A STUDY OF CHERT AND SHALE GRAVEL IN CONCRETE

JANUARY 1961

NO. 2

by

R.L. SCHUSTER
J.F MC LAUGHLIN

Joint
Highway
Research
Project

PURDUE UNIVERSITY
LAFAYETTE INDIANA

Technical Paper

A STUDY OF CHERT AND SHALE
GRAVEL IN CONCRETE

TO: K. B. Woods, Director January 25, 1961
Joint Highway Research Project

FROM: H. L. Michael, Assistant Director File: 5-9-6
Joint Highway Research Project Project: C-36-42F

Attached is a technical paper titled "A Study of Chert and Shale Gravel in Concrete" by R. L. Schuster and J. F. McLaughlin. This paper was presented at the Annual Meeting of the Highway Research Board in January 1961.

The material presented in the paper has been previously submitted to the Advisory Board in more detailed form. It was prepared in the attached summary form for publication and dissemination.

The technical paper is presented to the Board for the record and for approval for publication by the Highway Research Board.

Respectfully submitted,

Harold L. Michael

Harold L. Michael, Secretary

HLM:kmc

Attachment

cc: F. L. Ashbaucher
J. R. Cooper
W. L. Dolch
F. F. Havey
G. A. Leonards
J. F. McLaughlin
W. H. Goetz
R. D. Miles
R. E. Mills
C. E. Vogelgesang
J. L. Waling
J. E. Wilson
E. J. Yoder
G. A. Hawkins (M. B. Scott)

Technical Paper

A STUDY OF CHERT AND SHALE GRAVEL IN CONCRETE

by

R. L. Schuster, Associate Professor of
Civil Engineering, University of Colorado
and
J. F. McLaughlin, Research Engineer
Joint Highway Research Project and Associate
Professor of Civil Engineering, Purdue University

Joint Highway Research Project
File: 5-9-6
Project: C-36-42F

Purdue University
Lafayette, Indiana

January 25, 1961

INTRODUCTION

In spite of considerable past study of cherts and shales, there is still much to be learned about the physical properties of these materials and their effects on the durability of concrete.

In the past little has been done to differentiate between cherts and shales of the same general type, but that are obtained from different geographic areas. A purpose of this study was to determine if the basic properties of cherts and shales from one part of Indiana differ significantly from those of cherts and shales from other parts of the state, and if significant differences in the properties of these materials were found, to attempt to determine if these differences also result in differences in durability.

Another objective was to quantify the effects of different chert and shale gravels on the freeze-thaw durability of concrete test specimens containing small percentages of these materials.

The cherts and shales used in this investigation were obtained by hand-picking from glacial gravel deposits widely scattered throughout the state of Indiana. In this way six chert and five shale samples, constituting as widely divergent a group of cherts and shales as could be found in Indiana, were obtained. The geographic locations of the sources for these samples are shown in Figure 1.

FIGURE 1. MAP OF INDIANA LOCATING SOURCES OF CHERT AND SHALE GRAVELS USED IN THIS STUDY

FREEZE-THAW STUDIES OF CONCRETE CONTAINING CHERT AND SHALE GRAVELS

In order to determine the effect of presence of the deleterious materials on freeze-thaw durability of concrete, small percentages of the cherts and shales being studied were incorporated in 3- by 4- by 16-inch air-entrained portland-cement concrete beams made with a standard portland cement, crushed stone coarse aggregate, and natural sand fine aggregate. The aggregates were obtained from sources of proven good quality. The only variables purposely introduced into the experiment were the deleterious materials themselves. The mix design was held constant for all beams made except that varying small percentages of chert or shale were substituted for part of the crushed limestone coarse aggregate in all but the control beams. A water-cement ratio of 0.46 by weight was used throughout the study. This water-cement ratio produced a mix with good workability and a slump of about three inches. The cement factor was kept constant at six bags per cubic yard. An air-entraining agent was used to entrain approximately four per cent air in each batch.

In all beams the coarse aggregate was used in equal amounts of the No. 4 to 3/8-, 3/8 to 1/2-, 1/2 to 3/4-, and 3/4 to 1-inch sizes. Chert or shale was substituted for the 3/8 to 1/2-, 1/2 to 3/4-, and 3/4 to 1-inch crushed stone in two, four, six, and ten per cent blends in all but a few control beams in which the coarse aggregate consisted of 100 per cent crushed limestone. In the beams containing the deleterious materials, no deleterious materials in the No. 4 to 3/8-inch size range were substituted for the crushed limestone because a previous study by Sweet (1) has shown that deleterious particles passing a 3/8-inch screen have little effect on the freeze-thaw durability of concrete. All the coarse aggregate was vacuum saturated before mixing. The fine aggregate was not vacuum saturated, but was mixed with enough water to fill all surface-connected pores and left in this condition for 24 hours before mixing.

Mixing was accomplished by means of a modified food mixer with 1/4-cubic-foot capacity. The concrete was molded into 3- by 4- by 16-inch beams, and these were cured by immersion in lime water for 13 days following removal of the specimens from the molds one day after casting.

Freeze-thaw testing of the beams was conducted according to the ASTM Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water, ASTM Designation C 291-57 T (2). The end point adopted for this series of tests was 50 per cent relative dynamic modulus of elasticity or 300 cycles of freezing and thawing, whichever occurred first.

A durability factor was determined for each beam by the method shown in ASTM Designation C 291-57 T. These durability factors provided an index of the amount of deep-seated deterioration taking place in each beam.

During the freeze-thaw testing program, a number of popouts occurred on the surfaces of some of both the chert and shale beams. These popouts often occurred on beams which showed no deep-seated failure such as would be evidenced by low durability factors. Since these popouts were all found to be caused by failure of pieces of chert and shale during the freeze-thaw testing, this was further studied. During the freeze-thaw testing program, visual observation of any new popouts was made each time a beam was tested for its fundamental transverse frequency, i. e., every 10-20 cycles of freezing and thawing. The position of each popout was noted as well as the approximate size of the piece of deleterious material causing it.

