HIGHWAY RESEARCH BOARD Bulletin 143

Freeze-Thaw Durabitity of Aggregate in Concrete

National Academy of Sciences-

National Research Council

publication 435

HIGHWAY RESEARCH BOARD

Officers and Members of the Executive Committee 1956

OFFICERS

K. B. WOODS, *Chairman* **REX M. WHITTON**, *Vice Chairman* FRED BURGGRAF, *Director* ELME R M . WARD , *Assistant Director*

Executive Committee

C. D. CURTISS, Commissioner, Bureau of Public Roads

A . E. JOHNSON, *Executive Secietaiy, American Association of State Highway Officials*

Louis Jordan, *Executive Secretary, Division of Engineering and Industrial Research*, *National Research Council*

R. H. BALDOCK, State Highway Engineer, Oiegon State Highway Commission

PYKE JOHNSON, *Consultant, Automotive Safety Foundation*

G DONALD KENNEDY, *President, Portland Cement Association*

O. L . KiPP, *Consultant, Minnesota Depai tment of Highways*

BURTON W. MARSH, Director, Safety and Traffic Engineering Depaitment, American *Automobile Association*

C. H . SCHOLER, *Head, Applied Mechanics Department, Kansas State College*

REX M . WHITTON, *Chief Engineer, Mtssoui i State Highway Department*

K . B. WOODS, *Head, School of Civil Engineering and Directot, Joint Highway Research Project, Purdue University*

Editorial Staff

FRED BURGGRAF ELMER M. WARD HERBERT P. ORLAND **2101 Constitution Avenue Washington 25, D. C.**

> **The opinions and conclusions expressed in this publication are those of the authors and not necessarily those of the Highway Research Board.**

HIGHWAY RESEARCH BOARD BuUetin 143

Freeze-Thaw Durability of Aggregate in Concrete

PRESENTED AT THE Thirty-Fifth Annual Meeting January 17-20, 1956

1956 Washington, D. C.

Department of Materials and Construction

R. R. Litehiser, Chairman Engineer of Tests, Testing & Research Laboratories Ohio Department of Highways

GENERAL MATERIALS DIVISION

J . H. Swanberg, Chairman Engineer of Materials and Research Minnesota Department of Highways

COMMITTEE ON MINERAL AGGREGATES

Warren J . Worth, Chairman Engineer of Tests Board of Wayne County Road Commissioners, Detroit

- H. F. Clemmer, Engineer of Materials and Standards, D. C. Engineer **Department**
- **J. E . Gray, Field Engineer, National Crushed Stone Association, Washington, D. C.**
- **Fred Hubbard, Director of Research, National Slag Association, Youngstown, Ohio**
- **F. N. Hveem, Materials and Research Engineer, California Division of Highways**

Professor F. B. Legg, Jr. , Assistant Professor of Engineering Materials, University of Michigan

- **William Lerch, Administrative Assistant, Portland Cement Association**
- **D. W. Lewis, Chief Engineer, National Slag Association, Washington, D. C.**
- **J. F. McLaughlin, Research Engineer, Joint Highway Research Project, Purdue University**
- **Bert Myers, Materials Engineer, Iowa State Highway Commission**
- **D. H. Sawyer, Research Engineer, Kentucky Department of Highways Stanton Walker, Director of Engineering, National Sand and Gravel**

Association, Washington, D. C.

- **E. A. Whitehurst, Director, Tennessee Highway Research Program, University of Tennessee,**
- **D. O. Woolf, Bureau of Public Roads**

Contents

 \cdot

 \mathbf{I}

Freeze-Thaw Durability of Michigan Concrete Coarse Aggregates

F. E. LEGG, JR. , Assistant Laboratory Supervisor Michigan State Highway Department Laboratory

> The Michigan Highway Department is currently called upon to approve, each year, coarse aggregate for approximately one million cubic yards of concrete. These aggregates are furnished from deposits ranging in size from roadside pits up to large well-established commercial sources and are distributed over an area exceeding 600 miles between extremities. There is every indication that the rate of aggregate consumption will increase with the accelerated highway construction program. This will surely require evaluation of the freeze-thaw durability, both of deposits now considered of marginal quality and of entirely new sources as proven aggregate reserves become depleted.

The present paper presents laboratory freeze-thaw results as well as field observations on the major Michigan aggregate sources and is considered a first step in formulation of a policy regarding acceptance of future aggregates of unknown service behavior.

Brief description is made of automatic freeze-thaw equipment wherein considerable cost was saved by utilizing an existing large cold room as the source of freezing medium. Six cycles per day are obtained using either rapid freezing in air and thawing in water, or rapid freezing in water and thawing in water. Both types of cycle can be obtained simultaneously if desired. Every effort has been made to provide a rugged trouble-free design. The equipment has now operated continuously for two years with a minimum of attention.

Laboratory evaluation of coarse aggregate durability is achieved by following a rigidly standardized procedure wherein the aggregates are graded and placed in air-entrained concrete in a vacuum saturated condition. The ASTM rapid cycle of freezing in air to 0 F and thawing in 40 F water is used. Failure is considered to have occurred when tne sonic modulus of 3- by 4- by 16-in. specimens has decreased 30 percent. Durability factors at 300 cycles of freezing and thawing appear to adequately describe the behavior of the aggregates. Data are presented giving durability factors of the major aggregate sources, and also from three plants using heavy media methods of gravel improvement. Results of a special series of tests are presented wherein the effect of chert stil l remaining in the heavy media improved gravel is evaluated. The results indicate that this high gravity chert is far less harmful than low gravity chert. Correlation with the magnesium sulfate soundness test was found to be doubtful.

The laboratory durability results correlate reasonably well with known service behavior and it now seems possible to provide the highway administrator with reliable factual data for proper decision.

THE Michigan Highway Department is currently called upon to approve coarse aggregates for roughly one million cubic yards of concrete annually. These aggregates are incorporated in concrete whose use and importance range from a few yards for a small culvert, up to thousands of cubic yards necessary for paving an expressway. In addition, high quality aggregates are required for the many bridges and grade separation structures incident to an increasing construction program. The aggregate sources likewise range from small roadside pits producing from glacial deposits up to large wellestablished gravel plants. The upper Great Lakes region also has limestone sources which furnish coarse aggregate crushed from ledge rock. This is usually brought down by boat to the Detroit area on the eastern side of the state or to po'ts on the western shore. In addition, some limestone aggregates are furnished Michigan from nearby

deposits in Ohio, Indiana, and Illinois. The geography of the state itself poses a problem in coordination since the deposits are distributed over an area exceeding 600 miles between extremities.

The southern part of the state is more densely populated with consequent greater mileage of highways. Simultaneous with this is the observation that this portion experiences more cycles of freezing and thawing in the winter in the presence of moisture from melting snow, sleet, or rain and provides a challenging proving ground for genuinely frost resistant concrete. Chloride salts for ice removal are also used in abundance.

Selection of concrete aggregates for this severe exposure has been accomplished in the past by knowledge gained over the years as to sources which are definitely unsuitable, by careful indoctrination of aggregate inspectors, and by what may well prove to be injudicious reliance on the magnesium sulfate soundness test.

Some aggregate deposits have become depleted from use over the years and new sources are continually being investigated. Proven service record is naturally not available on the latter and there is every indication that this sequence of events will accelerate with an increasing highway construction program as well as by increasing demand by other users of concrete. Proper evaluation of these unproven sources as to suitability for use in highly durable concrete is becoming more and more urgent.

If the literature be examined for suitable methods for conducting freeze-thaw tests beginning with, for instance, Scholer's paper published in 1928 (1) up to the work of the early 40^7 s, there is insufficient unanimity to permit drawing firm conclusions. Development of air entrainment with its consequent remarkable improvement in durability of concrete gave impetus to many excellent studies. At present, the underlying reasons for this increase in durability of air-entrained concrete are pretty definitely established and are well summarized by Woods (2).

Briefly, the work of Powers (3) and others has shown that a system of well-distributed, discrete air bubbles in the cement paste fraction of hardened concrete provides reservoirs which relieve the stresses caused by expansive action accompanying forma tion of ice when water, if present, freezes. Destructive action may well be due to hydraulic pressures developed ahead of the advancing ice front. Since the role of the fine aggregate appears to be of subordinate significance in the over-all freeze-thaw durability of concrete, this paste, well-protected by entrained air, can be considered to be surrounding the fine aggregate particles, thus providing a well-protected mortar. It will be, in part, the object of the present study to show that when coarse aggregate is embedded in this protected mortar, the over-all concrete freeze-thaw durability may be profoundly influenced.

The implications of the above, as a method of studying aggregate durability as distinguished from mortar or paste durability, have been investigated by Woods and by Sweet (4), Lewis and Venters (5) and associates at Purdue, and form a basis for the $present$ study. Their studies indicated considerable promise for a scheme of studying concrete aggregate freeze-thaw durability wherein the aggregate to be investigated is placed, usually in a vacuum-saturated condition to increase its vulnerability to frost attack, in a ir-entramed concrete and the concrete is then repeatedly frozen m air and thawed in water. The latter freezing cycle does not promote saturation of the mortar and appears to successfully isolate possible destructive effects of the aggregates themselves. It is a common observation that if even well-protected air-entrained concrete containing excellent coarse aggregate be repeatedly frozen and thawed when completely immersed, disintegration will start at the surface and progress inward as saturation builds up toward the interior. If, simultaneously, interior destructive action is occurring due to inferior coarse aggregates, measurement of the mortar durability separately from the coarse aggregate durability then becomes very difficult.

