

# SYNTHETIC AGGREGATES FROM CLAY AND SHALE: RECOMMENDED CRITERIA FOR EVALUATION

W. B. Ledbetter, Texas Transportation Institute, Texas A&M University

The transformation of a clay mineral into an inert, stable, amorphous silicate under the combined action of heat and time is an extremely complex phenomenon that occurs under nonequilibrium conditions. Essentially what occurs is, first, the loss of free and absorbed water and, then, the loss of hydroxylated water coupled with the decomposition of minerals such as calcite, pyrite, and dolomite. These chemical transformations take time; therefore, depending on the amount of heat and size of the particle, different degrees of transformation can occur. An interesting special phenomenon is the process of "bloating" where some clays "puff up" slightly. Examination under the microscope of a properly bloated clay reveals a sponge-like structure where the holes, or "blebs" as they are termed, are numerous but small. The dry unit weight of this aggregate (coarse sizes) is often in the range of 35 to 45 lb/ft<sup>3</sup>. With such a small "bleb" structure, the resultant aggregate can be strong and durable. To fully develop the criteria for performance, or durability, the research was considered from four aspects: physical performance, chemical performance, mechanical performance, and volume change performance. The following findings relate to the limitations imposed on this study, and further generalizations may not be warranted: Sound, durable, high-quality highways and bridges can be constructed with synthetic aggregates, provided the aggregates meet certain requirements; not all functional uses of synthetic aggregates require aggregate of the same quality; and laboratory evaluations can, with reasonable assurance, predict field performance of synthetic aggregates.

•SYNTHETIC aggregates made from clay or shale are not new. Brick or ceramic tile is a "synthetic" rock in that it is man-made, and broken bricks, made from clay, were used by the Romans many centuries ago as an aggregate in concrete made with pozzolanic lime mortar (1). For the purposes of this report, synthetic aggregates are defined as those aggregates made by the thermal transformation of agglomerations of clays or shales into essentially amorphous silicates.

With the development of the rotary kiln process in 1917 by Hayde (2), the economical production of lightweight synthetic aggregates became a reality, and today a review of the technical literature reveals the almost universal use of lightweight synthetic aggregates in concrete. Bridges and buildings have been built in the United States (3), England, Germany, France, Russia, Japan, and Australia.

Concurrent with the rapid rise in the use of lightweight synthetic aggregates has been the depletion of available natural aggregates in many portions of the United States (4). For these and other reasons, the use of synthetic aggregates has continued to increase, and demand is expected to grow.

In 1964 the Texas Highway Department, realizing the increasing need for synthetic aggregates in all types of highway construction, in cooperation with the Federal Highway Administration, initiated a 6-year study on synthetic aggregate research. The objective of that study was to develop a recommended synthetic aggregate classification system that would identify quality synthetic aggregates for use in highway construction. In other words, the study was aimed at developing accelerated laboratory performance

criteria that, if met, would ensure that any qualifying synthetic aggregate made from clay or shale by a thermal process would perform satisfactorily throughout the service life of the highway system. This paper summarizes the results of that study.

#### TRANSFORMATION PROCESS

The transformation of a clay mineral into an inert, stable, amorphous silicate under the combined action of heat and time is an extremely complex phenomenon that occurs under nonequilibrium conditions. Essentially what occurs is, first, the loss of free and absorbed water and, then, the loss of hydroxylated water coupled with the decomposition of minerals such as calcite, pyrite, and dolomite. These chemical transformations take time; therefore, depending on the amount of heat and size of the particle, different degrees of transformation can occur.

An interesting special phenomenon is the process of "bloating" where some clays "puff up" slightly. Examination under the microscope of a properly bloated clay reveals a sponge-like structure (Fig. 1) where the holes, or "blebs" as they are termed, are numerous, but small. The dry unit weight of this aggregate (coarse sizes) is often in the range of 35 to 45 lb/ft<sup>3</sup>. With such a small "bleb" structure the resultant aggregate can be strong and durable. This bloating action is not fully understood, but it is surmised that two conditions must occur almost simultaneously. First the clay must reach the proper pyroplastic condition to be able to expand without rupturing (not too brittle) or subsequently collapsing (not too fluid). Second, the clay must generate the bloating force in the form of gas evolution at just the right time and in just the right amount. Needless to say, these two conditions are restrictive, and thus there are many clays that either do not bloat at all or do not bloat properly.

