4, and R-6) aggregates show absorption values at 14 days which are apparently related to the kiln retention time (remembering the sequences 1, 3, 5, and 2, 4, 6 represent increasing retention times). Here, for aggregates within the same unit weight range, the absorption tends to increase with a decreasing retention time. This behavior

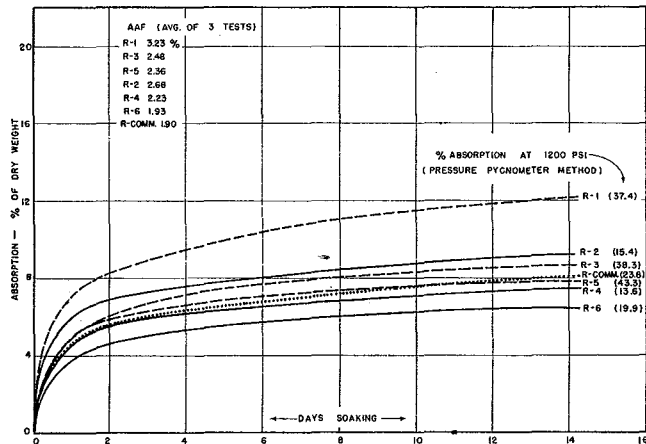


Figure 4-1. Absorption characteristics of aggregates R—pycnometer method.

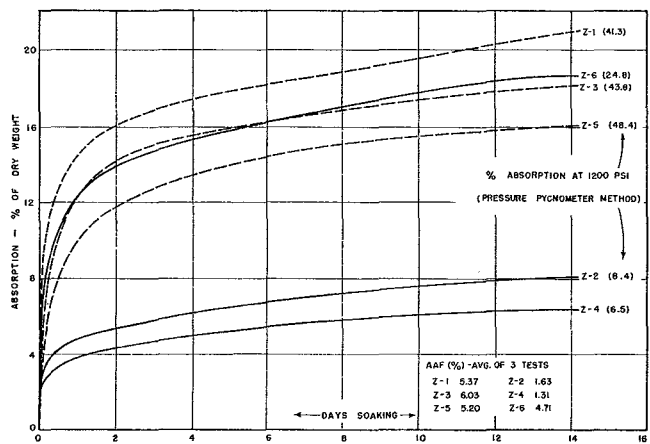


Figure 4-2. Absorption characteristics of aggregates Z—pycnometer method.

TABLE 4-2. AGGREGATE ABSORPTION DATA FOR AGGREGATES R

R-1		R-3		R-5		R-comm.	
Days Soaking	Absorption (%)	Days Soaking	Absorption (%)	Days Soaking	Absorption (%)	Days Soaking	Absorption (%)
0.083	3.48	0.083	2.20	0.083	2.12	0.083	2.42
0.19	4.59	0.21	3.08	0.74	4.39	1.0	4.49
0.88	7.05	0.90	4.93	0.85	4.78	3.0	5.66
1.01	6.99	1.03	5.18	1.05	5.09	6.0	6.39
1.21	7.57	1.31	5.44	1.78	5.64	7.0	6.94
1.94	8.31	1.96	6.06	4.76	6.62	8.0	7.35
4.9	9.47	4.92	7.09	5.74	7.00	10.0	7.41
5.9	10.38	5.9	7.53	7.0	7.29	13.0	7.94
7.2	10.87	7.2	7.74	8.8	7.56	14.0	8.12
9.0	11.31	9.0	8.13	11.8	7.56		
12.0	11.68	12.0	8.26	13.7	7.95		
13.9	12.21	14.0	8.61				

R-2		R-4		R-6	
Days Soaking	Absorption (%)	Days Soaking	Absorption (%)	Days Soaking	Absorption (%)
0.083	2.97	0.083	2.14	0.083	1.57
0.78	5.66	0.46	4.21	0.13	1.87
0.90	5.90	0.84	4.51	0.83	3.64
1.10	6.19	0.96	4.68	0.93	3.85
1.83	6.67	1.19	5.28	1.13	4.09
4.81	7.75	1.80	5.42	1.85	4.61
5.78	8.04	2.01	5.62	4.84	5.55
7.1	8.37	2.7	5.79	5.8	5.90
8.9	8.74	5.7	6.42	7.1	6.10
11.9	9.04	8.0	6.85	8.9	6.35
13.8	9.25	9.7	7.10	11.9	6.44
		12.8	7.32	13.8	6.61
		13.7	7.63		

may be explained when it is considered that the aggregates with short retention times experienced a relatively rapid evolution of gases and a short interval for surface fusion. These factors both tend to influence the characteristics of the surface coating of the aggregates. In

Figure 4-2 the same general behavior is noted with one exception (aggregate Z-6). The higher absorption behavior of Z-6 is probably due to the fact that its unit weight (42.5 pcf) was unusually low for the heavy aggregate group. This indicated that considerably more