Michigan has now used the freeze-in-air, thaw-in-water cycle for studying aggregates sufficiently to gam considerable confidence in the results obtained. Perhaps the most compelling evidence for success, using the method, has been the observation that for those specimens which deteriorate rapidly, destructive action is centered, in most cases, around those types of deleterious particles for which the department has imposed specification limits for the past 25 years and has trained its aggregate inspectors to

watch for diligently. On the other hand, aggregates of unquestioned durability in service have sustained a very high number of cycles with very little sign of deterioration when using the same technique.

Equipment

Automatic Freeze-Thaw Apparatus. Figure 1 shows a schematic diagram of automatic freeze-thaw equipment which has been used in this work and is much the same m principle as reported by Walker and Bloem (6), with one important exception. At the time of designing the equipment, the laboratory had just completed renovation of its 13- by $10\frac{1}{2}$ -ft cold room with installation of a modern 6-ton refrigeration unit utilizing hot vapor automatic defrosting. Calculation indicated this refrigeration would provide adequate cooling capacity for freeze-thaw work aside from other uses of the room. Consequently, the specimen chamber is

F i gure 1. Schemati c diagram o f automati c freeze-thaw equipment using existing cold room.

located in this room and is exposed to cooling at all times, regardless of whether in the freezing or thawing portion of the cycle. This differs from the usual equipment which provides an individual refrigeration unit for the freeze-thaw system. It has been found that heat loss from the surface of the 40 F water during the thawing period is inconsequential and the controls are simplified by not requiring that the cooling fans be turned off during this period. Defrosting of the unit coolers occurs approximately every six hours, depending upon the cold room load, at which time the air temperature rises to 14 F for about three minutes. This has been found to have little influence on the temperature of the specimen centers when it occurs during the freezing period. The thermal capacity of the specimen chamber was kept as low as possible to conserve heat with metallic connections through to the supporting framework reduced to a minimum. The metal tank rests on wood saddles and except for the open top, the whole is surrounded by 4 in. of cellular glass rigid insulation. The thawing water storage tank is in a pit to one side and below the freezing chamber and is surrounded by air at room temperature. The storage tank itself is similarly enclosed by 4 in. of cellular glass insulation.

The equipment furnishes a cycle which complies with the requirement of ASTM Method C-291, "Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water." In the freezing and thawing cycle used for evaluation of aggregates, $3-$ by 4 by 16-in. concrete specimens are placed in the specimen chamber on a rack which keeps them accurately positioned for uniform exposure to both the freezing air and thawing water. Freezing is accomplished by circulating the cold room air at 0 F to -5 F around the specimens. The temperature of the center of the specimens is brought from 40 \boldsymbol{F} to 0 F in $2\frac{1}{2}$ hours and remains at this temperature or slightly lower (temperatures of -2.5 F have been observed) for 30 minutes, at which time a switch turns on the centrifugal pump and rapidly circulates water at 42 F around the specimens to thaw them. The water continues to recirculate by means of the overflow and the specimen centers rise to 40 F in 45 minutes and thawing continues for an additional 15 minutes at which time the pump automatically stops, and the water in the specimen chamber drains back through the pump into the thawing water storage tank. The equipment thus provides six cycles per day. Temperature loss of the thawing water (about 3 F per cycle) is made up by five 1,000-watt thermostatically controlled immersion heaters in the thawing tank. The test chamber accomodates 36 freeze-in-air specimens and 12 freeze-in-water specimens. With a full load of test beams, approximately 140 gallons of thawing water surrounds the specimens. Since the capacity of the lower thawing water tank is rela**tively large (370 gallons), severe temperature changes of the thawing water are not encountered at any time.**

This freeze-thaw equipment has no interconnection, either mechanical or electrical, between the refrigeration system and freeze-thaw equipment, consequently maintenance has proven relatively simple and the equipment has now operated continuously for two years with very little trouble.

Vacuum Saturation Apparatus. Figure 2 shows a photograph of the vacuum saturation apparatus used to saturate the coarse aggregates under test. Vacuum is obtained by the high-capacity water jet pump shown and brings the pressure down to very close to the vapor pressure of the water (12-15 mm of Hg). The chamber accomodates four

Figure 2. Vacuum saturation apparatus used to saturate coarse ag**gregate .**

quarter-circle segmental shaped containers for simultaneously saturating four different aggregates. The watter valves shown are so arranged as to permit flooding the samples while still under vacuum. The gage glass permits observation that inundation is accomplished and after which saturation is completed at atmospheric pressure.

Concrete Mixer. A rotating drum mixer with inclined axis is used. This mixer is capable of handling a batch slightly larger than one cubic foot. Rotational speed is 22 rpm.

Preparation of Aggregates and Concrete

Effort has been made to establish a highly standardized procedure both for preparation of the aggregates for test and for making the concrete. Insofar as applicable, all procedures for fabricating and proportioning the specimens correspond with ASTM C-233, "Method of Test for Air-Entraining Admixtures for Concrete. " This method specifies use of $5\frac{1}{2}$ sacks air-entrained concrete with fixed sand-total aggregate ratios.

Test Coarse Aggregates. The air-dried aggregate is first separated into four sizes

Two 5,000-gram samples (1, 250 grams of each size) are then prepared, one for specific gravity and usual 24-hour absorption and the other for vacuum saturation absorption.

Vacuum saturation is accomplished by placing the coarse aggregate under vacuum for one hour after which water is admitted to flood the sample while still under vacuum. The vacuum is then released and the aggregate allowed to absorb water for 23 hours. After absorption and specific gravity tests are completed, these same samples are used to determine deleterious particles content by visual examination.

Fine Aggregate. The fine aggregate is not vacuum saturated and is placed in the mix in a dry condition. All sand used so far has come from a single glacial deposit in southern Michigan. The sand is separated into four size fractions and recombined immediately before placing in the mix in the following proportions:

Pass No. 4, retained No. 16 30 percent Pass No. 16, retained No. 50 53 percent Pass No. 50, retained No. 100 15 percent
Pass No. 100 2 percent Pass No. 100

Cement. Type I portland cement, consisting of a blend of three brands readily have been used are shown in Table 1.

TABLE 1 PROPERTIES OF CEMENTS USED

available in the Detroit area has been used. Properties of three lots of this blend which

Air-Entraining Admixture. Neutralized vinsol resin (NVX) in water solution has been used in all mixes.

Mixing Concrete. Twenty-four hours before mixing the concrete, the test coarse aggregate **IS** weighed into one of the segmental shaped containers in an amount sufficient for 0.8 cu ft batch of concrete (usually about 60 lb). Equal amounts of the four sizes are used, and the aggregate is given the same vacuum saturation treatment as indicated for the absorption test. Immediately before placing in the previously "buttered" mixer, most of the water which has been used to inundate the aggregate is poured off and the container and contents weighed. From this, the free and absorbed water contributed by the moist aggregate can be accurately determined for eventual calculation of the water-

Figur e 3. Fiel d record , durabilit y factor , and flexura l strengt h of faile d freez e and thaw beams o f twenty aggregates.

cement ratio. The dry sand and cement are then placed in the mixer and the air-entraining admixture and most of the water introduced at the instant of starting the mixer. A two-minute mix is first given during which time small increments of water may be added to adjust the estimated slump. This is followed by a three-minute rest period with the mixer stopped and the batch is then given a final one-minute mixing. The batch **IS** then dumped into a moistened pan and slump, pressure air content, and unit weight of the fresh concrete immediately determined, after which the following specimens are cast (1) four $3-$ by $4-$ by 16 -in. beams, one each for $7-$ and $28-$ day flexural strength and two for freeze-thaw durability; (2) four $4-$ by $8-$ in. compression test cylinders, two each for 7- and 28-day test.

A complete test on a single aggregate source consists of three such batches each made on a different day.

The molds are stripped the following day and all specimens cured in a constant temperature (74 \pm 1 F) moist cabinet. After 14 days moist curing, the durability specimens are cooled in water to 40 F, weighed, the initial transverse sonic modulus determined, and then placed in the freeze-thaw chamber. Sonic modulus measurements are made at intervals thereafter to trace the progress of deterioration of each specimen. The beams are vibrated in the direction of both the 3-in. and 4-in. dimensions and the average loss of modulus determined.

Freeze-Thaw Durability Results

Included herein are durability results on 20 aggregate sources having service performance from poor to excellent. The field service ratings shown in Figure 3 are the results of general observation and are not intended to represent precise evaluations. Long acquaintance with some of these aggregates, particularly those where the amount used is shown as "extensive," permits a reasonably satisfactory estimate of field performance which is sufficient for the present purpose.