The state of the art of the production of these bloated aggregates, termed structural lightweight aggregates, is well advanced, and standard specifications have been developed (such as ASTM Designation C 330). There are two methods of burning in wide use in the United States. One is the rotary kiln method where the clay particles are fed into the upper end of a slowly rotating, inclined tube (kiln) and travel slowly to the lower burner end where they reach temperatures as high as 2200 F. The clays are in the kiln for 30 min to an hour, during which time the transformation process is essentially completed. The other is the sintering method in which raw clays are carried on a moving grate through an ignition hood where the clays are heated rapidly to the desired temperature and then allowed to cool slowly as the grate continues to move. Total firing time may be as short as 10 min.

In 1966 there were 65 rotary kiln plants and 18 sintering plants in the United States and Canada. This number has increased significantly since that time.

#### DEVELOPMENT OF EVALUATION CRITERIA

##### Strength and Performance

Based on the wealth of research information, it became evident that synthetic aggregates could be made sufficiently strong for almost any anticipated use (3). Also it has been proved that some synthetic aggregates could satisfactorily perform throughout their service life (3). Furthermore the end-product evaluations for strength are generally well established [for example, compressive strength for portland cement concrete (PCC) and stability values for asphaltic concrete]. Thus the main thrust of this research was the development of performance criteria (as distinct from strength) to evaluate new aggregates with no performance records.

To fully develop the criteria for performance, or durability, the research was considered from four aspects: physical performance, chemical performance, mechanical performance, and volume change performance. These are defined in Table 1. It should be emphasized that performance involves aspects other than these four and further that these four are not independent of each other. But each aspect was treated somewhat independently in the conduct of this research.

### Physical Performance

The physical performance of lightweight aggregates for PCC was tied to the generally accepted rapid freeze-thaw test for concrete immersed in water (ASTM Designation C 666-Procedure A). The rationale behind this test was the contention that a high-quality, air-entrained concrete containing a synthetic aggregate would only show significant distress in 300 cycles of freeze-thaw if the synthetic aggregates were not physically durable. Results of this phase of the investigation have been reported (5, 6). Essentially it was found that, due to the relatively high saturation capabilities of many synthetic lightweight aggregates (Fig. 2), almost any aggregate can be rendered nondurable if it enters the PCC mix in a relatively saturated condition. Conversely almost any aggregate can be made durable if it is mixed in a relatively dry condition. The volume term "saturation" is used here rather than the more commonly used weight term "absorption." Saturation is the volume of voids filled with water divided by the total volume of voids available. Because many lightweight aggregates contain up to 50 percent voids, a considerable quantity of water can be absorbed into the aggregate.

Laboratory tests have shown that, when the degree of saturation is kept below about 25 percent (by volume of voids), the concrete exhibits good resistance to deterioration from freezing and thawing (Fig. 3) (6). It was also found that some synthetic aggregates reached this critical saturation after less than 30 min of immersion in water, while others had to be immersed several days before becoming critically saturated (Fig. 2). The latter aggregates are often termed "coated" aggregates because they possess a denser surface coating, or rind, that inhibits absorption. Because the degree of saturation is very difficult and time-consuming to determine in the field, it was recommended that a maximum rate of saturation be imposed on the highest quality aggregate to be used in concrete subjected to a freeze-thaw environment and a limit of 15 percent saturation after 100 min of immersion in water was selected<sup>1</sup>.

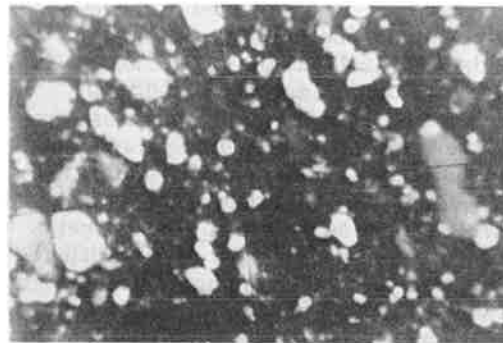
With the increasing emphasis on highway surfaces with higher and longer lasting skid resistance, the use of lightweight synthetic aggregate in asphaltic concrete and surface treatments has been shown to be an economical way to provide excellent, long-lasting, skid-resistant surfaces (7, 8). As with any new material, the physical performance of these aggregates must be demonstrated. It is reasonable to expect that a material that readily absorbs water might not perform satisfactorily, if sufficiently saturated and subjected to freezing and thawing. Gallaway and Harper developed an aggregate freeze-thaw test that approximates the nature of such exposure in the field (9). Based on these results, maximum permissible weight losses have been established for synthetic aggregates.