In order to compare the relative severity of surface deterioration of the beams, it was necessary to determine an index number for each beam which would give an indication of the relative popout damage suffered by that beam. An arbitrary numerical index based on sizes of the deleterious particles causing the popouts, number of popouts, and numbers of cycles at which the popouts occurred was developed and can be explained as follows:

$$\text{SDF} = \frac{s_1}{c_1} + \frac{s_2}{c_2} + \frac{s_3}{c_3} + \ldots\ldots\ldots + \frac{s_n}{c_n}$$

where

SDF = surface durability factor for each beam,

s = size factor, 1 for popouts caused by deleterious particles 3/8 to 1/2 inch in size, 2 for popouts 1/2 to 3/4-inch in size (average of 2 diameters),

c_1 = cycle factor, 1 for cycles 1 to 100, 2 for cycles 101 to 200, 3 for cycles 201 to 300.

For beams whose relative moduli of elasticity dropped below 50 before 300 cycles of freezing and thawing were attained, this arbitrary equation does not result in an index that can be compared with beams undergoing the full 300 cycles. In many cases the beams that were removed from the freeze-thaw test before 300 cycles would have suffered additional surface deterioration if they had been allowed to reach 300 cycles. It is difficult to devise a correction factor which would satisfactorily eliminate this failing of the equation. However, only a few of the beams tested fall in this category, and these were especially noted in tabulating the data so that no direct comparisons would be made.

Chert Studies

An experimental outline was set up in which three variables were introduced into the production of beams containing chert with all other controlled factors remaining constant. The three variables were:

(a) Source of Chert. Material from each of the six sources of chert from throughout the State of Indiana was used.

(b) Specific Gravity of Chert. The chert from each of the six sources was separated into three groups based on bulk specific gravity (saturated surface-dry basis). The specific gravity ranges selected for these groups were 2.55 plus, 2.45-2.55, and 2.45 minus. Separation was accomplished using mixtures of carbon tetrachloride (specific gravity 1.58) and acetylene tetrabromide (specific gravity 2.97). Beams were made containing chert from each source and at each level of specific gravity.

(c) Percentage of Chert. Chert from each combination of source and specific gravity group was included with the crushed stone coarse aggregate in the beams in amounts of two, four, and ten per cent, and a six per cent level was included for chert from the 2.45 minus specific gravity group.

Statistical analysis of durability factors obtained from freeze-thaw studies of the chert beams (Table 1) indicated that the sources of the chert had no effect on resistance of concrete to deep-seated freeze-thaw deterioration. Even though the cherts were from different sources throughout Indiana, they resulted in nearly equal

Table 1

Summary of Individual Durability Factors for Freeze-Thaw Testing Program of Concrete Beams Containing Small Percentages of Chert Course Aggregate

Per Cent Chert in Course Aggregate		Chert Source																	
		2063			2064			2066			2067			2072			2077		
		Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range		
		2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus
2%		97.5	98.2	94.1	98.2	98.2	96.4	98.8	97.3	89.1	97.9	96.9	96.1	98.2	97.0	93.8	98.2	98.2	94.6
		98.3	99.7	92.0	98.1	97.4	93.6	97.9	97.3	96.7	98.9	98.0	98.2	99.0	97.0	97.2	97.8	96.5	95.3
	Avg.	97.9	99.0	93.1	98.2	97.8	95.0	98.4	97.3	92.9	98.4	97.5	97.2	98.6	97.0	95.5	98.0	97.4	95.0
4%		99.8	98.3	88.9	98.2	97.3	95.5	98.0	96.5	95.5	97.7	96.4	95.5	99.0	94.8	96.5	97.2	97.3	91.5
		96.6	97.2	96.4	99.0	96.4	98.0	98.0	94.7	95.6	97.7	95.4	95.5	97.0	97.3	94.9	97.0	97.4	95.3
	Avg.	98.2	97.8	92.7	98.6	96.9	96.8	98.0	95.6	95.6	97.7	95.9	95.5	98.0	96.0	95.7	97.1	97.4	93.4
6%				96.3			94.7			88.7			84.5			92.1			87.8
				78.1			97.2			89.7			95.6			90.0			68.9
	Avg.			87.2			96.0			89.2			90.1			91.1			78.4
10%		98.2	71.0	24.4	99.1	82.5	11.2	97.0	93.5	29.6	99.1	83.2	57.3	98.9	92.0	24.7	97.9	96.3	30.0
		98.2	94.7	29.5	96.4	98.0	25.0	98.0	95.5	46.5	97.2	94.6	38.4	98.9	91.9	38.3	97.2	92.9	26.4
	Avg.	98.2	82.9	27.0	97.8	90.3	18.1	97.5	94.5	38.1	98.2	88.9	47.9	98.9	92.0	31.5	97.6	94.6	28.2

degrees of freeze-thaw deterioration when used in concrete in equal amounts having the same specific gravity ranges.

A definite difference in durability factors was found, however, for beams containing cherts of different specific gravity ranges. It was found that beams containing chert from the 2.45 minus specific gravity group had significantly lower durability than those containing chert from the 2.55 plus and 2.45-2.55 specific gravity ranges. This is in accord with the work of Sweet and Woods (3) which indicated low-specific gravity chert to be the most susceptible to freeze-thaw deterioration.

The percentage of chert used also had a significant effect on the durability factors, but only at the 2.45 minus specific gravity level. At the 2.55 plus and 2.45-2.55 specific gravity levels, there was no significant difference among the durability factors of the beams containing different percentages of chert. Even ten per cent of chert from these specific gravity groups caused no deep-seated failure of beams in which it was included.

In the 2.45 minus specific gravity level the percentage of chert had a strong effect on durability of the concrete. Without exception, beams containing ten per cent of 2.45 minus chert suffered severe deep-seated deterioration. Every beam was intersected by at least one deep-seated crack caused by failure of the chert. The lowest durability factors recorded in the freeze-thaw testing occurred for this combination.

At the six per cent level of the 2.45 minus specific gravity group, no deep-seated cracks occurred and the durability factors were found to be not significantly lower than those of the two and four per cent levels of this gravity range. However, more variability in the data occurred at this level than at the two and four per cent levels, i.e., individual beams containing six per cent of 2.45 minus chert had durability factors as low as 68.9 and 78.1 while others were as high as 97.2. The few low durability factors at the six per cent level, while not nearly as low as those at the ten per cent level and not low enough to cause significant differences in the cell means, were low enough to suggest that some deterioration can occur in concrete containing as little as six per cent chert with a bulk specific gravity of less than 2.45.