TABLE 4-3. AGGREGATE ABSORPTION DATA FOR AGGREGATES Z

Z-1		Z-3		Z-5	
Days Soaking	Absorption (%)	Days Soaking	Absorption (%)	Days Soaking	Absorption (%)
0.083	8.83	0.083	5.98	0.083	4.30
0.13	9.49	0.21	7.92	0.73	9.20
0.73	13.60	0.83	11.62	0.83	9.62
0.88	14.12	0.98	11.67	1.10	10.30
1.04	14.50	1.14	12.63	1.75	11.17
1.74	15.65	1.83	13.62	2.74	12.50
2.78	16.95	2.87	14.70	3.80	13.33
5.8	18.10	5.9	16.25	6.8	14.45
8.1	19.00	8.1	17.05	9.1	15.30
9.8	19.70	10.1	17.55	10.8	15.60
12.9	20.30	13.0	17.88	13.6	15.75
13.7	21.10	14.8	18.55		

Z-2		Z-4		Z-6	
Days Soaking	Absorption (%)	Days Soaking	Absorption (%)	Days Soaking	Absorption (%)
0.083	2.98	0.083	2.21	0.083	7.62
0.17	3.48	0.67	3.43	0.27	9.91
0.79	4.50	0.77	3.58	0.89	12.21
0.94	4.69	1.03	3.76	1.03	12.57
1.09	4.89	1.69	4.06	1.19	12.90
1.80	5.24	2.68	4.49	1.89	13.80
2.83	5.68	3.74	4.83	2.93	14.62
5.8	6.69	6.7	5.63	5.9	16.03
8.1	7.37	9.0	6.09	8.2	17.02
9.9	7.73	10.7	6.28	9.9	17.58
12.9	7.99	13.7	6.35	13.0	18.26
13.8	8.21			13.9	18.69

bloating occurred than desired in the design of the research program. Although the desired unit weight of approximately 50 pcf could normally be obtained, a shortage of raw material prevented an adjusted kiln run for this aggregate.

In addition to the relationship discussed, note also in Figures 4-1 and 4-2 that the lighter weight materials generally show higher absorptions with time than the heavier materials. This seems logical when one considers the total void systems available for saturation. The relative magnitudes of the void systems are related in a general way by the bulk specific gravities and the absorptions at 1200 psi pressure. In this respect more useful data are given in the following section on saturation.

The effect of surface permeability is shown by Figures 4-1 and 4-2. In Figure 4-1 it is obvious that although the lighter weight aggregates have much larger void systems, the fusion of the surface layers has effectively limited the increase in absorption. On the other hand, the more porous structure of the surface layers of aggregates Z-1, Z-3, Z-5, and Z-6 has allowed a considerable saturation of voids of these bloated aggregates.

Saturation—In this report the degree of saturation refers to the percentage of the total void system of the aggregates that is filled with water. In the study of the saturation properties the data obtained by the absorption pycnometer procedure were employed in conjunction with those from the pressure pycnometer procedure. The manner in which these calculations were made is important and is explained here. The derivation which follows is based on calculations in c.g.s. units.

$$\text{Abs. Vol. Voids}^* = \text{Bulk Vol.} - \text{Abs. Vol.}$$

$$= \frac{\text{Dry Wt.}}{\text{Bulk SG}} - \frac{\text{Dry Wt.}}{\text{Abs. SG}}$$

$$= \text{Dry Wt.} \left[\frac{1}{\text{Bulk SG}} - \frac{1}{\text{Abs. SG}} \right] \dots\dots\dots 1$$

*The term absolute is used on the basis of the assumption that all of the aggregate's voids are water saturated at 1200 psi pressure.

**S.G. refers to specific gravity.

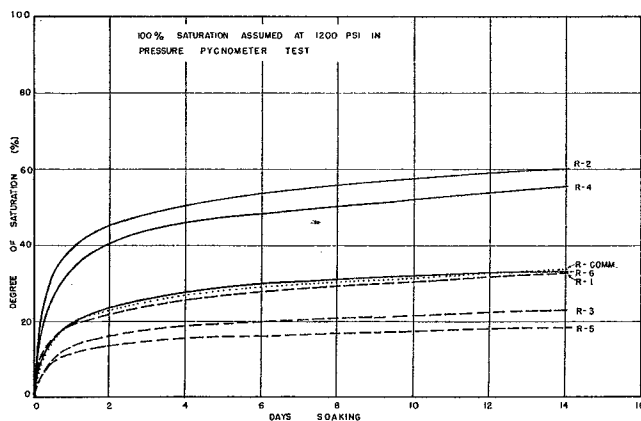


Figure 4-3. Void saturation characteristics of aggregates R.