The top line of bars shows the durability factor of each beam, together with the average of the six beams (in two cases, four beams) for each aggregate. The aggregates have been arbitrarily ranked in order of increasing average durability factor. The durability factor shown is based upon discontinuing the test at 300 cycles or at a 30 percent reduction in sonic modulus whichever occurs first. Discussion of this durability factor, as properly representing the resistance to freezing and thawing wil l be made later. The line of bars directly below, in the figure, represents the corresponding flexural strength of the failed beams (or after 1,000 cycles of freezing and thawing if they have not yet failed) expressed as percent of strength of the 28-day beams. The very substantial loss in strength caused by disruptive action of bad aggregate is apparent. Two strengths are obtained on each beam, using center point loading on an $8^1/2$ -in. span with the load applied in the direction of the 3 -in. dimension. It will be observed that considerable variation occurs in these strengths and does not appear to be related to the durability factor ranking. The average flexural strength of the failed beams is 54 percent of the 28 day strength which provides a highly convincing criterion of failure to the highway engineer who is conscious of the necessity for maintaining a high flexural strength in paving concrete. It should be pointed out that the durability beams are cured only 14 days and presumably attain about 85 percent of the 28-day strength before freezing and thawing started.

The rather large variations in durability factor and in flexural strength following failure for a given aggregate, both within batch and from batch to batch, are now regarded not as the result of an inherently faulty test method but as the normal consequence of a random distribution of deleterious particles in the specimens. The sonic modulus test appears to give an average value of the disintegration of the specimen and does not foretell that certain portions of the beam may have suffered severe mternal breakdown.

In the case of specimens which failed in a relatively few number of cycles, it was observed that this failure centered around the types of deleterious particles which, with one exception, are already well-known in Michigan; namely, iron clay stones, chert, shale, soft sandstone and the like. Examination of the beams following failure in freezing and thawing frequently permits accurate prediction that one or the other flexural breaks will be unusually low due to a concentration of cracks near one end of the beam. The crack pattern will frequently be radial, fanning out from an underlying particularly expansive particle. The fracture, after breaking, is often highly irregular, following a random path greatly weakened by disruptive action of bad aggregate and the fractured face usually exposes the offending particle. A type of particle which had not heretofore been considered deleterious in Michigan practice has been observed in these tests in several Michigan deposits, namely, a limestone pebble which ordinary techniques have been unable to identify. Work to date which is only suggestive, using differential thermal analysis, indicates these to be calcium carbonate rock pebbles containing small amounts (less than 10 percent) of clay minerals, probably kaolinite.

Inference might be drawn from the above that the freezing and thawing tests constitute

primarily a confirmation of knowledge already gained from field observations as to the types of particles which are deleterious. To some extent, this is true for aggregates showing low durability. However, for the aggregates which exhibit intermediate durabilities, the freeze-thaw tests provide a continuous scale of measured values obtained by a method not subject to personal judgment, and extend knowledge beyond that gained by visual examination of the aggregates. Figure 4 presents the results of hand-picking these same aggregates for the three classes of deleterious particles as presently defined by the Michigan specifications; namely, (1) "soft particles" which include "shale, soft sandstone, ochre, coal, iron bearing clay, weathered schist, shells, floaters, partially disintegrated particles, cemented gravel and any other particles which are structurally weak or which fail to meet the soundness test, $\frac{1}{2}$ "chert, "and (3)"hard absorbent particles. " Hard absorbent particles are less-easily defined but cover, in general, an intermediate class of particles, less objectionable than the soft particles, which experience over the years, have proven deleterious. It is observed from Figure 4 that although, generally, durability increases with diminishing amounts of deleterious particles, the relation is not a satisfactory one. The ranking of the aggregates obviously would be quite different if the deleterious particles content was used as the sole criterion.

It seems possible to consider the over-all aggregate durability as consisting of two parts: (1) breakdown due to identifiable deleterious particles and (2) ultimate breakdown due to "basic" durability of the aggregate. For instance, the poor durability of aggregate reference No. 2985, as evidenced by an average durability factor of only 22 — contrasted with some of the other aggregates having comparable, or more amounts of recognizable deleterious particles, may indicate a lower "basic" durability for this particular aggregate or, of course, it may also mean that the deleterious particles found in this aggregate are unusually destructive. Consideration had been made as to whether more

useful data could be acquired by removing the recognizable deleterious particles prior to conducting the freeze-thaw tests to enable a better estimate of this basic durability. The present opinion is that removal of the deleterious particles prior to the test infers a degree of knowledge of their relative destructiveness which is not now justified. Data presented later, on the durability of chert in connection with heavy media separation plants, confirms the wisdom of this decision.

Durability Factor versus Total Cycles for Failure

Consideration has been given as how best to express the numerical rating of the durability of a particular aggregate. For those aggregates whose beams all fail at less than, for instance, 300 cycles of freezing and thawing, the average number of cycles for failure would appear to be an entirely satisfactory method. However, some aggregates have beams which do not fail at even several hundred more cycles. This, inci-

Figure 5. Average durability factor of vacuum saturated coarse aggregate versus number of cycles for failure.

dentally, ties up freezer space. It is desirable in the latter case to make recourse to the durability factor to express the rating of such an aggregate so as to permit removal of the beams from the freezer at a specified time. Durability factors shown are based upon terminating the test at 300 cycles, or at a 30 percent reduction in sonic modulus, whichever occurs first. It should be emphasized that such a choice of 300 cycles and 30 percent reduction in modulus provides a unique scale of durability rating and is, so to speak, an irrevocable choice. The values cannot usually be transferred to a durability factor of another base, nor can they be compared with, for instance, published data unless that likewise was based upon 300 cycles and 30 percent reduction. For beams which fail at greater than 300 cycles, the durability factor can be likened to taking a snapshot of the beam's condition at 300 cycles and then predicting from this at what number of cycles it actually will fail. The extent to which this prediction is successful is shown in Figure 5 where number of cycles for failure is plotted against average durability factor. It is observed that the durability factor, once its peculiarities are learned, reasonably successfully predicts the total number of cycles for failure and it is concluded that almost as much useful information is obtained by terminating the test at 300 cycles as by continuing on to failure. Terminating the test at 300 cycles certainly simplifies the procedure greatly by permitting orderly scheduling of freezer space.

In this series of tests, the durability factor does not bear a linear relationship to the total cycles for failure except at the low end of the scale. For instance, aggregate having an average durability factor of 45 sustained about 190 cycles, whereas the curve in dicates that aggregate having twice this durability factor would fail at much more than twice this number of cycles, actually at about 630 cycles. To state it differently, for very durable aggregates, the number of cycles at which failure occurs is underestimated by the durability factor.