Durability factors for beams containing two and four per cent of 2.45 minus chert were shown by analysis of variance to be significantly lower than durability factors for beams containing the same percentages of chert from the two heavier specific gravity ranges. However, all the durability factors for these beams are high enough to indicate very little deep-seated deterioration (Table 1). For example, the lowest durability factor computed for the four per cent level of the 2.45 minus specific gravity range was 88.9 and the lowest cell mean was 92.7. These values are high enough to be considered indicative of sound concrete.

In summary, it appears that concrete containing up to four per cent chert with a bulk specific gravity (saturated surface-dry basis) of 2.45 or less, and at least ten per cent chert with a specific gravity greater than 2.45, can successfully withstand laboratory freeze-thaw exposure without undergoing deep-seated deterioration.

The effect of size of the individual chert particles on their freeze-thaw durability in concrete is of interest. Previous studies of deleterious substances have indicated a relationship between size of unconfined particle and lack of freeze-thaw durability. Wray and Lichtefeld (4) found in their study of Missouri cherts that saturated 1 to 1 1/2-inch particles had less resistance to freezing-and-thawing failure than saturated 3/4 to 1-inch particles. Thomas (5) saturated prisms of different sizes from the same rock and found that damage was greater the larger the specimen.

The effect of size on the durability of particles of deleterious materials in concrete is not so clear, however. Sweet and Woods (5) embedded saturated chert pieces of three sizes, 3/4 to 1 inch, 1/2 to 3/4 inch, and 3/8 to 1/2 inch, in 1-inch mortar cubes and subjected these cubes to up to 309 cycles of freezing and thawing. They found that the cubes failed at an earlier cycle for the 3/4 to 1-inch pieces than for the 1/2 to 3/4-inch pieces, and that no failure occurred in the cubes containing the 3/8 to 1/2-inch pieces. Flieger (6), however, found no apparent relationship between size of unsound aggregate particles and durability as long as the air content of the mortar was held constant. R. Walker and McLaughlin (7) demonstrated that lightweight chert less than 3/8-inch in size would not cause deep-seated freeze-thaw deterioration in concrete, but their method of test did not distinguish between the degrees of resistance to freeze-thaw deterioration exhibited by different sizes of chert larger than 3/8-inch.

In this study no attempt was made to determine the effect of size on durability. However, for those beams that had suffered deep-seated cracking as a result of freezing and thawing, a qualitative study was conducted to determine the sizes of the pieces of chert intersected by each crack. In each case it appeared that the 3/4 to 1-inch piece had provided most of the disruptive force. In no case was a crack caused by 1/2 to 3/4- or 3/8 to 1/2-inch pieces alone.

This, of course, does not mean that 1/2 to 3/4- or 3/8 to 1/2-inch pieces could not cause deep-seated failure of concrete, but that they are not as harmful as the larger pieces. Larger chert particles in concrete appear to have less resistance to freeze-thaw deterioration than smaller ones.

Although freeze-thaw testing of concrete specimens is primarily intended to cause deep-seated failure and subsequent loss of strength of concrete specimens containing unsound aggregates, surface deterioration of the concrete, which can be equally as important as deep-seated failure, often occurs in these tests. A part of this study was to determine how surface deterioration is influenced by each of the variables introduced into the freeze-thaw study.

Surface deterioration factors for the chert beams are presented in Table 2. These data indicate no significant difference in severity of popout damage among the six chert sources. The major differences in severity of surface deterioration apparently were caused by material from different bulk specific gravity ranges. For all six cherts a negligible amount of surface deterioration occurred in beams containing material from the 2.55 plus and 2.45-2.55 specific gravity ranges. Material from the 2.45 minus gravity range, however, resulted in a significant amount of popout damage in beams made from each of the six cherts. In general, the six to ten per cent levels within the 2.45 minus specific gravity range had higher surface deterioration factors than the two and four per cent levels, but this was not true for all six cherts. For example, in the 2.45 minus specific gravity range for chert 2077, the four per cent level had an average surface deterioration factor of 1.9, while the six per cent level had an average factor of only 0.2. This seeming anomaly is probably due more

Table 2

Summary of Surface Deterioration Factors for Freeze-Thaw Testing Program of Concrete Beams Containing Small Percentages of Chert Coarse Aggregate

Per Cent Chert in Coarse Aggregate		Chert Source																	
		2063			2064			2066			2067			2072			2086		
		Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range		
		2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus
[illegible]	Avg.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	Avg.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	Avg.			[illegible]			[illegible]			[illegible]			[illegible]			[illegible]			[illegible]
[illegible]	Avg.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

* Removed from freeze-thaw test at less than 300 cycles

to random positioning of the pieces of chert than to any error in procedure or real differences in the material.

It also should be noted that, in most cases, larger factors were obtained for beams in the six per cent level of the 2.45 minus specific gravity range than in the ten per cent level. This was primarily due to failure of most of the ten per cent beams to complete a full 300 cycles of freezing and thawing while all the six per cent beams lasted the full 300 cycles. Thus the ten per cent specimens were not exposed to as many cycles of freezing and thawing as those containing six per cent chert.

In summary, surface deterioration in the beams containing chert paralleled the deep-seated failure of these beams. In both cases the different sources had little, if any, effect. For all sources failure occurred primarily in the six and ten per cent levels of the 2.45 minus specific gravity range.

Shale Studies

An experimental outline was formulated in which two variables were introduced into design of the concrete beams containing shale with all controlled factors remaining constant. This experimental design differed from that of the chert study in that no specific gravity separation of the shale was made. It was set up as a two-way crossed classifica tion. The two variables in the design were:

(a) Source of shale. Material from each of the sources was blended with the crushed stone coarse aggregate in different beams.

(b) Percentage of shale. Shale from each of the sources was combined with the crushed stone coarse aggregate in blends of two, four, six, and ten per cent.

Study of the durability factors for concrete beams containing shale (Table 3) indicates that no combination of sources and percentages (up to ten per cent) of shale resulted in deep-seated failure of the beams. Only a few beams were found to have durability factors below 90, and these few values appear to be well distributed throughout the data. Only one cell mean is below 90 and this is at the four per cent level, while the durability factors at the six and ten per cent levels for this same shale (2066) are well above 90. This indicates that the low mean for the four per cent level is probably due to random error.