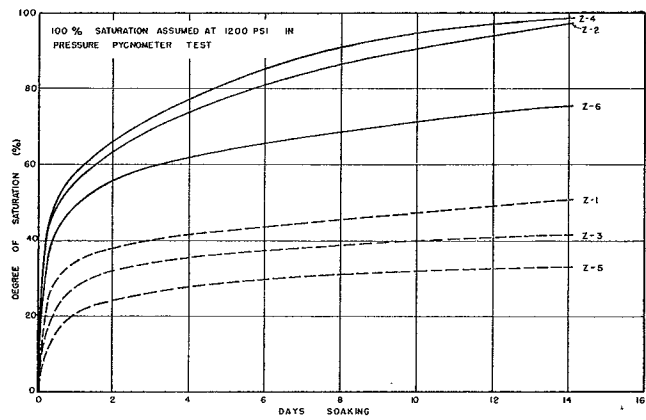


Figure 4-4. Void saturation characteristics of aggregates Z.

Once the absolute volume of voids is determined, the degree of saturation at any value of absorption (A) can be calculated. This calculation can be made as follows.

$$\begin{aligned} \text{Deg. of Sat.} &= \left[\frac{\text{Vol. Absorbed H}_2\text{O}}{\text{Abs. Vol. Voids}} \right] 100 \\ &= \left[\frac{(\%A) (\text{Dry Wt.})/100}{\text{Dry Wt.} \left[\frac{1}{\text{Bulk SG}} - \frac{1}{\text{Abs. SG}} \right]} \right] 100 \\ &= \frac{\%A}{\left[\frac{1}{\text{Bulk SG}} - \frac{1}{\text{Abs. SG}} \right]} \dots\dots\dots 2 \end{aligned}$$

Thus, once the specific gravity values are found, the saturation characteristics can be determined with the aid of absorption data. The significance of this material property, termed degree of saturation, is not fully established although its superiority over the absorption term seems certain. An effort to justify this statement is made in the remainder of this section and additional comments are found in Section 3.4 on freeze-thaw durability.

When the absorption data obtained by the pycnometer method are converted to degree of saturation by means of equation 2 the resulting relationships are those shown in Figures 4-3 and 4-4. These relationships can be directly compared with those of absorption given by Figures 4-1 and 4-2. By this comparison several interesting facts are seen. First, note that the aggregates showing relatively low total absorptions at 14 days (aggregates Z-2, Z-4, R-4, and R-6) have high saturation values at 14 days. In addition, the shapes of the initial portions of the saturation curves show that the rates of saturation are generally higher for these four aggregates. Also note that the saturation rates seem to be inversely related to the absorption rates as indicated by the shape of the initial portions of the absorption curves of Figures 4-1 and 4-2.

Although this seemingly contradictory behavior can be explained by a consideration of the porosity and the surface characteristics of the aggregate, it does serve to demonstrate possible shortcomings in the use of absorption properties for predicting certain aspects of aggregate durability. For example, if the expansion of ice in a closed volume is deemed an important durability factor, then most probably the degree of saturation and not absorption is a controlling factor in this respect. If a measure of rate of moisture intake by the aggregate is of interest, then a rate measurement by means of the saturation curves should be meaningful. In summary, it appears that the saturation characteristics can furnish not only the same type of information as obtained from absorption properties, but also information which is more directly related to those physical aspects which affect aggregate durability. An additional discussion of the particular problem of freeze-thaw durability is found in Section 3.4.

Effect of Crushing on Absorption—Since many synthetic aggregate manufacturers size their products by crushing, a comparison of the 14-day absorption values for crushed and noncrushed aggregates was made, and the results are shown in Figure 4-5. From Figure 4-5 it can be seen that crushing resulted in an increased absorption in every case. Also, the increase was generally larger for those aggregates with the greater pore volumes. This is particularly true in the case of aggregates R-1, R-3, and R-5 where the fracture of the fused surfaces is evident in the increased absorptions.

Porosity—For the purposes of this report, the coarse aggregate porosity is defined as the volume of aggregate voids saturated in a pressure pycnometer at 1200 psi water pressure, expressed as a percent of the bulk volume of the aggregate. This relationship is easily obtained from equation 1. If equation 1 is divided by the expression for bulk volume (dry weight/bulk specific gravity) the following equation for porosity is obtained:

$$\text{Porosity} = \left[1 - \frac{\text{Bulk SG}}{\text{Abs. SG}} \right] 100 \dots\dots\dots 3$$

Using equation 3 the porosities for all fourteen aggregates are given in Table 4-4.

TABLE 4-4. COARSE AGGREGATE POROSITY DATA

Aggregate	Porosity, Percent
R-comm	34.1
R-1	43.4
R-3	42.1
R-5	44.6
R-2	25.5
R-4	23.5
R-6	30.3
Z-1	47.5
Z-3	48.1
Z-5	49.8
Z-2	14.0
Z-4	11.5
Z-6	35.7
Hearne	.6

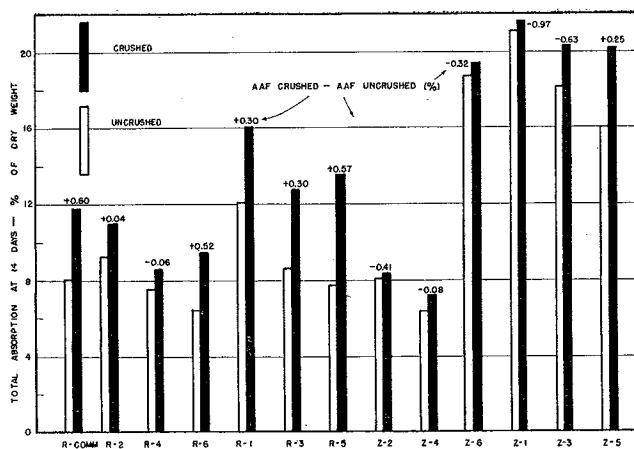


Figure 4-5. Fourteen-day absorption values for crushed and uncrushed aggregates.