TABLE 2 COARSE AGGREGATE ABSORPTION AND SPECIFIC GRAVITY AND CONCRETE DATA FOR DURABILITY MIXES

									Concrete Mix Data					Strength, psi		
		Coarse Aggregate				Sand,			Wt per Actual							
No.	Aggregate Bulk Sp Gr (dry basis)		Absorption, % 24-hour Vacuum Ratio		No	Batch % of total Slump Agg	\mathbf{n}	cu ft, lb	sk/cyd	Cement Net Water gps	Aır. %	7-days	Compressive ^a $28 - days$	Flexural ^b	7-days 28-days	Durability Factor
48 Gravel	2, 61	1 73	2 14	0, 81	1 2 3	36 36 36 36	$\frac{27}{3}$ 3%	1458 1447 147.5	5 51 5 45 5,56	4 78 4 93 5 02 491	6.4 6.4 5.5	2750 2395 2475 2540	3320 3330 3260 3305	625 515 500	705 650 625 660	12
2968 Gravel	2, 53	2, 63	3, 14	0,84	Avg 1 2 3 Avg	36 36 36 36	34 3 ² 3 4% зу	146 0 1457 146 1 144 1 145, 3	551 5 5 6 5, 57 548 5 54	481 4.78 5,06 488	6, 1 49 48 60 52	2415 2825 2150 2465	3305 3500 3085 3295	545 645 595 580 605	755 735 760 750	17
2343 Gravel	2 58	2, 12	2, 54	0.83	1 2 3 Avg	38 38 38 38	$\frac{47}{26}$ $\frac{3}{2}$	142 8 147.5 145 6 145.3	5 40 5, 58 5,50 5 49	491 496 5 02 4 96	7.3 52 6, 3 6, 3	2700 2910 2390 2665	3320 3720 3530 3525	660 665 720 680	765 820 710 765	19
2985 Gravel	2 64	1,45	1 60	0.91	1 2 3 Avg	36 36 36 36	24 2 $2^{1/2}$ $2^{1/2}$	148.4 149 3 148.9 1489	5 5 8 5.61 5.60 5.60	4 89 4 97 4 89 4 9 2	58 51 55 55	2540 2900 2780 2740	3300 3640 3860 3600	685 615 790 695	780 785 800 790	22
1878 Gravel	2, 56	2,66	2 81	0,95	1 2 з Avg	36 36 36 36	4% 3% 4 4	144, 7 144, 7 145, 8 145.1	5,48 5 49 5, 53 5,50	5 17 498 5 01 5 05	6. 0 6, 1 5. 2 5, 8	2460 2410 2310 2395	3260 3380 2935 3190	590 585 545 575	720 710 640 690	29
2382 Crushed Gravel	267	1, 25	1 48	0.84	1 2 3 Avg	38 38 38 38	1% 14 з $2^{\frac{1}{2}}$	149.2 149.0 147.5 148.6	5.59 5, 56 5 50 5, 55	480 5 06 5 10 500	4, 9 5, 0 70 5. 6	2660 2280 2490 2475	3815 3090 3210 3370	740 645 615 865	845 670 700 740	35
2445 Gravel	2 65	1, 33	1, 70	0,78	1 2 3 Avg	36 36 36 36	3½ $\frac{27}{37}$	1480 146 3 146, 7 147 0	5 54 5.47 5,49 5,50	494 5 25 5, 02 5,07	4.7 4,9 5, 6 5, 1	2735 2360 2445 2516	3115 2995 3235 3115	745 720 570 680	825 840 660 775	40
3170 Gravel	264	119	161	0.74	1 2 з Avg	36 36 36 36	$2^{3}/_{1}$ 2 $2^{1}/$	147 0 1499 148, 5 148 5	5, 52 5 66 5 59 5 59	4, 99 447 4, 75 4 74	6, 3 4, 3 6, 2 5.6	2430 3310 2760 2835	3420 3720 4080 3740	690 705 660 685	850 835 825 835	41
1865 Gravel	264	1, 20	1, 65	0, 73	1 2 Avg	36 36 36	$3\frac{1}{2}$ $\frac{3}{3}$	146,5 1477 147 1	5 54 5, 59 5 57	499 5 00 5 00	5, 6 5, 2 6, 4	2925 2625 2775	3540 3315 3430	750 665 710	785 755 770	42
2778 Gravel	2.62	1.62	2 00	0, 81	1 2 3 Avg	36 36 36 36	5 $\frac{1}{2}$	1449 149.7 149 1 147 9	5.44 5.65 5 49 5, 53	5 27 4 66 4 70 488	6, 6 4, 0 4, 4 5. 0	2205 2490 2860 2520	2915 3555 3765 3410	705 755 635 700	730 855 730 770	47
684 Gravel	2 61	1 73	196	0,88	1 2 3 Avg	36 36 36 36	$\frac{3}{3}$ / 1 2°	146 0 146 3 149.7 1473	5, 51 5 51 5 65 5 56	5 04 5.02 4 94 5 00	63 6, 3 4, 1 5, 6	2505 2655 2825 2660	3320 3270 3800 3465	625 635 585 615	715 720 645 695	49
1237 Gravel	2 63	1,60	2 03	0, 79	1 2 з Avg	36 36 36 36	2^{7} 3 $\frac{5}{3}$	1477 1473 147.1 1474	5 55 5 54 5, 54 5.54	4 94 4 9 2 475 487	6, 4 6, 0 64 6, 3	3005 2840 2685 2845	4070 3750 3395 3740	575 705 715 865	715 790 790 765	54
722 Gravel	267	153	1 70	0 90	1 2 з Avg	36 36 36 36	$\frac{4}{5}$ $\frac{1}{2}$ 3%	147.0 146, 3 151.5 148 3	5.51 5,46 5,66 5, 54	4 65 5 01 4 91 4 86	6, 8 7, 1 4, 5 61	2455 2660 2825 2645	3250 3425 3860 3520	590 610 565 590	665 665 625 650	56
1856 Lumestone	257	3.25	3.79	0,86	1 2 Avg	41 41 41	$1\frac{3}{2}$ $\frac{1}{1}$	146 7 145, 7 146, 2	5 55 5.49 5.52	5.69 6 02 5 86	5, 2 5, 0 5. 1	3410 3190 3300	4615 4385 4500	795 755 775	950 880 915	70
3551 Limestone	261	1, 28	142	0,90	1 2 з Avg	41 41 41 41	$\frac{1}{1}$ 1 14	146, 5 146 2 1472 146 6	5 54 5 52 55 5 5 54	504 502 5 30 512	59 51 45 5, 2	2710 2900 2535 2715	3200 3420 3435 3350	725 690 660 690	835 920 765 840	73
683 Limestone	271	145	178	081	1 2 з м	41 41 41 21	ر/ 4⁄4 2 $\overline{2}^2/$	152 2 147 5 149 2 149 6	63 5 5 44 50 5 D D2	5.16 540 541 5 J.Z	4.5 63 5, 1 , ,	3135 2700 2600 281U	3820 3255 3535 3535	655 655 605 64 U	660 690 625 660	75
3545 Crushed Gravel	2 60	153	2, 39	0, 64	1 2 з Avg	38 5 38 5 38 5 38 5	1% 3 $\frac{2^3}{2^7}$	147 4 147 1 148 1 147 5	56 5 554 5 60 5.57	5 01 5 05 477 494	49 49 51 5,0	3165 3125 2960 3085	4240 4405 4190 4280	695 745 720 720	880 895 890 890	90
1074 Limestone	262	2, 14	2 94	0.73	1 2 3 Avg	41 41 41 41	X 3 2% 2½	149 2 146 5 146.8 147.5	5 57 5 45 5, 47 5 50	524 551 554 5 43	5, 1 5, 7 56 55	3030 2960 2855 2950	4215 3915 3885 4005	790 725 710 740	865 975 925 920	97
1225 Limestone	2 64	0.76	1 10	0, 69	1 2 з Avg	41 41 41 41	$\frac{1}{3}$ $\frac{37}{26}$	146 8 144 1 144 5 145.1	5,50 5 40 5, 41 5.44	541 5 42 5.54 5 4 6	5, 3 6, 7 7, 6 65	2880 2645 2625 2715	3780 3360 3210 3450	690 735 800 740	810 870 885 855	100
49 Crushed Gravel	2 68	1, 45	1, 71	0.85	1 2 3 Avg	38, 5 38 5 38 5 38 5	2% $\frac{3}{1}$ $2\frac{1}{2}$	146 6 146 0 148.9 147 2	5 45 5 44 5.55 5 4 8	515 503 492 5 03	6, 6 70 48 61	2420 2485 2810 2570	3290 3070 3670 3345	535 615 580 575	725 730 725 725	102
	³ Each value shown is aversge strength of two 4 x 8 in cylinders															

^a Each value shown is average strength of two 4 x 8 in cylinders
^b Each value shown is average of two breaks on one 3 x 4 x 16 in. b

Table 2 shows certain characteristics of the coarse aggregates together with usual concrete mix data. The 24 -hour cold water immersion absorption is shown for the aggregates and also the vacuum saturation absorption together with the ratio of the two. No useful relation between these values and durability factor has been discovered, nor does specific gravity appear to have particular significance for these twenty aggregates.

Close scrutiny of the proportioning and strength data in Table 2 reveals occasional anomalous values which seem unavoidable in making such a series of batches of concrete, but generally the averages appear satisfactory. Average compression strengths appear related fairly well to water-cement ratio whereas flexural strengths presumably reflect different structural qualities and surface texture of the aggregates. Neither

Figur e *6.* **Durabilit y and deleteriou s particle s content befor e and afte r processin g i n thre e commercial heavy media plants. Als o** durability and accelerated soundness when diluted with chert of different specific gravities.

type of strength appears related to freeze-thaw durability.

Sand content of the mixes was proportioned by interpolation between 36 and 41 percent for gravel aggregates containing appreciable amounts of crushed particles.

Consideration has been given as to whether the inadvertent changes in air content for the individual batches were affecting durability. For an aggregate on which three batches were made, the average durability of the two beams from each batch has been calculated and three observations made as to whether the durability increased or decreased with an increased air content. For the twenty aggregates, 56 such observations can be made (only two batches each were made on two aggregates). In 25 cases durability increased with increased air content and in 26 cases durability decreased. In five cases, observation could not be made since the air content was the same. It is concluded that within the range used (average 5-7 percent), air content was not significantly affecting the aggregate durability. Field observations amply support the viewpoint that air-entrainment has not alleviated the problem of deleterious aggregates.

Service Record versus Durability Factor

Establishment of a rating of these aggregates in actual services poses numerous problems. The environment of the concrete certainly influences the behavior of the aggregates, — moist conditions favor pop-outs from deleterious particles. On the other hand, the nature of the environment may well influence the desired performance. For instance, almost complete absence of unsightly pop-outs is desired on the exposed

architectural concrete of a bridge or grade separation structure. It is not considered that too much reliance should be placed on behavior of these aggregates in concrete prior to the days of air-entrainment. Mortar disintegration of non-air-entrained concrete may well not be distinguished from distress due to aggregates. Insofar as our present knowledge extends, the durabilities reported herein correlate reasonably well with service performance of air-entrained concrete, particularly in distinguishing the aggregates which show excessive surface pop-outs in paving concrete as well as those of unquestioned durability. The department now has a service performance program underway for determining the extent of surface defacement caused by deleterious particles in pavements constructed since 1945, — approximately the time when reliable, extensive air-entrainment construction records became available. The work to date in this program shows promise of confirming the laboratory data.

The aggregate samples on which tests are shown herein, represent current production. It **IS** common knowledge that the character of glacial deposits as well as lime stone ledges may vary greatly within a distance of a few feet, consequently, rigorous correlation of laboratory tests of present production with field performance of past production presents a serious obstacle.

Heavy Media Separation Plants

Three commercial heavy media separation plants have now been operating m Michigan sufficiently long to gain some knowledge of their performance in improving gravel aggregates. In the case of two of these plants, freeze-thaw studies have been made of the gravel both before and after heavy media separation. The product of the third plant, before processing, had such a high content of deleterious particles that freeze-thaw tests were not conducted.