Thus it appears from these data that in amounts of up to ten per cent, little difference in the resistance of concrete to deep-seated deterioration was caused by the different shales even though they were from widely separated areas throughout the state and had significantly different basic properties. This is in accord with the findings of Lang (8) who noted that for pavement concrete containing small percentages of shale, the only harmful effect of the shale due to freezing and thawing consisted of surface deterioration of the concrete.

A comparison of durability factor data for the cherts and shales shows that the only deep-seated deterioration caused by either of these materials was due to chert with a bulk specific gravity (saturated surface-dry basis) of less than 2.45. Since some of the shale samples contained a considerable quantity of material which is of low bulk specific gravity even for shale (2.15 minus or 2.25 minus), and since none of these shales produced any deep-seated failure, specific gravity apparently does not have the same relationship to resistance to deep-seated failure for shales as it does for cherts.

Table 3

Summary of Individual Durability Factors for Freeze-Thaw Testing Program of Concrete Beams Containing Small Percentages of Shale Coarse Aggregate

Per Cent Shale in Coarse Aggregate		Shale Source				
		2063	2066	2068	2075	2076
2%		100.0	97.8	99.5	96.5	96.0
		98.8	96.0	85.5		96.5
		92.0	92.8	94.0	88.5	96.0
			94.5		97.2	95.4
			95.3		97.2	95.8
					98.2	96.3
	Avg.	97.0	95.3	93.0	95.5	96.0
4%		96.3	96.5	94.3	96.4	96.3
		85.3			96.4	94.0
		97.1	90.8		97.2	97.2
			80.5	97.1		93.0
				96.5		96.0
			87.2	95.3		99.5
	Avg.	92.9	88.8	95.8	96.7	96.0
6%		99.0	97.4		98.5	99.8
		98.2	92.5	98.4	87.0	89.0
		96.0	98.2		96.5	100.0
		99.0	96.3	87.9		97.0
		98.7	87.6	85.1		89.0
		100.0	96.5			88.4
	Avg.	98.5	94.8	90.5	94.0	93.9
10%		95.5	87.0			90.8
		86.5	96.5	83.0	98.0	95.4
		95.0			96.2	95.5
			97.0	95.3	96.4	
			96.0	93.5	98.2	
			96.2	91.6	96.4	
	Avg.	92.3	94.5	90.9	97.0	93.9

Surface deterioration factors for the shale beams are presented in Table 4. It is evident from these data that shale 2068 caused considerably more popout damage than any of the other shales. Beams containing shale 2068 had higher surface deterioration factors than beams containing the other shales at every percentage level, and shale 2068 was the only shale to cause even a single popout in beams containing two to four per cent of this material.

Only a small difference in performance relative to popout damage could be detected among the other four shales. No surface deterioration occurred for any of these shales when used in amounts up to and including four per cent. At the six to ten per cent levels, shales 2066 and 2076 caused little more popout damage than shales 2063 and 2075, but the difference is slight.

Comparison of surface deterioration factors for the cherts and shales indicates that, except for sample 2068, the shales caused about the same amount of surface deterioration as cherts of the 2.45-2.55 specific gravity range. In general, the shales caused greater popout damage than cherts of the 2.55 plus specific gravity range and lesser damage than cherts of the 2.45 minus gravity range. Shale 2068, however, resulted in more severe surface deterioration than any of the cherts of any specific gravity range.

Comparison of Air Void Parameters and Durability of Concrete Mixed by Hand and by Machine

During the early stages of the freeze-thaw study of concrete beams containing shales, a few beams were prepared from hand-mixed concrete to see how their durabilities would compare with those of the regular beams from machine-mixed concrete used in the freeze-thaw testing program. It was found that in

Table 4

Summary of Surface Deterioration Factors for Freeze-Thaw Testing Program of Concrete Beams Containing Small Percentages of Shale Coarse Aggregate

Per Cent Shale in Coarse Aggregate			Shale Source				
			2063	2066	2068	2075	2076
2%			0.0	0.0	2.0	0.0	0.0
			0.0	0.0	2.0		0.0
			0.0	0.0	2.0	0.0	0.0
				0.0		0.0	0.0
				0.0		0.0	0.0
						0.0	0.0
		Avg.	0.0	0.0	2.0	0.0	0.0
4%			0.0	0.0	0.0	0.0	0.0
			0.0			0.0	0.0
			0.0	0.0		0.0	0.0
				0.0	6.2		0.0
					0.3		0.0
				0.0	1.		0.0
		Avg.	0.0	0.0	2.0	0.0	0.0
6%			0.0	0.0		0.0	1.0
			0.0	0.5	4.2	0.0	0.0
			0.0	0.3		1.0	1.0
			2.0	2.0	0.3		0.0
			0.0	0.0	2.5		0.0
			2.0	0.0			1.0
		Avg.	0.7	0.5	2.3	0.3	0.5
10%			0.0	0.5			4.0
			0.0	1.0	1.2	0.0	3.3
			0.0			1.0	0.3
				0.0	5.9	0.0	
				0.0	3.0	1.0	
				0.0	8.5	0.0	
		Avg.	0.0	0.3	4.7	0.4	2.5

nearly all cases the hand-mixed concrete had lower durability factors than machine-mixed concrete of identical composition. It was felt that this presented an opportunity to study the air-void parameters of these two classes of concrete in an attempt to explain the observed differences.

By means of the linear traverse technique outlined by Fears (9), the total percentage of entrained and entrapped air was determined for each beam. Using the values obtained for total percentage of air and voids per inch of traverse, it was possible to compute specific surface areas and bubble spacing factors for the beams using Powers' method (10). Powers theorized that the increase in durability afforded concrete by means of air entrainment is largely a function of the spacing of the air voids in the concrete. He suggested that a concrete containing air voids with high specific surface area, and thus with a low void spacing factor, would receive more protection from the air voids than one containing air bubbles having a low specific surface area and a high spacing factor. He considered a void spacing of about 0.01 inches to be critical; those concretes with spacing factors lower than 0.01 inch were thought to be well protected from freezing-and-thawing deterioration; those with spacing factors greater than 0.01 inches were thought to be poorly protected.