4.2 Fine Aggregate Properties

The fine aggregate used in this study was a regular weight, river-run, siliceous sand. Engineering properties of the fine aggregate are given in Table 4-5.

4.3 Design, Mixing and Control Tests for Concrete

Presoaking of Coarse Aggregate—All synthetic coarse aggregates of this study were presoaked to a specific degree of saturation prior to mixing. A saturation of 20 percent was selected for the freeze-thaw concretes and a saturation of 30 percent was selected for the reactivity test program. The basis for selection of these saturations is discussed in *Research Report 81-6* (1).

Mixing Sequence—At the conclusion of the soaking period, the preweighed coarse aggregates were drained, reweighed, and then placed into a 3 cu. ft. capacity rotary tilting drum mixer. The natural siliceous fine aggregate was then added to the mixer along with the proper quantity of Type I portland cement. These three components were dry mixed for about one minute.

TABLE 4-5. PROPERTIES OF FINE AGGREGATE H USED IN ALL CONCRETE BATCHES

Specific Gravity (SSD) (at 24-hr. soaking)	—	2.60
Absorption, percent (same condition above)	—	0.6 percent
Dry Loose Unit Weight, pcf	—	105.6
Sieve Analysis		
Sieve Size	Cumulative Percent Retained	
No. 8	12.8	
No. 16	27.5	
No. 30	50.1	
No. 50	90.6	
No. 100	98.9	
No. 200	99.9	
Pan	100.0	

In determining the proper amount of mixing water, it was assumed that a saturated-surface-dry moisture content existed in the coarse aggregates at the predetermined degree of saturation. In this way the difference between the weight of the drained coarse aggregates and the initial weight of the dry coarse aggregates plus water required for saturation could be applied as a correction to the design water content.

About one-half of the required water was then added to the mixer, and all the contents were agitated for approximately three minutes. At this time a neutralized vinsol resin was added with the remaining portion of the mix water for the entrainment of air in the freeze-thaw concretes. No air entrainment was used in the reactivity concretes.

After the final mix water was added, the mixing action was continued for approximately five minutes. Following mixing, the properties of the fresh concrete were determined.

Properties of Fresh Concrete—At least one measurement of the slump was determined for each concrete batch, and the method used conformed to the procedures given by ASTM C143-66.

At least one air content determination was made for each concrete batch in accordance with the procedure given by ASTM C173-66.

Preparation of Test Specimen—For all concrete batches six 6 in. × 12 in. compression test cylinders were prepared according to the procedures set down in ASTM C192-66. In addition, two 3 in. × 3 in. × 16 in. prisms were prepared from each batch of the freeze-thaw series. Also, six 2 in. × 2 in. × 11¼ in. prisms were prepared from each batch of the reactivity series. In the preparation of these prisms, the general procedures given by ASTM C192-66 were followed.

Compression Tests—Three 6 in. × 12 in. cylinders from each batch of concrete were moist cured 28 days and then tested in accordance with the general provisions of ASTM C39-66. The results are reported as the average maximum compressive stress.

Mix Design for Reactivity Concretes—The mix designs for the reactivity concretes conformed to the general provisions set down for the lime popout test of ASTM C330-64T. In this case, a laboratory test was made to determine the loose volume of one coarse aggregate (crushed synthetic aggregate —½ in. to No. 30 grading) combined* with an appropriate amount of natural siliceous fine aggregate. Since all coarse aggregates showed similar sieve analyses, it was assumed that identical proportioning of coarse and fine aggregates by the absolute volume technique would result in similar loose volumes of combined aggregates. In this way the required cement was calculated by a 6:1 ratio of loose volume combined aggregates to loose volume of cement as required by ASTM C330-64T for the proportioning of mixes for shrinkage strain studies.

All mix designs were similar with respect to absolute volumes of coarse and fine aggregates, cement factor and water-cement ratio.

*Proportioned so that combined grading met the requirements of ASTM C330-64T.

Data—The concrete mix designs and measured properties of all concrete batches are given in Tables 4-6 through 4-9.

4.4 Reactivity Test Procedure

In the development of a testing procedure for the reactivity analysis of synthetic aggregates, considerable use was made of the many early studies as noted in Sections 2.1 and 2.2. The resulting test is described as follows in the form of the step by step procedure used. The method basically involves the selective curing of concrete prisms for the purpose of detecting disruptive reactivity.