Figure 6 summarizes the durability tests on these three commercial plants and demonstrates the substantial decrease in deleterious particles content and corresponding

TABLE 3 MISCELLANEOUS TÉSTS OF DURABILITY CONCRETE MIXES MADE WITH GRAVEL BEFORE AND AFTER COMMERCIAL HEAVY MEDIA PROCESSING
ALSO MEDIA PROCESSED GRAVEL CONTAINING CHERT OF VARIOUS GRAVITIES

 b Each value shown is average of two breaks on one 3 x 4 x 16 in beam

increase in durability of the processed material. Table 3 gives the concrete mix data for these batches together with specific gravity and absorption data. It is gratifying to observe from the table that 17 of the 18 test beams made with the media processed gravel from the three plants had durabilities of 70 or greater, demonstrating that the non-
durable particles have been quite successfully removed by the processing.

Routine daily tests on the processed gravel from plant No. 3 indicated that the small Routine daily tests on the processed gravel from plant No. 3 indicated that the small
ount of deleterious particles which were not removed by the heavy media processing. amount of deleterious particles which were not removed by the heavy media processing was predominantly chert. Question naturally arose as to how deleterious this small
emount of relatively high gravity chert actually is perticularly in view of the weak o amount of relatively high gravity chert actually is, particularly in view of the work of Wuerpal and Rexford (7) who concluded that the unsound chert was confined largely to
that hering a bull greef is grewity of less then 2.40. In ender to investigate this sen that having a bulk specific gravity of less than 2.40. In order to investigate this conclusion using Michigan aggregates, approximately one ton of the gravel rejected and wasted by the flotation process was brought to the laboratory and the chert removed by hand picking. The chert was then graded into the four size groups used in the previous durability studies, namely, 1 to $\frac{3}{4}$ in., $\frac{3}{4}$ to $\frac{1}{2}$ in., $\frac{1}{2}$ to $\frac{3}{6}$ in., and $\frac{3}{6}$ in. to No. 4, and then separated into four specific gravity ranges in a saturated condition by laboratory separation processes using acetylene tetrabromide-carbon tetrachloride mixtures, as follows: minus 2.45, 2.45 to 2. 50, 2. 50 to 2. 55 and 2. 55 plus.

The choice of values for specific gravity ranges was arbitrary. It seemed probable, however, from previous work (5) that commercial operations would usually float off objectionable material somewhere between 2.50 and 2.60 gravity. As a consequence, this range was split in the middle at 2.55 to provide more detailed information. The two range was split in the middle at 2.55 to provide more detailed information. lower gravity groups, minus 2.45 and 2.45 to 2. 50, would provide chert to substantiate trends in aggregate durability reported elsewhere.

In the chert series of durability tests, decision was made to replace the flotationimproved gravel by 10 percent chert of each gravity group and to distribute it in equal amounts down through the four coarse aggregate sizes. For example, one sample of gravel for durability study was preparec using 90 percent flotation-improved gravel plus 10 percent chert of minus 2.45 specific gravity. The choice of 10 percent chert with which to dilute the gravel in each case was based on the premise that if the chert were actually deleterious there would surely be enough to be readily detected.

Figure 6 includes the results of the chert durability studies from plant No. 3. The data indicates the flotation processed material to be most frost resistant. Marked reduction in durability occurs when this flotation-improved gravel is diluted with 10 percent chert of minus 2.45 gravity but the durability successively improves as higher and higher gravity chert is introduced, leading to the conclusion that the high-gravity cher: remaining in the media-processed gravel should be less harmful than "average" chert of variable specific gravity.

The results of the accelerated soundness test (5 cycles of MgSO $_4$) are also shown. It is there observed that the soundness test is entirely unsatisfactory for predicting freeze-thaw durability of the aggregate diluted with chert. For instance, the aggregate containing 10 percent of the lowest gravity chert exhibits poor freeze-thaw durability but shows a magnesium sulfate soundness loss of only 2.4 percent. Since much higher soundness losses have been observed in the past on samples from other sources at this laboratory, it seems proper to regard the soundness test as a highly selective one which is destructive to some types of particles but not to chert.

Summary

Included herein is a review of the efforts of one organization to solve the long perplexing problem of laboratory evaluation of the freeze-thaw durability of concrete coarse aggregates. Considerable promise is shown for a method using automatic equipment to provide a freeze-thaw cycle wherein the aggregates under test are placed in concrete in a vacuum saturated condition to make them more vulnerable to frost attack. The mortar fraction of the concrete is made highly resistant to frost action by using dry sand and by incorporation of entrained air. The cured concrete is then alternately subjected to freezing in air to 0 F and rapid thawing is water to 40 F. This cycle does not promote saturation of the concrete and destructive action appears to be centered around

deleterious particles in the aggregate, thus indicating considerable success for isola-
ting the effect of aggregate durability from possible lack of mortar durability. Sonic ting the effect of aggregate durability from possible lack of mortar durability. modulus measurements are used to detect internal breakdown of the concrete.

The field surveys necessary for more positive proof, than reported here, of precise correlation of the laboratory durabilities with service performance may be time consuming. Surface defacement studies of existing air-entrained concrete using many of the aggregates reported are now underway, A desirable supplement to this would be acquisition of field data aimed at measuring the internal distress of the concrete from freezing and thawing action.

Three types of evidence make it appear that the laboratory data is consistent within itself and that the method is indeed measuring the characteristics for which it was designed

1. Early breakdown of the laboratory concrete is associated with types of deleterious particles which Michigan has long recognized as being necessary to limit in frost resistant concrete.
2. The method

The method has successfully measured the improvement in freeze-thaw resistance of commercially produced heavy media processed gravel over the unprocessed gravel such as was predicted by others.

3. The method has successfully measured the loss in durability when aggregate is diluted with low gravity chert so as to confirm work done some 15 years ago, and extends that work to show there to be a definite scale of chert durability.

ACKNOWLEDGMENT

Appreciation is expressed for the encouragement and help given this work by W. W. McLaughlin, Testing and Research Engineer, Michigan State Highway Department, and by Professor Earnest Boyce, Chairman, Department of Civil Engineering, University of Michigan, also to the many members of the staff of the Michigan State Highway Laboratory, particularly Louis Gombos who performed much of the design and detail work of the program. Dr. E. William Heinrich, Department of Mineralogy, University of Michigan, aided in identification of the limestone pebbles and directed the D. T. A. analysis.

REFERENCES

1. Scholer, C. H. , "Some Accelerated Freezing and Thawing Tests of Concrete," Proceedings, ASTM, VoL 23, pp. 472-489 (1928).

2. Woods, Hubert, "Observations on the Resistance of Concrete to Freezing and Thawing," Proceedings, A.C. L, Vol. 51, pp. 345-349 (Dec. 1954).

3. Powers, T. C., "The Air Requirement of Frost Resistant Concrete," Proceedings, Highway Research Board, VoL 29, p. 184 (1949).

4. Sweet, Harold S., "Research on Concrete Durability as Affected by Coarse Aggregate," Proceedings, ASTM, VoL 48, pp. 988-1019 (1948).

5. Lewis, D. W., and Venters, Eduards, "Deleterious Constituents of Indiana Gravels, " Highway Research Board Bulletin No. 94 (1954).

6. Walker, Stanton, and Bloem, D. L. , "Performance of Automatic Freezing and

Thawing Apparatus for Testing Concrete," Proceedings, ASTM, VoL 51, p. 1120 (1951).

7. Wuerpel, C. E., and Rexford, E. P., Proceedings, ASTM, VoL 40, p. 1021 (1940).

Effect of Heavy Media Separation on Durability Of Concrete Made with Indiana Gravels

LT. RICHARD D. WALKER,* USAF, and

J. F. **MCLAUGHLIN**, Research Engineer, Joint Highway Research Project Purdue University

Alternate freezing and thawing are among the most destructive of the natural weathering conditions to which concrete is subjected. Indiana pavements undergo a large number of freezing and thawing cycles each year, and in addition, several aggregates of questionable durability are found in the state. The Joint Highway Research Project at Purdue University has, therefore, done considerable research concerning the freezing and thawing of concrete.

In 1946, Woods, Sweet, and Shelburne (1) reported results of extensive field surveys of over 2,600 miles of rigid pavements constructed in Indiana prior to 1935. Statistical analysis of the data indicated that the field performance of the pavements was directly related to the source of coarse aggregate used. Further investigation revealed a relationship between the mineralogical composition of gravel coarse aggregate and pavement field performances (2).

It appeared evident that the aggregates which were shown to have poor field performance met, and still would meet, most of the commonly employed acceptance tests developed for specification purposes (3). This fact gave impetus to many laboratory research programs directed toward development of a test, the results of which could be correlated with field performance. Among these programs was a study by Sweet (4) in which he concluded that freezing and thawing tests could be used to differentiate coarse aggregates with poor field performance from those with good performance. He suggested that all coarse aggregates tested should be put into concrete after being vacuum satuiated so that the moisture condition of the aggregates would be similar to the degree of saturation that may be encountered in a pavement.