This theory is supported by the results of the linear traverse studies (Table 4a and Figure 2). For the 14 beams studied, it was found that those having high durability factors (83 and above) all had spacing factors below 0.01 and those with low durability factors (21 and below) had spacing factors above 0.01. Also, the specific surface areas of the beams with high durability factors were all higher than those with low durability factors.

Table 4A

Results of Air Void Studies of Certain Concrete
Beams by Means of the Linear Traverse Technique

Beam Number and Description	Durability Factor	% Air	Voids per Inch	Calculated Specific Surface Area of Voids (sq.in./cu.in.)	Calculated Void Spacing Factor (Inches)
S6-3 (4% Shale #2063, machine mixed)	97.1	3.3	4.2	506	.0080
S6-6 (4% Shale #2063, hand mixed)	21.1	4.1	2.9	287	.0131
S8-6 (10% Shale #2063, machine mixed)	95.0	3.8	4.8	512	.0075
S8-1 (10% Shale #2063. hand mixed)	6.0	3.8	2.0	208	.0185
S9-5 (10% Shale #2068, machine mixed)	83.0	2.7	3.5	523	.0088
S9-1 (10% Shale #2068, hand mixed)	8.3	3.3	2.7	324	.0126
S20-2 (10% Shale #2066, machine mixed)	96.5	3.5	3.8	441	.0091
S20-5 (10% Shale #2066, hand mixed)	14.2	3.0	2.0	269	.0165
S21-2 (6% Shale #2075, machine mixed	72.4	3.0	3.8	512	.0083
S21-6 (6% Shale #2075, hand mixed	10.6	3.1	2.1	273	.0154
S22-1 (10% Shale #2076, machine mixed)	90.8	2.8	3.2	451	.0098
S22-4 (10% Shale #2076, hand mixed)	30.1	2.7	2.2	326	.0139
S23-3 (10% Shale #2075, machine mixed)	96.2	3.1	5.0	637	.0066
S23-4 (10% Shale #2075, hand mixed)	5.3	3.8	3.0	316	.0121

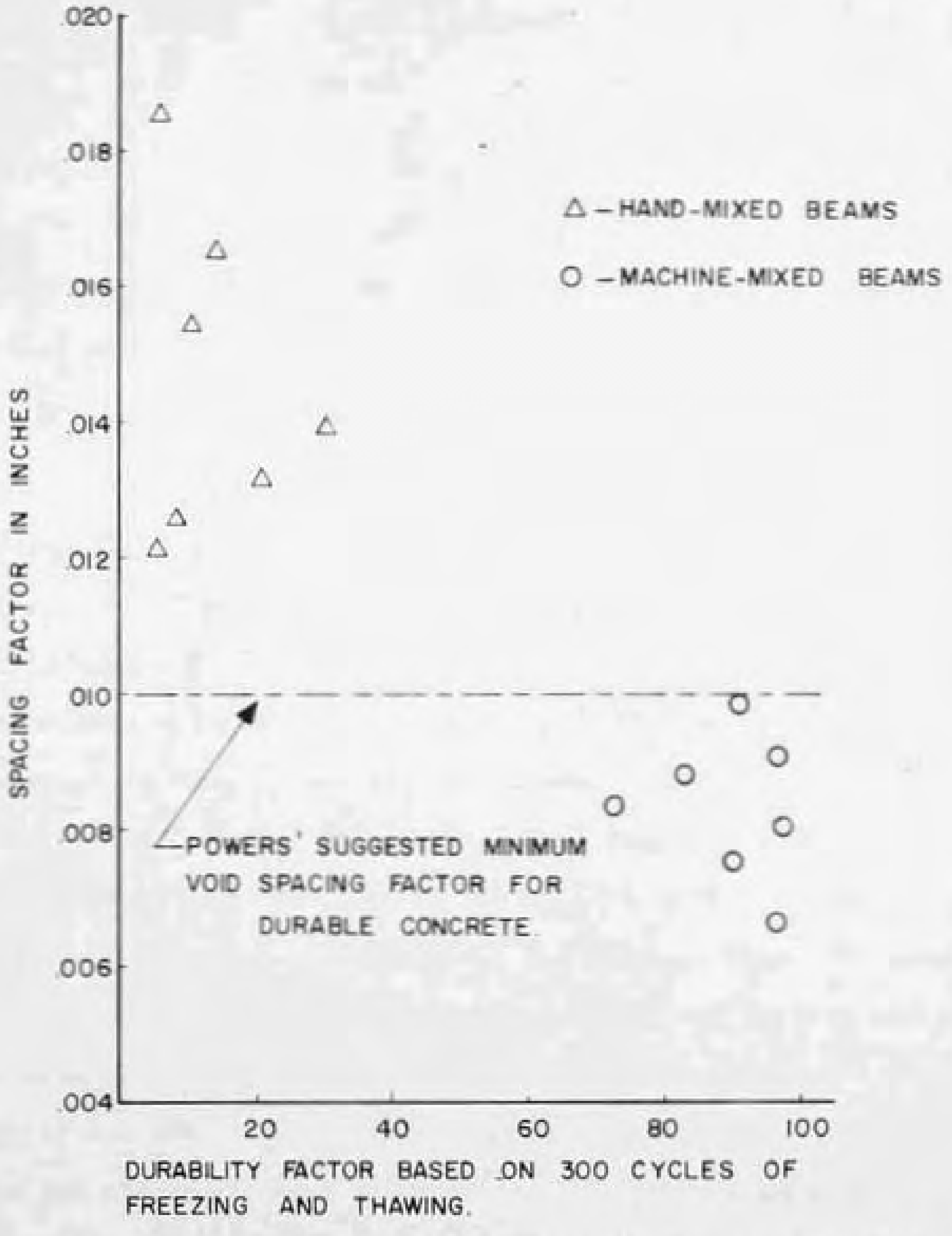

FIGURE 2 RELATIONSHIP OF DURABILITY FACTORS TO VOID SPACING FACTORS FOR AIR-ENTRAINED CONCRETE

INFLUENCE OF BASIC PROPERTIES OF THE CHERTS AND SHALES ON FREEZE-THAW DURABILITY

The primary objective of this portion of the study was to study the basic properties of cherts and shales in Indiana's gravel aggregates and to determine how these properties affect the freeze-thaw durability of these materials. The properties discussed are porosity, absorption, mineralogy, and texture and microstructure.