### Chemical Performance

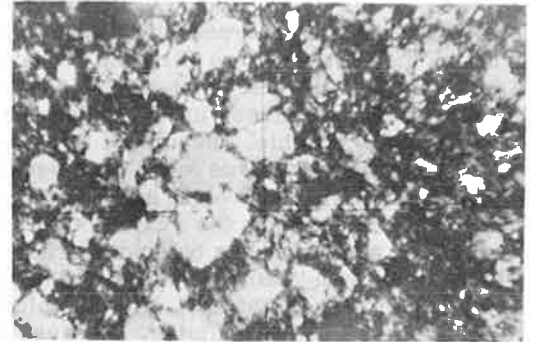
The excellent record of many structural lightweight aggregates in concrete (3) attests to their chemical stability. Further, bricks made from clay have lasted for centuries. However, it is a fact that synthetic aggregates have been transformed from raw clay or shale. What if this transformation is incomplete? Some accelerated laboratory testing was needed to indicate the completeness of this thermal transformation. Such a test, the pressure slaking test, was developed and reported (10). The test involves the cooking of the aggregates underwater in a common pressure cooker and then subjecting them to severe agitation in water. The agitation in water disperses any rehydrated material, and it also produces some abrasion loss. The total loss in weight through a specified sieve is a measure of the degree of transformation. Pressure-slaking losses were shown to be a function of firing temperature, and the results were compared with losses of commercially produced aggregates (both from plants and from in-service locations) (Fig. 4).

<sup>1</sup>The original manuscript of this paper included Appendix A, Texas Highway Department Test Method 433-A; Appendix B, Texas Highway Department Test Method 109-E(Part I); Appendix C, Texas Highway Department Test Method 432-A; and Appendix D, Texas Highway Department Test Method 431-A. The appendixes are available in Xerox form at cost of reproduction and handling from the Highway Research Board. When ordering, refer to XS-44, Highway Research Record 430.

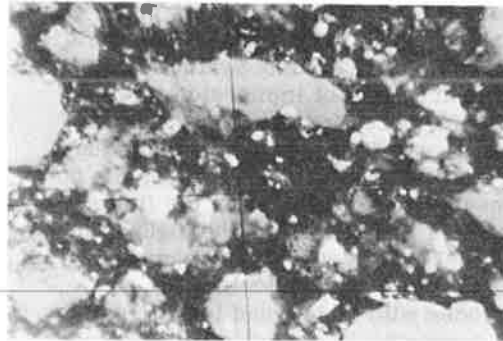
Figure 1. Photomicrographs of sample RGR.



1800 °F



2000 °F



2200 °F

Nichol prisms partly crossed black areas indicate amorphous glass matrix; gray areas indicate pores, and white areas are crystalline minerals (52X).

Table 1. Performance concepts.

Type of Performance	Definition
Physical	The resistance of aggregate to repeated stressing, either from internal sources such as freezing and thawing or from external sources such as fatiguing from traffic loading.
Chemical	The resistance of aggregate to the various chemical reactions occurring on, or in, the aggregate during its service life. An example would be any rehydration of synthetic aggregate back to a clay or shale.
Mechanical	The resistance of aggregate to the abrasive wear of traffic on its exposed surface.
Volume change	The resistance of aggregate to detrimental volume changes of PCC from shrinkage and creep.

Figure 2. Degree of aggregate saturation versus immersion time in water.

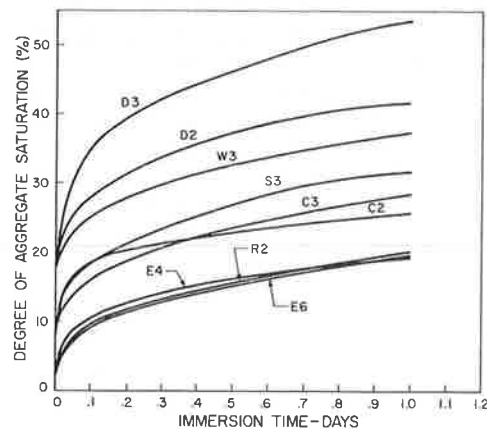
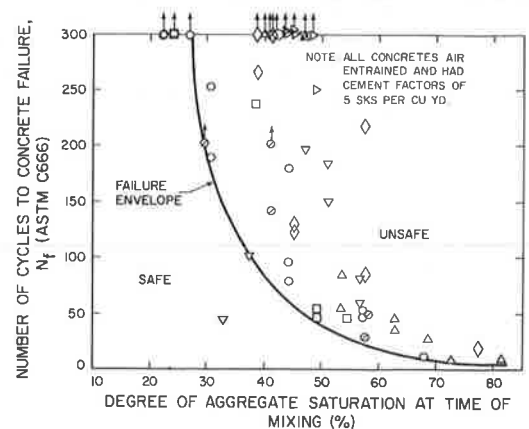


Figure 3. Concrete freeze-thaw failure envelope (6).