Coarse Aggregate Preparation—All synthetic aggregates were crushed and screened to a grading range of —½ in. to +No. 30 sieve size. Sieve analysis data for each aggregate tested are given in Table 4-10. Immediately prior to mixing, the crushed aggregates were soaked in water for the length of time required for the saturation of 30 percent of the aggregate's voids.

Fine Aggregate—A naturally occurring siliceous fine aggregate (Hearne-minus No. 4 mesh) meeting ASTM C33-64 (13) was used in all concrete batches.

Concrete Mix Design—The aggregates were proportioned so that the sieve analysis of the combined aggregates met the grading requirements of ASTM C330 (13) for ½ in. to 0 grading range. The cement was proportioned by loose volume as specified in the lime popout test of ASTM C330 (13). The mix water was

TABLE 4-6. FREEZE-THAW CONCRETE BATCH DATA FOR AGGREGATES R

Concrete Parameter	R-2	R-4	R-6	R-1	R-3	R-5	R-comm.
Percent Cement ¹	8.7	8.7	8.6	8.6	8.7	8.7	8.7
Percent Water	17.9	17.8	17.7	17.7	17.8	17.8	15.8
Percent Fine Aggregate	34.3	34.1	34.0	34.0	34.1	34.1	38.2
Percent Coarse Aggregate	33.1	32.9	32.7	32.7	32.9	32.9	33.8
Percent Air ²	6.0	6.5	7.0	7.0	6.5	6.5	4.0
C.F. (sk/c.y.) ³	4.9	4.9	4.9	4.9	4.9	4.9	4.9
W/C (gal/sk) ⁴	7.4	7.4	7.4	7.4	7.4	7.4	6.7
Slump (in.)	4.0	5.0	6.0	5.5	5.5	5.5	3.0
f'c (psi) ⁵ 28 days	3190	3190	2240	2410	2670	2190	4240
Aggregate Voids ⁶ Saturated (Percent)	20	20	20	20	20	15	38.2
Aggregate Soak Time	2h, 15m	4h, 40m	25h, 47m	27h, 47m	156h, 20m	88h, 20m	264h
Batch Quantity (cu. ft.)	1.9	1.9	1.9	1.9	1.9	1.9	4.0

¹By absolute volume.

²By Rolla Meter—ASTM C173-66.

³Cement factor.

⁴Water cement ratio.

⁵Avg. of 3 cyl. (compressive strength).

⁶Based on Absorption and Pressure Pycnometer Tests (Equation 6).

TABLE 4-7. FREEZE-THAW CONCRETE BATCH DATA FOR AGGREGATES Z

Concrete Parameter	Z-2	Z-4	Z-6	Z-1	Z-3	Z-5
Percent Cement	8.7	8.7	8.8	8.6	8.7	8.6
Percent Water	17.8	17.9	17.9	17.6	17.8	17.5
Percent Fine Aggregate	34.2	34.3	34.4	33.8	34.0	33.6
Percent Coarse Aggregate	32.9	33.1	33.1	32.5	32.7	32.3
Percent Air	6.4	6.0	5.8	7.5	6.8	8.0
C.F. (sk/c.y.)	4.9	4.9	4.9	4.9	4.9	4.9
W/C (gal/sk)	7.4	7.4	7.4	7.4	7.4	7.4
Slump (in.)	1.5	1	1	3	3	4
f'c (psi) 28 day	3050	2730	3980	2790	2780	2330
Aggregate Voids Saturated (Percent)	20	20	20	20	20	20
Aggregate Soak Time	19m	22m	32m	1h, 54m	7h, 4m	20h, 8m
Batch Quantity (cu. ft.)	1.9	1.9	1.9	1.9	1.9	1.9

determined on the basis of a water-cement ratio of approximately 9.5 gal/sk.

Concrete Specimens—Three concrete prisms 2 in. × 2 in. × 11¼ in. were prepared from each concrete batch in accordance with the general provisions of ASTM C157 (13) and ASTM 192 (13). The embedment of stainless steel gage points as provided in ASTM C490 (13) furnished the means by which length changes could be determined.

Initial Curing—The specimens were moist cured for 24 hours according to the procedure set forth in ASTM C151-64 (21) (Autoclave Expansion of Portland Cement).