Using Sweet's conclusions and recommendations, other research programs were conducted. Studies of the effect of freezing and thawing on airentrained concrete using poor aggregates were conducted by Bugg (5) and Blackburn (6). Venters (7) found that the deleterious portions of the gravels have high absorption and a potentially high degree of saturation. He was able to separate the deleterious particles from the gravels by means of heavy liquid flotation. Commercial heavy media (or heavy liquid) separation methods now exist and have been applied successfully,toward the improvement of certain gravels (8).

All of the studies mentioned have produced laboratory data which can be correlated with the field performance of aggregates, but in each, either all gravel or all crushed stone coarse aggregate was used. Indiana's present field practice, however, is to blend crushed stone (for the larger sizes of the coarse aggregate) with gravel. Also, the development of commercial heavy media separation methods makes worthy of consideration the more extensive use of these methods for the improvement of gravel aggregates. These two factors merited laboratory research and form the basis of this study.

• THE purpose of this study was to obtain laboratory freezing and thawing data that would indicate what effect, if any, the addition of crushed stone and the use of heavy media separation have on the durability of concrete made with gravel coarse aggregate. In order to carry out this work, four gravels were chosen, representing the northern,

* Formerly Research Assistant, Joint Highway Research Project, Purdue University.

middle, and southern parts of the state of Indiana. Three crushed limestones were selected to be used with these gravels on the basis of their use m field practice; i. e., a northern stone is used with a northern gravel, etc. The stone used with the northern gravel was a reef-type material from the Huntington limestone formation. Stone from this formation has a very high porosity but has shown relatively good performance in the past. One source of stone from the St. Genevieve formation was used with the other gravels since a large part of the limestone quarried in the central and south central parts of the state is from this formation. A third limestone from the Glen Dean formation was also used with one of the southern gravels because it is of unknown quality but in the future may be used extensively in the southern part of the state.

In the heavy media study, the larger sizes of gravel in the gravel-crushed stone combinations were separated at two different specific gravity levels. This was an attempt to apply a procedure that might prove to be commercially economical. If heavy media separation is to be economically feasible in Indiana, it would not be desirable to treat the entire gradation of the aggregate. In addition, other investigators have found that if the deleterious particles are eliminated from the larger sizes, considerable benefit will result $(9, 13)$. The deleterious particles might then be crushed into smaller sizes, which would minimize their harmful effects.

To complete the study, six series of tests were conducted. Five different mix designs were included in each series. Batches of concrete for each of the five mix designs of each series were mixed on one day and then repeated on three other days. Three, $3-x$ 4-x 16-in. beams were made from each batch of concrete, resulting in sixty beams. 3-x 16-in. because were made from each batch of concrete, resulting in stars, resulting from each series. The results are based on freezing and thawing tests on a total of 360 for each series. The results are based on freezing and thawing tests on a total of 360 beams.

Test Series and Mix Designs

The first test series was designed to bring out the influence of the size of deleterious particles on the durability of a concrete mix. The results from this series were used in designing the remainder of the experiment.

In series 1, a single coarse aggregate (a gravel of mediocre field performance) was subjected to heavy liquid separation to remove deleterious particles from different parts of the gradation. Five mixes were included which were separated as follows:

> Mix A - No heavy media separation Mix B - Separation used on 1 in. - $\frac{3}{4}$ in. fraction Mix C - Separation used on 1 in. $-\frac{1}{2}$ in. fraction Mix D - Separation used on 1 in. $\frac{3}{8}$ in. fraction Mix E - Separation used on 1 in. - No. 4 fraction

A heavy liquid having a specific gravity of 2. 50 was used for all heavy media treatments in this series.

Five additional test series were made, each of which was identical in all respects except for the source of the coarse aggregate used. Again, five mix designs were used for each series and these were intended to determine the differences in durability among concretes made with gravel, stone, a combination of gravel and stone, and a combination of gravel and stone in which the gravel had been treated by heavy media techniques.

More specifically, the following five mix designs were used for Series 2 through 6:

- Mix F Gravel aggregate for the entire coarse aggregate gradation which ranged from a 1-in. top size to material retained on a No. 4 sieve.
- Mix G Crushed stone, subsequently to be combined with the gravel used in Mix F was used for the entire aggregate gradation in mix design G.
- Mixes These mixes utilized the stone of Mix G as 35 percent of the H. I. total coarse aggregate in the sizes ranging from the 1-in.
- H, I, total coarse aggregate in the sizes ranging from the 1-in.
and J stop size to $\frac{1}{2}$ -in. The remaining 65 percent of the total a
- top size to $\frac{1}{2}$ -in. The remaining 65 percent of the total aggregate was the gravel used in Mix F, ranging from a top size of $\frac{3}{4}$ -in. to the No. 4 sieve. Mix designs I and J differed

from H in that the $\frac{3}{4}$ - to $\frac{1}{2}$ -in. portion of the gravel had been separated in a heavy liquid; i.e., the particles which floated were discarded and the materials which sank were incorporated in the mix. A heavy liquid having a specific gravity of 2. 35 was used for Mix I and one having a specific gravity of 2. 45 was used for Mix J.

The test series, mix designs, and coarse aggregate gradations are summarized in Table 1.

MATERIALS AND PROCEDURES

Mineralogic composition, field performance records, and the sources of the coarse

TABLE 1

SUMMARY OF TEST SERIES, MIX DESIGNS, AND COARSE AGGREGATE GRADATIONS

^a Series 2 through 6 differed from each other in the source of the coarse aggregates used.

Series	Material	Percent Absorption Sp Gr	Bulk	Field Perfor- mance History	Geologic Source
$\mathbf{1}$	gravel 1 gravel 1, separated at sp gr 2.50	2.49 1.12	2.54 1.70	poor	Lower Wabash River terrace
$\mathbf{2}$	gravel 2 gravel 2, separated at sp gr 2.35 gravel 2, separated at sp gr 2.45	4.01 1.63 0.98	2.38 2.54 2.59	bad	Dredged, lower Ohio River
	stone 1 (fine grained limestone from central Indiana)	0.70	2.68		St. Genevieve formations, Mississippian age
3	gravel 3 gravel 3, separated at sp gr 2.35 gravel 3, separated at sp gr 2.45	2.77 1.54	2.50 2.56 2.59	bad	Dredged, lower Ohio River
	stone 2 (coarse grained limestone from southern Indiana)	1.80	2.61	no record	Glen Dean formation, Mississippian age
4	gravel 4 gravel 4, separated at sp gr 2.35 gravel 4, separated at sp gr 2.45	1.51 1.01 0.98	2.68 2.73 2, 74	good	Dredged, upper Ohio River
	stone 1		See Series 2		
5	gravel 5 gravel 5, separated at sp gr 2, 35 gravel 5, separated at sp gr 2.45	2.36 2.19 1.94	2.60 2.62 2.64	fair	Glacial outwash, northcentral Indiana
	stone 3 (porous dolomitic reef rock, northcentral Indiana)	2.99	2.63	good	Huntington formation, Sılurıan age
6	gravel 6 gravel 6, separated at sp gr 2.35 gravel 6, separated at sp gr 2.45	2.82 1.85 1.51	2.57 2.63 2,65	poor	Lower Wabash River terrace
	stone 2		See Series 3		

TABLE 2 COARSE AGGREGATE DATA

TABLE 3

aggregates used in this study are shown in Tables 2 and 3. A single fine aggregate sam-

ple from a river terrace deposit of glacial origin was used in all concrete mixes. This material met Indiana's specifications for concrete sand and had the following characteristics: F.M. - 3.13, bulk specific gravity - 2. 61, absorption - 1. 65 percent.

Type I portland cement from a single clinker batch was used for all mixes. Since only one cement lot was used, the cement characteristics do not affect the conclusions

TABLE 4

DURABILITY OF SERIES 1 (Gravel 1)

² Asterisk indicates significance at the 5 percent level. Required difference for significance is 22.9.

and, therefore, are not included here. Darex, added at the mixer, was used as the airentraining agent.

The heavy liquids were prepared by mixing carbon tetrachloride (specific gravity 1. 58) and acetylene tetrabromide (specific gravity 2. 97). The specific gravities of the mixtures were checked with a hydrometer.

The concrete mixes were designed by the B/B_O method (14) which is essentially the same as the "Recommended Practice for Selecting Proportions for Concrete" published by the American Concrete Institute (15). All mixes were designed for six bags of cement per cubic yard of concrete, 4. 5 percent air, a 3-to 4-in. slump, and to produce 0. 5 cubic feet of concrete. Details of aggregate gradations used are shown in Table 1.

Absorption and bulk specific gravity values were determined for the coarse aggregates by means of vacuum saturation techniques. The fine aggregate was a commonly used sand for which data were available.

In making the specimens, trial mixes were used to establish the water and air-entraining agent requirements. The water-cement ratios and slumps of most of the mixes conformed to the specifications for paving concrete of the Indiana State Highway Department. The mixing was done in a 1, 5 cubic foot tub-type mixer. Three $3 - x 4 - x 16 - in$. specimens were molded from each batch according to the procedure specified by ASTM designation C192-52T. After curing for one day in a humid atmosphere, the beams were removed from the molds and cured in water having a temperature of about 80 F for 13 days after which they were weighed and tested for their relative dynamic modulus of elasticity according to ASTM designations C215-52T and C290-52T. If space were available, sets of three beams were placed in the freezing and thawing apparatus. If not, the beams were placed in cold storage in a freezer where the ambient temperature was about -15 F. When space became available, the specimens, in sets of three, were transferred from cold storage to the freezing and thawing apparatus. An attempt was made to have all 15 specimens that were fabricated on the same day placed in the freezing and thawing apparatus following curing, or to have all of them placed in cold storage.