The other major adverse chemical reaction that was expected was the alkali-aggregate reactivity in PCC. Because the transformed clay mineral is composed of silicates, some of these might react with PCC. Laboratory investigations of many aggregates did reveal a very few adverse reactions. However, no suitable aggregate test was found that would isolate reactive synthetic aggregates. Thus, a more time-consuming concrete autoclave expansion test, patterned after ASTM Designation C 151, was recommended as a source qualification test. This phase of the study is covered elsewhere (11).

#### Mechanical Performance

The ability of any highway surface to satisfactorily resist abrasion and wear from traffic has long been of concern to highway engineers and even more so in recent years as prolonged high skid resistance must now be provided. In this study several abrasion tests were evaluated, including the standard Los Angeles abrasion test (ASTM Designation C 131) and the concrete abrasion resistance test (ASTM Designation C 418). In addition an aggregate sandblast test was developed to measure a synthetic aggregate's resistance to abrasion (12). The overall result was that, although the standard Los Angeles abrasion test was open to considerable doubt as to its applicability, no suitable alternative test was found. Therefore, the Los Angeles abrasion test (ASTM Designation C 131) was recommended as a mechanical performance criterion.

#### Volume Change Performance

Volume changes in PCC from shrinkage and creep can be extremely detrimental. The role of aggregate, as an inert filler, has been to reduce the volume change from that experienced with cement paste alone (1). Lightweight synthetic aggregates, because of their water-absorbing characteristics, can contribute to the concrete volume change phenomena, both constructively and destructively (3). In this phase of the investigation the shrinkage-cracking characteristics of lightweight aggregate concrete were investigated and reported (13). Although the aggregates used were found to influence the amount of shrinkage experienced, no new tests were found to be needed to identify or control shrinkage of lightweight concrete. ASTM Designation C 330 specifies a concrete shrinkage that, in most cases, has been found to be satisfactory.

#### Evaluation Criteria

Based on the four performance concepts discussed, a classification system for synthetic coarse aggregates was developed (Table 2). The table divides the material into two classes: Class I is subdivided into four groups (A, B, C, and D) of descending physical requirements, and class II is subdivided into three groups of descending physical requirements (A, B, and C).

This classification system is not intended to replace existing requirements for high-quality aggregates in highway construction; rather it is offered as a supplement to existing aggregate requirements. For example, the requirements for clean, sound, durable aggregates of specified gradations (depending on their use) are not mentioned here because they are adequately covered in current highway department specifications.

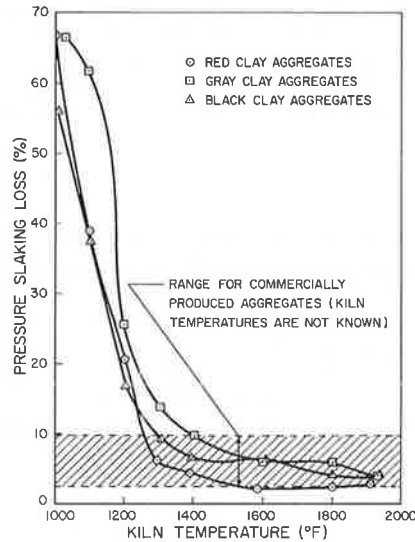
A functional grouping of coarse synthetic aggregates is given in Table 3. In this table the recommended permissible coarse-aggregate group defined in Table 2 is shown for each highway function, from surface treatments to base materials.

### CONCLUSIONS

It should be emphasized that the findings of this paper relate to the limitations imposed on this study, and further generalizations may not be warranted. Conclusions are summarized as follows:

1. Sound, durable, high-quality highways and bridges can be constructed utilizing synthetic aggregates, provided the aggregates meet certain requirements.
2. Not all functional uses of synthetic aggregates require the same quality aggregate. Thus, any classification system must recognize, and allow, synthetic aggregates of differing qualities for different uses.

**Figure 4. Pressure slaking loss versus maximum kiln temperature (10).**



**Table 2. Classification system for synthetic coarse aggregates.**

Class	Group	Dry Loose Unit Weight (lb/ft <sup>3</sup> ) <sup>a</sup>		Maximum 100-Min Saturation (percent) <sup>f</sup>	Maximum Aggregate Freeze-Thaw Loss (percent) <sup>d</sup>	Maximum Pressure Slaking Value (percent) <sup>e</sup>	Maximum Los Angeles Abrasion Loss (percent) <sup>g</sup>
		Maximum	Minimum <sup>b</sup>				
I (bloomed)	A	55	40	15	7	6	30
	B	55	35	15	7	6	35
	C	55	35	20	15	6	40
	D	55	35	—	—	10	45
II (nonbloomed)	A	—	55	—	7	6	35
	B	—	55	—	15	6	40
	C	—	55	—	—	10	45

<sup>a</sup>Tested in accordance with ASTM Designation C 330.