TABLE 4-8. REACTIVITY-SHRINKAGE CONCRETE BATCH DATA FOR AGGREGATES R

Concrete Parameter	R-2	R-4	R-6	R-1	R-3	R-5	R-comm.
Percent Cement	8.3	8.4	8.4	8.6	8.5	8.6	8.7
Percent Water	22.1	22.1	22.1	22.9	22.4	22.8	23.1
Percent Fine Aggregate	28.6	28.8	29.0	28.8	28.3	28.2	28.4
Percent Coarse Aggregate	37.5	37.8	38.0	37.7	37.3	36.9	37.3
Percent Air	3.5	3.0	2.5	2.0	3.5	3.5	2.5
C.F. (sk/c.y.)	4.7	4.8	4.8	4.9	4.8	4.9	4.9
W/C (gal/sk)	9.5	9.5	9.5	9.5	9.5	9.5	9.4
Slump (in.)	1	1	1.5	1	2	2	1
f'c (psi) 28 day	3000	3080	2810	2540	2350	2300	2840
Aggregate Voids Saturated (Percent)	30	30	30	30	30	30	30
Aggregate Soak Time	3h, 30m	3h	17h, 54m	36h, 12m	90h, 48m	87h, 30m	25h, 25m
Batch Quantity (cu. ft.)	2.3	2.3	2.2	2.3	2.3	2.3	2.3

TABLE 4-9. REACTIVITY-SHRINKAGE CONCRETE BATCH DATA FOR AGGREGATES Z

Concrete Parameter	Z-2	Z-4	Z-6	Z-1	Z-3	Z-5
Percent Cement	8.9	8.8	8.7	9.0	9.1	8.8
Percent Water	23.1	23.7	23.1	23.9	24.0	23.4
Percent Fine Aggregate	28.3	28.2	28.0	27.3	27.7	27.6
Percent Coarse Aggregate	37.2	36.9	36.7	35.8	36.2	36.2
Percent Air	2.5	2.4	3.5	4.0	3.0	4.0
C.F. (sk/c.y.)	5.0	5.0	4.9	5.1	5.1	5.0
W/C (gal/sk)	9.4	9.6	9.5	9.5	9.5	9.5
Slump (in.)	1	1.5	1	0.5	0.5	1
f'c (psi) 28-day	3470	3100	3540	2990	3200	2602
Aggregate Voids Saturated (Percent)	30	30	30	30	30	30
Aggregate Soak Time	17m	38m	10m	1h, 4h	6h, 58m	18h, 45m
Batch Quantity (cu. ft.)	2.3	2.3	2.3	2.4	2.3	2.4

Autoclaving—After the initial curing period, length measurements were made and then the specimens were autoclaved according to the procedures set forth in ASTM C151-64. Following autoclave testing, the specimen lengths were again measured and the specimens were examined for evidence of cracks, popouts and chemical reactions. The apparatus used for determining the change in length of the specimens is the comparator

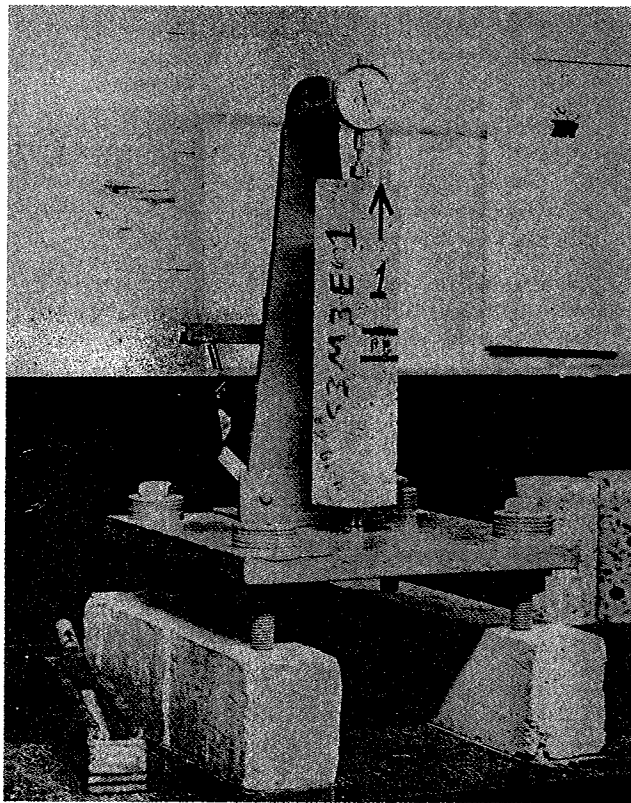


Figure 4-6. Comparator used in determining changes in length of reactivity and shrinkage specimens.

shown in Figure 4-6. This device conforms to the specifications as given by ASTM C490-65T (21).

Long Time Reactivity Curing—After the final length measurement following autoclaving, the specimens were placed in a curing tank in which water was continually circulated and held at a temperature of $104^{\circ}\text{F} \pm 2^{\circ}\text{F}$. The tank is 6 ft long, 31 in. wide and $5\frac{1}{2}$ in. high (see Figure 4-7). The specimens were positioned horizontally on a rack so that the surface of the water was approximately one inch below the bottoms of the specimen (see Figure 4-8).

Test Measurements During Long Time Curing—Measurements of specimen length were made on the approximate schedule of the 1st, 2nd, and 4th day of reactivity curing followed by one reading in each of the 2nd, 3rd and 4th weeks of curing. Subsequent readings were made monthly.

Because of the temperature differential between the curing tank and the testing laboratory, specimens were removed for readings one at a time, and the measurements were made as rapidly as possible.