TABLE 5

DURABILITY OF SERIES (Gravel 2 and Stone 1)

a Asterisk indicates significance at the 5 percent level. Required difference for significance is 11. 5.

It was assumed that the results were not affected by the length of time that the beams were stored. This procedure facilitated convenient scheduling of sets of three beams in the testing program.

The freezing and thawing cycle corresponded to ASTM designation C291-52T, rapid freezing in air and thawing in water. The concrete specimens were exposed to approximately seven cycles per day through the use of automatic equipment. In this equipment, the air temperature was reduced to 0 F in about one hour of the freezing cycle and within $2\frac{1}{2}$ hours the centers of the beams also reached 0 F. At this time, the thaw water was circulated and the ambient temperature quickly rose to 40 F. The centers of the specimens reached 40 F within 30 minutes. After 35 minutes elapsed, the water was pumped out and then the freezing cycle began again.

Periodically, weight and dynamic modulus of elasticity determinations were made on the specimens. A specimen was removed from the test program when it reached a point where its relative dynamic modulus of elasticity was 50 percent or less. In addition, the test program was ended at 300 cycles of freezing and thawing for all except series 3.

A durability factor described by Stanton Walker (16) was used as a measure of relative durability among the various types of concrete used. This factor was calculated for the test results at 100 cycles and at 200 and 300 cycles where applicable. Analysis of variance was used to detect differences in the durability performance of the mix designs.

RESULTS AND DISCUSSION

In this section, the results are presented and discussed for each test series. The graphs (Figures 1 through 6) represent the average performance of each series. When the average is calculated from 12 beams (the original number of specimens for each mix design), a solid line is used on the graph and this continues to the point where one or more beams failed. Following this, the curve is completed by using a broken line to connect this point with one more point which represents the average number of cycles at which freezing and thawing was discontinued on the beams, and the average relative E value at the time or removal of the beams from test. These final points are usually

close to 50 percent relative E, since that was the criterion of failure.

(Gravel 3 and Stone 2) Tables 4 through 9 present information concerning differences between the mix designs of the various test series. The av-**F gravel 3 8.6** erage durability factors **and** the results of **G stone 2 78.4** the **Statistical analyses Obtained** after 100, 200, or 300 cycles of freezing and thawing are presented for each series. The information derived from the statistical analyses

is presented in the form of limits, within which the differences of the average durability factors of the two mix designs may be expected to fall at least 95 percent of the time. These limits are listed only for those comparisons that indicated a significant difference at the 5 percent level.

Series 1

Series 1 was undertaken to obtain some basic information concerning the effect of particle size and heavy media treatment on concrete durability. A graphical summary of the freezing and thawing data is shown in Figure 1 where percent relative dynamic modulus of elasticity is plotted against cycles of freezing and thawing. In this series, the lightweight constituents of the gravel were progressively removed by heavy media separation at a specific gravity of 2. 50, starting with the larger sizes and proceeding to the smaller.

In general, Figure 1 indicates that the larger particles of deleterious materials are more harmful to concrete durability than the smaller. This is borne out by the comparisons of durability factors shown in Table 4. At the top of the table is listed the average durability factor for each mix. Differences between mix averages were tested for significance and the results of this analysis are listed in the lower portion of Table 5. Significant differences were found between mix designs A and C, and between designs B and C, but none between C and D or E.

These results and the graphical presentation indicate that in this case, the removal

TABLE 7

DURABILITY OF SERIES 4 (Gravel 4 and Stone 1)

² Asterisk indicates significance at the 5 percent level. Required difference for significance is 5.4.

TABLE 8

DURABILITY OF SERIES 5 (Gravel 5 and Stone 3)

^a Asterisk indicates significance at the 5 percent level. Required difference for significance is 13. 5.

of lightweight particles of sizes smaller than *%-ia.* will not add to the durability of the concrete. Most strongly indicated is the importance of the removal of deleterious particles down to the $\frac{1}{2}$ -in. size. Further work along these lines would seem to be warranted.

Series 2

The results of the tests in Series 2 (gravel 2, stone 1) are shown in Figure 2 and Table 5.

In this series, gravel coarse aggregate from a source on the lower Ohio River was employed. Pavements constructed with this gravel have not performed well. For the past several years, crushed stone from the same geologic formation as stone 1 has been used in combination with this gravel and these tests attempted to measure what improvement in durability might be expected from this combination over the gravel alone.

The results in Figure 2 and in Table 5 indicate that the concrete made with the gravel-

crushed stone combination of design H is considerably more resistant to alternate freezing and thawing than that made with gravel alone. The difference might be even greater than indicated, because the durability of the gravel concrete was almost too low to measure with any degree of accuracy.

The removal of low specific gravity particles from the $\frac{3}{4}$ - to $\frac{1}{2}$ -in. size of gravel in the gravel-crushed stone combination further aided concrete durability. The improvement of designs I and J over design F was almost twice that of design H over design F (Table 5), indicating an advantage gained by heavy media separation. However, as the heavy media separation levels used in this study lowered the chert content of the gravel from 70 percent of total untreated gravel to 40 percent for the treated gravel, it is obvious that a large portion of the $\frac{3}{4}$ - to $\frac{1}{2}$ -in. fraction was removed in this process. With the larger sizes of the aggregate already sufficiently scarce to make necessary the use of crushed stone in their place, the amount of improvement in durability would have to

TABLE 9

DURABILITY OF SERIES 6 (Gravel 6 and Stone 2)

^a Asterisk indicates significance of the 5 percent level. Required difference for significance is 9. 2.

be considerable to make heavy media **in the separation** $\mathbf{MSE} = \mathbf{S} \cdot \mathbf{N} \mathbf{S} \mathbf{S} \mathbf{P} \mathbf{A} \mathbf{A} \mathbf{X} \mathbf{S}$

Series 3

Series 3 is similar to Series 2 in that gravel 3 was actually obtained from the same plant as gravel 2 but was dredged from a different location in the river. However, stone 2 is one which does not have any record of field performance but which is being considered for use with this particular gravel in the future.

The results of the tests performed in Series 3 are shown in Figure 3 and in Table 6.

Figure 5. **Summary of freezing and thawing data - Series** 5.

In this series, the gravel-crushed stone combination (Mix H) produced results similar to those produced by Mix H in Series 2. The somewhat lesser durability of stone 2 might have been a factor accounting for the the slightly lower durability of Mix H in Series 3. However, to counterbalance this, gravel 3 had 44 percent chert compared to over 70 percent chert for gravel 2.

Table 6 shows the average durability factors for the five mixes in Series 3. The analysis of variance was not performed on this series because of lack of homogeneity of the variances and therefore no statement may be made regarding the significance of differences in these means. However, the data appear to be similar to those already discussed in Series 2.

Series 4

Gravel 4 is a material that is dredged from the Ohio River at a point near the southeastern corner of Indiana. Pavements in which this aggregate has been used have good performance records. As was the case for the material used in Series 2, crushed stone from the same geologic formation as stone 1 is often used with gravel 4.

The untreated gravel contained only 10 percent chert, and reference to the graph in Figure 4 and to Table 7 shows this gravel to be durable. The untreated gravel concrete had a durability factor of 79. 3 at 200 cycles of freezing and thawing. When combined with crushed stone, an increase in durability factor to 89. 6 is obtained. Heavy media treatment of this gravel reduced the chert content from 10 to 7 percent but no significant increase in durability was found over that obtained for Mix H, the untreated gravel-stone combination. This is brought out by the comparisons of durability factors shown in the lower half of Table 7.

Series 5

A gravel from a glacial outwash area in north central Indiana was studied in Series 5. This aggregate itself has a fair field performance record. It is used in combination with a crushed stone from a coral reef of Silurian Age, similar to several outcrops which occur across northern Indiana. Although the stone has a high porosity, the voids are large enough so that the aggregate is a durable one, yielding good performance in concrete pavements subjected to freezing and thawing.

The results of the tests made on the five mix designs of Series 5 are presented in Figure 5 and in Table 8. Figure 5 shows that, with the exception of Mix G, the plain stone mix, specimens began to fail between 100 and 150 cycles. The graph also indicates improvement in durability by the substitution of stone for some of the gravel (Mix F versus Mix H) and reference to Table 8 shows that this increase is significant. At 200 cycles of freezing and thawing Mix F had a durability factor of 39. 0 compared to 61. 5 for Mix H. Further comparisons show no difference between Mix H and Mixes I and J, the mixes in which the gravel had been treated. This may be explained by reference to Table 3 in which it can be seen that heavy media treatment made essentially no

Figure 6. Summary of freezing and thawing data - Series 6.

change in the mineralogical composition of this gravel. Benefits from heavy media treatments are not indicated in this case.

Series 6

Gravel 6 is a Wabash River terrace gravel found near the western border of Indiana. Its field performance record is poor. In Series 6, this gravel was tested alone and in combination with stone 2.