Porosity

Much work relating porosity and durability of crushed limestone aggregates has been done by Sweet (11) and Fears (12). Early studies by Cantrill and Campbell (13), Wuerpel and Rexford (14), and Sweet and Woods (3) correlated porosity and durability for cherts. However, little correlation of this type has been attempted for shales, and the chert studies mentioned were made before air-entrained concrete had come into use and before freeze-thaw testing had reached its present state of development. In addition, little was done in a quantitative sense in these previous studies. For example, it was determined in a qualitative way that lightweight chert causes deterioration when used in concrete exposed to freezing-and-thawing, but nothing has been done to determine the <u>quantity</u> of this material required to cause deterioration.

Since porosity is so important in the freezing-and-thawing durability of concrete aggregates, several studies of voids in the cherts and shales were made. Total volume of voids and volume of voids less than and greater than 5 microns in diameter were determined for the three specific gravity groups of cherts 2067 and 2077, and total porosity was determined for the five shale samples.

Total Porosity

Total porosity was calculated by means of the following relationship between bulk and true specific gravity:

$$n = \frac{V_v}{V} = 1 - \frac{S_b}{S_t}$$

where

n = porosity,

v = total volume,

V_v = volume of voids,

S_t = true specific gravity,

S_b = bulk specific gravity.

The total porosity of chert is inversely related to its bulk specific gravity (a more easily measured characteristic than porosity) for materials of the same true specific gravity, and as such is generally reflected in specifications for chert in concrete aggregate. It has been found that the most porous cherts (those with the lowest bulk specific gravities), cause the most severe freeze-thaw deterioration.

Freeze-thaw studies of concrete beams containing chert showed that significant deep-seated and surface deterioration took place only in beams containing six to ten per cent of material from the 2.45 minus specific gravity group. For the samples tested, the porosity of chert in this specific gravity group was nearly 13 per cent (Table 5). Chert from the 2.45-2.55 and 2.55 plus specific gravity groups, which caused very little freeze-thaw deterioration, had porosities of only about seven and three per cent respectively. This relationship definitely supports the ideas of Wuerpel and Rexford (14) and Sweet and

Woods (3) that cherts with high porosity are more susceptible to freeze-thaw deterioration than those with low porosity, and further demonstrates that this concept holds for air-entrained concrete as well as concrete with no entrained air. It also suggests that the 2.45 bulk specific gravity level (saturated surface-dry basis) suggested by Sweet and Woods as the critical level of separation between unsound chert and durable chert is realistic even for air-entrained concrete.

The lack of protection afforded porous aggregates such as these lightweight cherts by air-entrained cement paste has been explained in general terms by Powers (15). When saturated aggregate particles surrounded by air-entrained cement paste are subjected to freezing, the water in the paste is able to move to the "escape boundaries" provided by entrained bubbles in the paste, and no excess hydraulic pressures are able to develop. Thus the paste itself is protected from dilation. However, saturated porous rock particles enclosed by the paste still perform as virtually enclosed containers and are only a little better off than if the paste were not air-entrained. Probably the paste bubbles near the contact between aggregate particle and paste do accept a small amount of the excess water produced by freezing the saturated aggregate, but for saturated aggregates of high porosity, the amount of excess water is too large to be taken on by the bubbles in the paste immediately adjacent to the aggregate. For this reason, protecting the paste by air-entrainment, while possibly successful for aggregates of low porosity, fails to protect saturated highly porous aggregate particles. This concept helps to explain the findings of Axon, Willis, and Reagel (16) who noted that the entrainment of air resulted in a definite improvement in durability of concrete containing limestones with good service records, but only caused a slight increase in durability for concrete made with chert-rich aggregate with a fair service record, and affected no appreciable improvement in durability of concrete made with chert-rich aggregate with a poor service record.

As shown in Table 6, the porosities of the different shales varied widely. For example, shale 2068, which was the softest and weakest of the shales, was nearly twice as porous as any of the other shales, and over five times as porous as shale 2063, the least porous and the most-indurated of the samples. The widely varying porosities of the shales had no effect on the amount of deep-seated freeze-thaw deterioration caused by these materials, however. In amounts up to ten per cent, none of the shales caused any deep-seated damage to the concrete in which they were used. This lack of deep-seated deterioration of concrete containing shales of relatively high porosity was due to the inherent structural weakness of these materials. Since the shale is considerably weaker than the surrounding mortar, it will fail internally due to the pressures developed in freezing rather than disrupt the mortar. This was demonstrated by comparison between pieces of chert and shale that had failed in the freeze-thaw test and subsequently had been removed from the beams. The chert pieces, which had caused deep-seated deterioration, had broken into many pieces, but the individual pieces were still relatively hard and firm. The shales, on the other hand, had disintegrated into weak crumbly masses although they had caused no deep-seated deterioration. From all appearancesthese shales had failed internally before the pressures due to freezing could develop enough to break the surrounding mortar.

Table 6

Total Porosities of Shale Samples

Source	Bulk Specific Gravity	True Specific Gravity	$\frac{S_b}{S_t}$*	Porosity, n (per cent)
2063	2.28	2.38	0.958	4.2
2066	2.06	2.39	0.862	13.8
2068	2.00	2.58	0.775	22.5
2075	2.24	2.45	0.914	8.6
2076	2.08	2.47	0.842	15.8

* $n = (1 - \frac{S_b}{S_t})100$

Where an individual shale particle occurred close to the surface of a beam, the enclosing mortar layer was often not strong enough to resist the hydrostatic pressures developed by freezing the saturated particle. In this case the mortar was disrupted, resulting in surface deterioration in the form of a popout or pit. The relative porosities of the shales had a marked relationship to severity of surface deterioration. Shale 2068, the material having the highest porosity of the group (Table 6), caused considerably more popout damage than any of the other shales or any of the cherts. It appears that this larger amount of deterioration is related to the greater porosity of shale 2068, but, as will be brought out in a succeeding section, other factors such as the size of the pores and the amount of absorption of the shale probably have a greater effect on the durability of the aggregate than the total porosity.