4.5 Freeze-Thaw Test Procedure

Because little or no previous research has been conducted on the effects of thermal transformation on the freeze-thaw durability of synthetic aggregate concrete, an experimental program was devised with the purpose of emphasizing these effects. From the discussions of the preceding sections, as well as Report 81-6 (1), it is logical to conclude that highly saturated aggregates are likely to cause freeze-thaw failure of concrete regardless of their structural quality. Therefore, in a study in which an increased sensitivity to structural quality is desired, all aggregates should be batched in concrete at a low degree of saturation. In Report 81-6, studies on this subject have shown that a wide variety of synthetic aggregates have produced durable concretes* when batched at 25 percent or less saturation. It was therefore concluded that if all aggregates of the current study were presaturated to a 20 percent degree of saturation, the effect of degree of thermal transformation upon the freeze-thaw durability of concrete would be more effectively exhibited.

*Tested according to ASTM C290 (freeze-thaw test for concrete durability).

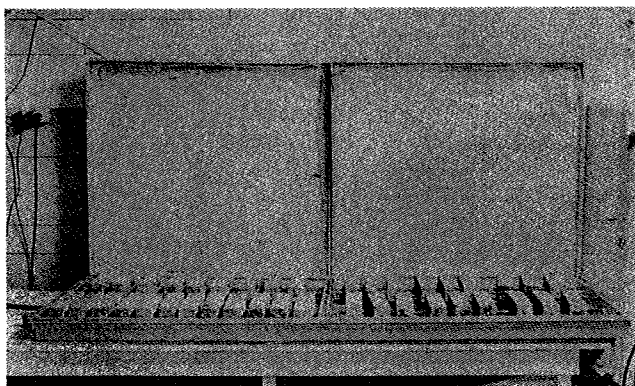


Figure 4-7. Curing tank used in reactivity testing.

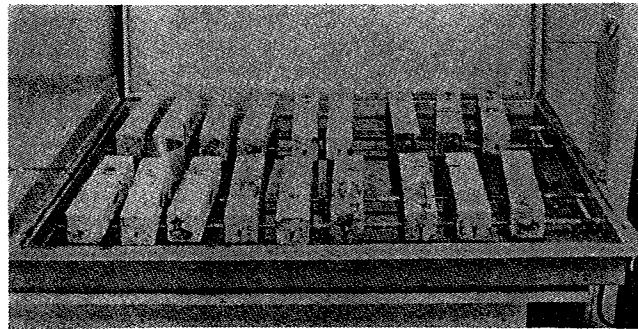


Figure 4-8. Reactivity specimens during long time moist curing.

Obtaining a Given Degree of Saturation—In devising a technique to presaturate all twelve research kiln produced aggregates, equation 2 of Section 4.1 was used to calculate the percent absorption corresponding to 20 percent saturation. Once the absorption value was known for each aggregate, the time absorption data could be used to determine the length of soaking time required for the desired saturation.

Concrete Batch Procedures—All aggregates* (see Table 4-11 for coarse aggregate sieve analyses) were soaked for the proper time periods and then immediately batched according to the procedures set down in 4.1. An effort was made to hold constant as many batch parameters as possible (water-cement ratio, cement factor, air entrainment, slump, and mixing procedures).

Freeze-Thaw Testing—Two** $3\text{ in.} \times 3\text{ in.} \times 16\text{ in.}$ concrete specimens for each aggregate type were made, cured and tested according to the procedures outlined in the standard ASTM test for rapid freezing and thawing of concrete in water (ASTM C290-67).

*No freeze-thaw batch of commercial aggregate R was necessary, since data from a previous study existed (1).
**The use of two specimens instead of the standard three was necessitated by equipment availability. However, very good agreement in test results indicated this modification did not significantly limit the conclusions reached in this study.

TABLE 4-10. SIEVE ANALYSES FOR CRUSHED AGGREGATES USED IN REACTIVITY-SHRINKAGE CONCRETES

Aggregate	Cumulative Percent Retained on Sieve Indicated				
	% in.	No. 4	No. 8	No. 16	No. 30
R-1	15.0	55.9	82.2	98.2	99.9
R-2	9.2	44.5	75.0	96.7	99.8
R-3	10.9	50.2	79.2	97.0	99.6
R-4	14.4	55.8	84.5	98.6	99.9
R-5	10.3	47.9	76.7	97.1	99.9
R-6	14.1	51.6	80.5	98.5	99.9
R-comm.	13.0	57.9	85.0	98.6	99.9
Z-1	7.7	35.9	66.1	95.5	99.8
Z-2	8.1	36.8	70.0	96.6	99.8
Z-3	15.7	58.6	84.7	98.0	99.8
Z-4	8.3	40.0	74.5	97.5	99.9
Z-5	12.4	48.1	76.8	97.6	99.8
Z-6	6.2	34.5	69.4	97.5	99.8