<sup>b</sup>This minimum should not preclude the experimental use of a lighter weight aggregate from any new source or upgraded existing source.

<sup>c</sup>Tested in accordance with Texas Test Method 433-A.

<sup>d</sup>Tested in accordance with Texas Test Method 432-A.

<sup>e</sup>Tested in accordance with Texas Test Method 431-A.

<sup>f</sup>Tested in accordance with ASTM Designation C 131.

**Table 3. Functional grouping of synthetic coarse aggregates.**

Function	Permissible Aggregate Group
Surface treatments <sup>a</sup>	IA
Asphaltic concrete surfaces <sup>b</sup>	IB, IA, IIA
Asphaltic concrete bases	IA, B, C, D, IIA, B, C
Exposed lightweight PCC structures <sup>c</sup>	IA, B
PCC pavements <sup>c</sup>	IA, B
Unexposed PCC bases <sup>c</sup>	IA, B, C, IIA, B
Flexible base materials	IA, B, C, D, IIA, B, C

<sup>a</sup>The aggregate should be kept dry during construction.

<sup>b</sup>The 100-min saturation requirement can be waived.

<sup>c</sup>A maximum concrete autoclave expansion (ASTM Designation C 151 modified) of 1,500  $\mu\text{in./in.}$  should be required.

3. Laboratory evaluations can, with reasonable assurance, predict field performance of synthetic aggregates. Thus new synthetic aggregate sources, if they satisfactorily pass certain laboratory requirements, can reasonably be expected to perform satisfactorily in the field.

#### REFERENCES

1. Taylor, W. H. Concrete Technology and Practice, 3rd Ed. Angus and Robertson Ltd., London, 1969, 667 pp.
2. Shideler, J. J. Manufacture and Use of Lightweight Aggregates for Structural Concrete. Portland Cement Assn., Skokie, Development Dept. Bull. D40, Jan. 1961, 18 pp.
3. Guide for Structural Lightweight Aggregate Concrete. Proc., Jour. ACI, Vol. 64, No. 8, Aug. 1967, pp. 433-469.
4. Witczak, M. W., Lovell, C. W., Jr., and Yoder, E. J. A Generalized Investigation of the Potential Availability of Aggregate by Regional Geomorphic Units Within the Conterminous 48 States. Highway Research Record 353, 1971, pp. 31-42.
5. Buth, E., and Ledbetter, W. B. The Importance of Moisture Absorption Characteristics of Lightweight Coarse Aggregate. Highway Research Record 226, 1968, pp. 35-49.
6. Ledbetter, W. B., and Buth, E. TTI Study Explores Durability of Structural Lightweight Concrete for Buildings. Highway Research News 41, Autumn 1970, pp. 31-38.
7. Gallaway, B. M., and Hargett, E. R. Blending Lightweight Aggregates With Natural Aggregates for the Production of Bituminous Concrete. Highway Research Record 273, 1969, pp. 42-52.
8. Gallaway, B. M., and Epps, J. A. Tailor-Made Aggregates for Prolonged High Skid Resistance on Modern Highways. In An Interamerican Approach for the Seventies, Materials Technology—I. 2nd Interamerican Conf. on Materials Technology, ASME, New York, Aug. 1970, pp. 90-99.
9. Gallaway, B. M., and Harper, W. J. Laboratory and Field Evaluation of Lightweight Aggregates as Coverstone for Seal Coats and Surface Treatments. Highway Research Record 150, 1966, pp. 25-81.
10. Moore, W. M. Fired-Clay Aggregates for Use in Flexible Bases. Highway Research Record 307, 1970, pp. 33-43.
11. Martin, J. R., Ledbetter, W. B., Ahmad, H., and Britton, S. C. Nonbloated Burned Clay Aggregate. Pending publication by ASTM.
12. Houston, J. T. A Sandblast Abrasion Test for Synthetic Aggregate Evaluation. Materials Research and Standards, MTRSA, Vol. 10, No. 8, pp. 17-20, 42.
13. McKeen, R. G., and Ledbetter, W. B. Shrinkage-Cracking Characteristics of Structural Lightweight Concrete. Proc., Jour. ACI, Vol. 67, No. 10, Oct. 1970, pp. 769-777.