It should be noted that among the other four shales the relationship between porosity and surface deterioration is not so clear. Shales 2076 and 2066 are considerably more porous and more absorptive than 2063 and 2075, yet caused only a little more popout damage than 2063 and 2075.

In summary of the relationship of total porosity to freeze-thaw deterioration of concrete containing cherts and shales, the following points should be brought out:

(a) Although other pore characteristics such as pore size and absorptivity may have a strong influence on the resistance of the cherts tested to both deep-seated and surface deterioration, there is a definite relationship between total porosity and the freeze-thaw resistance of these materials. The more porous fractions from all six chert groups caused more freeze-thaw deterioration than the less porous material.

(b) Total porosity of shales was related to severity of surface deterioration of concrete in which the shales were used, but in spite of widely varying porosities, none of the shales resulted in deep-seated deterioration of concrete in which they were used in amounts up to ten per cent. Shale 2068, which was considerably more porous than the other shales, caused much more surface deterioration than the others but caused no deep-seated deterioration.

Size of Pores

Although recognizing a relationship between total porosity and freeze-thaw durability of aggregates, Sweet (1) and Fears (12) have contended that the durability of aggregates is dependent more on the size and continuity of aggregate pores than on total porosity. Lewis and Dolch (17) maintained that the harmful pore size is that large enough to permit water readily to enter much of the pore space but not large enough to permit easy drainage. Studies by Sweet (1), and Fears (12) have indicated that critical pore size for freezing-and-thawing durability of limestone aggregates is about 5 microns. Blanks (18) has shown that, under natural conditions of freezing and thawing, voids less than 5 microns in diameter, and particularly those less than 4 microns in diameter, will drain effectively only at hydrostatic pressures that exceed the tensile strengths of some rocks and concrete.

These previous investigations indicate the importance of microvoids (pores less than 5 microns in diameter) in the durability of aggregates. Therefore part of the present study was devoted to determining if this relationship between pore size and durability holds for Indiana cherts. For cherts 2067 and 2077, the percentage of total volume of aggregate occupied by voids greater than 5 microns in diameter was determined by a linear traverse study of polished surfaces and this percentage was subtracted from the total porosity to obtain the percentage of total aggregate volume occupied by microvoids.[1] The linear traverse technique used was similar to that reported by Fears (9) for study of air-voids in hardened concrete. Recording of traverse lengths was accomplished by means of a Hunt-Wentworth recording micrometer of the type commonly used for micrometric mineralogical analyses.

The results of the pore size studies are shown in Table 7. It was found that the volume of microvoids was somewhat less than expected. Sweet (1) had noted that in Indiana limestone aggregates the volume of microvoids, expressed as a ratio of the total volume, was less than 0.057 for aggregates with with good field performance records and greater than 0.091 for aggregates with poor service records. If Sweet's criteria were to be applied to the chert fractions whose void ratios are presented in Table 7, it would seem that none of the material in these fractions would be susceptible to freeze-thaw deterioration since none of this material has microvoid ratios[2] as high as 0.091. The

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[1] As used here, the term "microvoids" refers to voids less than 5 microns in diameter.

[2] As used here the term "microvoid ratio" refers to the ratio of volume of voids less than five microns in diameter to the bulk volume of the aggregate.

Table 7

Relation of Pore Size to Degree of Saturation for Cherts 2067 and 2077

Source	Specific Gravity Range	Total Porosity (per cent)	Per Cent of Bulk Volume Consisting of Voids > 5 Microns in Diameter	Per Cent of Voids Volume Consisting of Voids > 5 Microns in Diameter	Per Cent of Bulk Volume Consisting of Voids < 5 Microns in Diameter	Per Cent of Voids Volume Consisting of Voids < 5 Microns in Diameter	Degree of Saturation (per cent)
2067	2.55 plus	3.0	0.6	20.0	2.4	80.0	82.3
	2.45-2.55	7.6	1.9	25.0	5.7	75.0	92.5
	2.45 minus	12.9	6.5	50.4	6.4	49.6	100.0
2077	2.55 plus	3.0	0.4	13.3	2.6	86.7	78.3
	2.45-2.55	6.4	2.3	35.9	4.1	64.1	87.7
	2.45 minus	12.8	7.9	61.7	4.9	38.3	90.5

highest ratio, 0.064 for the 2.45 minus specific gravity group of chert 2067, is only slightly higher than the 0.057 ratio designated as the upper limit for aggregates with good service records. In spite of these relatively low microvoid ratios, the 2.45 minus specific gravity chert from sources 2067 and 2077 caused serious freeze-thaw deterioration in concrete in which it comprised ten per cent of the coarse aggregate.

It also should be noted that practically no freeze-thaw deterioration occurred in concrete containing chert from the 2.45-2.55 specific gravity range even though this material contained nearly as large a ratio of microvoids as did chert from the 2.45 minus specific gravity group. In spite of the lack of difference in volume of microvoids between the 2.45-2.55 and 2.45 minus specific gravity ranges, there is considerable difference in total porosity between these ranges. As shown in Table 7, the high total porosity of the 2.45 minus chert as compared to the 2.45-2.55 material is primarily due to the increase in voids larger than 5 microns in diameter in the 2.45 minus material. Table 7 shows that as the total porosity of the chert increases in going from material of high to low bulk specific gravity, the voids larger than 5 microns in diameter constitute an increasingly larger percentage of the total pore space, and conversely, the microvoids make up an increasingly smaller percentage of the pore space. For example, for chert 2067, the microvoids constitute 80 per cent of the total pore space in the 2.55 plus chert, 75 per cent in the 2.45-2.55 material, and only 50 per cent in the 2.45 minus range.

On the basis of this study, it appears the microvoids ratio is not as reliable an indicator of freeze-thaw durability of chert aggregate as Sweet (1) and Fears (12) found it to be for limestone aggregate. The results of this study indicate that some other pore characteristic or, more probably, a combination of characteristics (perhaps including volume of microvoids) is probably the main factor in determining chert durability.