TABLE 4-11. SIEVE ANALYSES FOR COARSE AGGREGATES USED IN FREEZE-THAW CONCRETES

Aggregate	Cumulative Percent Retained on Sieve Indicated				
	¾ in.	½ in.	¾ in.	No. 4	No. 8
R-1	9.7	40.8	61.0	93.5	100
R-2	5.7	34.2	54.8	89.8	100
R-3	7.8	37.2	55.4	89.2	100
R-4	7.5	40.0	57.1	91.2	100
R-5	13.5	40.7	56.8	88.5	100
R-6	6.8	36.4	54.3	88.7	100
R-comm.	1.7	31.1	55.5	94.2	99.2
Z-1	8.8	33.1	47.7	90.0	100
Z-2	7.8	28.6	52.1	91.8	100
Z-3	13.8	41.6	60.9	91.4	100
Z-4	9.5	34.3	54.7	90.3	100
Z-5	17.6	44.4	64.5	94.3	100
Z-6	10.7	32.6	52.8	90.7	100

The measurement of the concrete deterioration was taken as the relative squared flexural frequency of vibration which is directly related to the dynamic modulus of elasticity as determined by the oscilloscope method decrease in the modulus of elasticity is an indication of deterioration, then a related measure of deterioration is given by

$$P_c = \frac{n_2^2}{n_1^2} \times 100 \dots\dots\dots 4$$

where

P_c = relative dynamic modulus of elasticity (percent after c cycles of freeze-thaw testing)

n_1 = fundamental transverse frequency at zero cycles of testing

n_2 = fundamental transverse frequency at c cycles of testing.

4.6 Shrinkage Test Procedure

Preparation and Testing of Specimens—The general procedures outlined in ASTM C330-64T were followed in preparing three 2 in. × 2 in. × 11¼ in. concrete prisms for the shrinkage study of each concrete type. These specimens were molded from a portion of the same concrete batches used for making the reactivity specimens. Therefore the mix procedures, designs and properties are the same as those given for the reactivity concretes (see Section 4.1).

At an age of 7 days, the concretes were removed from moist curing and placed in a controlled temperature-humidity room as required by ASTM C330-64T. The requirements of 73.4 ± 2°F and 50 ± 2 percent relative humidity were met in general, although some humidity fluctuations were encountered. The comparator pictured in Figure 4-6 was used to take periodic length measurements for the calculations of strains.

The shrinkage data are given in Tables 4-12 and 4-13.

TABLE 4-12. CONCRETE SHRINKAGE STRAIN DATA FOR AGGREGATES R

R-1		R-3		R-5		R-comm.	
Days Drying	Strain*	Days Drying	Strain*	Days Drying	Strain*	Days Drying	Strain*
0.9	133	1.0	57	1.1	73	1.1	10
2.9	70	2.0	93	2.0	93	4.1	120
6.7	150	2.9	130	6	213	4.9	137
13.7	273	7	223	10	287	11.9	263
21	320	11	293	17	427	19	310
28	413	18	390	21	470	26	423
38	450	22	447	28	507	36	450
44	450	29	467	35	477	43	477
55	450	36	440	42	560	54	433
62	430	43	517	52	557	61	443
77	480	53	510	59	550	76	507
		60	517	70	545		
		78	487	77	527		
		92	520	92	583		
R-2		R-4		R-6			
2	15	1	73	4	173		
5.1	110	2.1	93	5	193		
5.8	130	4.0	123	8	240		
12.8	255	9.2	253	12	387		
20	290	11	290	15	417		
27	390	15	253	19	463		
37	400	22	403	26	500		
44	425	29	393	33	483		
55	405	36	470	40	563		
62	395	46	480	50	560		
77	460	53	513	57	563		
		64	477	68	543		
		71	477	75	537		
		85	493	90	594		

*All strains in units of 10⁻⁶ in./in.

TABLE 4-13. CONCRETE SHRINKAGE STRAIN DATA FOR AGGREGATES Z

Z-1		Z-3		Z-5	
Days Drying	Strain*	Days Drying	Strain*	Days Drying	Strain*
0.5	63	1.0	57	0.8	70
1.2	90	3.1	116	4.9	190
2.3	93	3.9	133	8.8	250
4.2	150	10.8	263	16	387
7	193	18	320	20	477
14	340	25	426	27	524
18	364	35	480	34	507
25	490	42	473	41	595
36	533	53	477	51	595
43	494	60	470	58	613
50	600	75	540	69	580
60	561			76	564
78	537			91	640
85	544				
100	597				

Z-2		Z-4		Z-6	
Days Drying	Strain*	Days Drying	Strain*	Days Drying	Strain*
0.7	63	0.3	60	1	43
3.8	110	1.1	87	2	70
4.8	130	2.1	117	4.1	63
10.8	240	5.2	150	7.9	163
14.8	270	12	267	14.9	253
22	353	16	283	22	290
33	427	27	367	39	443
40	400	34	437	46	500
47	490	41	410	57	437
57	484	48	500	64	437
64	507	58	497	79	507
75	483	65	527		
82	483	76	490		
97	540	83	477		
		98	647		

*All strains in units of 10^{-6} in./in.

4.7 References

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