Figure 6 and Table 9 report the results from this test series. It can be seen that the addition of stone caused a marked increase in durability of the concrete. The average durability factor at 100 cycles increased from 54. 0 for the gravel alone

 $(Mix F)$ to 71.6 for the gravel-stone combination $(Mix H)$. This trend also continues beyond 100 cycles.

No significant difference was found between the gravel-stone mixture and the two mixes in which the gravel had been treated by heavy media separation, even though the chert content was reduced from 20 percent to 14 percent by heavy media treatment at a specific gravity level of 2. 45.

Variability in the Results

Alternate freezing and thawing tests of concrete made with gravel aggregates are difficult to evaluate. Failure of an individual specimen is brought about by the presence of a sufficient number of deleterious particles. In a single concrete mix there are a given number of these particles, and there are an infinite number of combinations in which these particles may be distributed m the beams. If one beam receives most of the particles, a large specimen variance results. This would be of little importance if the average durability remained valid, but this is not always the case.

Concrete made with gravel 2 had such a large percentage of lightweight chert particles that no specimen to specimen variance resulted. For all four mixes of design F in Series 2, each specimen received enough deleterious material to produce rapid deterioration. Series 5 on the other hand, evinced the most specimen to specimen variance, because with only a limited quantity of deleterious particles, no one beam received exactly its share.

In many instances, one day of mixing produced specimens with durability of a different magnitude than those produced on a second day. Efficiency of vacuum saturation, atmospheric temperature, skill of labor, and many other factors may influence day to day variance. Also, a single mix is a sample from a larger one, and like the specimens can have a shortage or a surplus of the influential aggregate particles.

By including all the variables to be studied in the mixing program on a single day, and repeating the same program on several other days, differences between the variables can be detected without the day to day variation having a significant effect. The problem of the specimen to specimen variance is not so easily solved. In order to use available statistical methods, this variance must at least be uniform. In Series 2, 3, and 5, the data from two of the five mix designs had very low variances, while the other three had uniformly high variances. This meant that the three designs with high variances could be analyzed alone and later be included with the two designs of low variance for the comparisons. This, however, involves the sacrifice of some reliable data whereas if the specimen variance could be controlled, more reliable results would be available.

CONCLUSIONS

The alternate freezing and thawing test in the hands of an experienced engineer, familiar with economics, geography, and the aggregates of the state, could enable him to do a satisfactory job in passing or rejecting an aggregate to be used for construction work. The result of this work indicates that in the future these subjective requirements may not be necessary. Freezing and thawing testing can be used to detect differences between two kinds of concrete. Freezing and thawing tests can be used to benefit both the aggregate producer and the consumer. Poor aggregates must be used when other aggregates are not available, but they must be improved to be of acceptable qualother aggregates are not available, but they must be improved to be of acceptable qual-
ity ... Fach economic agnest must be examined ...including better drainage facilities for ity. Each economic aspect must be examined, including better drainage facilities for pavements built with poor aggregates.
Finally, heavy media separation techniques could be exhaustively studied as a pro-

mising means of improving a poor quality gravel aggregate. Definite information is needed concerning the effect of deleterious particle size in concrete pavements. If treatment of the larger aggregate particles from a gravel source is not feasible because of their shortage, more extensive use should be made of combining durable crushed stone with the gravel. If it is shown that smaller particles are not deleterious, the expense of using only small size particles might be investigated. This may open up the pense of using only small size particles might be investigated. This may open up the
nossibility of erushing all of the larger narticles into smaller ones. eliminating waste possibility of crushing all of the larger particles into smaller ones, eliminating waste of material.
Specific conclusions of this study are:

i

1. Concrete made with the crushed stone-gravel combinations resulted in significant improvement in durability over that made with the gravel aggregate alone.

2. Concrete made with the crushed stone-gravel combinations, where the gravel 2. Concrete made with the crushed stone-gravel combinations, where the gravel
In had noon field norformange, was significantly improved with the heavy media used had poor field performance, was significantly improved with the heavy media
treatments treatments.

3. The durability results of concrete made with gravel aggregates alone compared favorably with the field performance of the aggregates.

4. Results of Series 1 indicated that further improvement of gravel aggregates was not obtained by heavy media treatment of particle sizes of $\frac{3}{4}$ -in. or smaller.

REFERENCES

1. Woods, K. B. , Sweet, H. S. , Shelburne, T. E., "Pavement Blowups Correlated with Source of Coarse Aggregates," Proceedings, Highway Research Board, Vol 25, p. 147 (1946).

2. Soon, A. C., "Concrete Aggregate Study," A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Civil Engineering, Purdue University, 1947.

3. Lewis, D. W., and Woods, K. B, , "Research as Related to the Development of Aggregate Specifications," Proceedings, 34th Annual Road School, Extension Series No. 69 (1949).

4. Sweet, H.S., "Research on Concrete Durability as Affected by Coarse Aggregates," Proceedings, American Society for Testing Materials, Vol 43, p. 988 (1948).

5. Bugg, S. L. , "Effect of Air Entrainment on Durability Characteristics of Concrete Aggregates," Proceedings, Highway Research Board, Vol 27, p. 156 (1947).

6. Blackburn, J. B. , "Freeze and Thaw Durability of Air Entrained Concrete Using Indiana Aggregates," Proceedings, Highway Research Board, Vo) 28, p. 171 (1948).

7. Venters, E., "Determination of the Non-Durable Constituents of Indiana Gravels," A thesis submitted to the faculty of Purdue University in partial fulfillment of the requirements for the degree of Master of Science in Civil Engineering, August, 1951.

8. Lewis, D. W. , "Heavy Liquid Media for Separating Aggregates with Different Durability Characteristics," Proceedings, 38th Annual Road School, Extension Series No. 78 (1952).

9. Sweet, H. S., "Chert as a Deleterious Constituent in Indiana Aggregates," Proceedings, Highway Research Board, Vol 20, p. 599 (1940).

10. Jackson, F.H. , "The Durability of Concrete in Service," Proceedings, American Concrete Institute, Vol 43, p. 165 (1942).

11. Allen, C. W., "Influence of Mineral Aggregates on the Strength and Durability of Concrete," in "Symposium on Mineral Aggregates," Special Technical Publication No. 83, American Society for Testing Materials, (1948).

12. Bartel, F. F., "Effect of Aggregate Characteristics on Durability of Concrete," A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Civil Engineering, University of Maryland, 1942.

13. Wray, F. H. , and Litchtefeld, H. J., "The Influence of Test Method on Moisture Absorption and Resistance of Coarse Aggregate to Freezing and Thawing," Proceedings, American Society for Testing Materials, Vol 40, p. 1007 (1940).

14. Goldbeck, A. T., and Gray, J. E., "A Method of Proportioning Concrete for Strength, Workability, and Durability," Bulletin No. 11, National Crushed Stone Association, (1953).

15. Walker, S., and Bloem, D. L. , "Performance of Automatic Freezing and Thawing Apparatus for Testing Concrete," Proceedings, American Society for Testing Materials, Vol 51, p. 1120 (1951).

16. Walker, S. "Freezing and Thawing Tests of Concrete Made with Different Aggregates," Proceedings, American Concrete Institute, Vol 40, p. 573 (1944).

HRB OR-33

i

THE NATIONAL ACADEMY OF SCIENCES—NATIONAL RESEARCH COUNCIL is a private, nonprofit organization of scientists, dedicated to the furtherance of science and to its use for the general welfare. The **CIL is a private, nonprofit organization of scientists, dedicated to the** furtherance of science and to its use for the general welfare. The **ACADEMY itself** *was* **established in 1863 under a congressional charter signed by President Lincoln. Empowered to provide for all activities appropriate to academies of science, it was also required by its charter to act as an adviser to the federal government in scientific matters. This provision accounts for the close ties that have always existed between the ACADEMY and the government, although the ACADEMY is not a governmental agency.**

The NATIONAL RESEARCH COUNCIL was established by the ACADEMY in 1916, at the request of President Wilson, to enable scientists generally to associate their efforts with those of the limited membership of the ACADEMY in service to the nation, to society, and to science at home and abroad. Members of the NATIONAL RESEARCH COUNCIL receive their appointments from the president of the ACADEMY. They include representatives nominated by the major scientific and technical societies, representatives of the federal government designated by the President of the United States, and a number of members at large. In addition, several thousand scientists and engineers take part in the activities of the research council through membership on its various boards and committees.

Receiving funds from both public and private sources, by contribution, grant, or contract, the ACADEMY and its RESEARCH COUNCIL thus work to stimulate research and its applications, to survey the broad possibilities of science, to promote effective utilization of the scientific and technical resources of the country, to serve the government, and to further the general interests of science.

The HIGHWAY RESEARCH BOARD was organized November 11, 1920, as an agency of the Division of Engineering and Industrial Research, one of the eight functional divisions of the NATIONAL RESEARCH COUNCIL. The BOARD is a cooperative organization of the highway technologists of America operating under the auspices of the ACADEMY-COUNCIL and with the support of the several highway departments, the Bureau of Public Roads, and many other organizations interested in the development of highway transportation. The purposes of the BOARD are to encourage research and to provide a national clearinghouse and correlation service for research activities and information on highway administration and technology.