As shown in Table 7, the degree of saturation of the cherts increases with increasing total porosity and decreasing bulk specific gravity. This increase is probably caused by the larger percentage of voids larger than 5 microns in diameter in the more porous material. Under conditions of vacuum saturation and frequently repeated immersion as used in these tests, a piece of aggregate containing numerous large voids as well as microvoids would probably reach a high degree of saturation more readily than a particle containing only microvoids, because of the greater ease of flow through the larger voids. In this way it is thought that 2.45 minus chert reaches a higher degree of saturation than heavier less porous fractions, and that this high degree of saturation is a prime factor in the lack of durability of the 2.45 minus material.

Besides being a factor in the permeability of the chert, the size of the pores undoubtedly determines whether dilation occurs. Pores or bulges in pores that are large enough to act as escape boundaries for the water under hydrostatic pressure (15) will cause no dilation. Obviously some of the large pores in the 2.45 minus material are in this category. The critical size between pores which will cause dilation and those which will act as escape boundaries depends on the length and tortuosity of the pores. It is possible that dilation in lightweight cherts occurs entirely within voids less than 5 microns in diameter which are supplied with water by the larger voids, but it is quite probable that some of the voids larger than 5 microns are too small to serve as escape boundaries and thus contribute to dilation of the particle.

In summary, the microvoids ratio does not provide a satisfactory indication of the freeze-thaw durability of chert. Instead chert durability is probably more closely related to the degree of saturation of these small voids (and larger voids that are too small to act as escape boundaries) which is strongly influenced by the presence of voids greater than 5 microns in diameter. Based on this concept and the results of the freeze-thaw tests, it appears that total porosity, as reflected in bulk specific gravity, serves as a satisfactory criterion for predicting the freeze-thaw durability of chert.

Absorption

Lewis and Dolch (17) have stated that, "The lack of durability of an aggregate in freezing and thawing is primarily dependent upon its ability to become and stay highly saturated under the given conditions of moisture." Thus besides being porous, an aggregate must be absorptive in order to be susceptible to freeze-thaw deterioration. This section discusses the relationship between (a) vacuum-saturated absorption and (b) rate of absorption of cherts and shales, and the resistance of these materials to freeze-thaw deterioration.

Vacuum-Saturated Absorption

Comparison of the results of the vacuum-saturated absorption tests of the chert groups (Table 8) to the results of freeze-thaw tests of concrete containing the cherts indicates a direct relationship between percentage of absorption and lack of freeze-thaw durability for chert. Cherts of the 2.45

Table 8

Vacuum-Saturated Absorption Values for Chert Samples

Source	Saturated Surface-Dry Bulk Specific Gravity Group	Size Range (inches)	Absorption (per cent)
2063	2.55 plus	3/4-1	1.26
		1/2-3/4	1.21
		3/8-1/2	1.12
			Avg. 1.20
	2.45-2.55	3/4-1	3.02
		1/2-3/4	2.84
		3/8-1/2	3.30
			Avg. 3.05
	2.45 minus	3/4-1	6.26
		1/2-3/4	6.13
		3/8-1/2	6.88
			Avg. 6.42
2064	2.55 plus	3/4-1	0.82
		1/2-3/4	0.93
		3/8-1/2	1.00
			Avg. 0.92
	2.45-2.55	3/4-1	2.86
		1/2-3/4	2.78
		3/8-1/2	2.90
			Avg. 2.85
	2.45 minus	3/4-1	5.71
		1/2-3/4	5.56
		3/8-1/2	5.62
			Avg. 5.63

Table 8 (continued)

Vacuum-Saturated Absorption Values for Chert Samples

Source	Saturated Surface-Dry Bulk Specific Gravity Group	Size Range (inches)	Absorption (per cent)
2066	2.55 plus	3/4-1	1.14
		1/2-3/4	1.30
		3/8-1/2	1.09
			Avg. 1.18
	2.45-2.55	3/4-1	2.76
		1/2-3/4	3.12
		3/8-1/2	2.86
			Avg. 2.91
	2.45 minus	3/4-1	6.12
		1/2-3/4	6.36
		3/8-1/2	6.11
			Avg. 6.20
2067	2.55 plus	3/4-1	1.09
		1/2-3/4	1.05
		3/8-1/2	1.19
			Avg. 1.11
	2.45-2.55	3/4-1	2.96
		1/2-3/4	2.84
		3/8-1/2	2.67
			Avg. 2.82
	2.45 minus	3/4-1	5.58
		1/2-3/4	5.59
		3/8-1/2	5.62
			Avg. 5.60

Table 8 (continued)

Vacuum-Saturated Absorption Values for Chert Samples

Source	Saturated Surface-Dry Bulk Specific Gravity	Size Range (inches)	Absorption (per cent)
2072	2.55 plus	3/4-1	1.02
		1/2-3/4	1.11
		3/8-1/2	1.11
			Avg.1.08
	2.45-2.55	3/4-1	2.55
		1/2-3/4	2.80
		3/8-1/2	2.96
			Avg.2.77
	2.45 minus	3/4-1	5.26
		1/2-3/4	5.39
		3/8-1/2	5.91
			Avg.5.52
2077	2.55 plus	3/4-1	1.06
		1/2-3/4	0.92
		3/8-1/2	1.04
			Avg.1.01
	2.45-2.55	3/4-1	2.31
		1/2-3/4	2.69
		3/8-1/2	2.27
			Avg.2.42
	2.45 minus	3/4-1	4.77
		1/2-3/4	4.20
		3/8-1/2	4.51
			Avg.4.49

minus specific gravity ranges for all six sources had absorption percentages about twice as great as those for the 2.45-2.55 groups and five times as great as the 2.55 plus groups. The absorptions of these materials are directly related to their porosities. As noted in Table 7, the higher percentage of pores larger than 5 microns in diameter found in the highly porous 2.45 minus material as compared to that in the 2.45-2.55 and 2.55 plus fractions apparently facilitated absorption in the lightweight chert with a resulting higher degree of saturation than could be obtained in the heavier materials.

As has been shown previously, chert with a specific gravity of less than 2.45 caused the only deterioration in the freeze-thaw test. From the data it appears that cherts with vacuum-saturated absorptions of less than about three per cent will not cause significant freeze-thaw deterioration when included in concrete in amounts up to ten per cent of the coarse aggregate for the size groups studied. Apparently chert with absorptions of about four per cent or greater will cause freeze-thaw failure when used in amounts as low as six per cent of the coarse aggregate.

The vacuum-saturated absorption values for the shales (Table 9) varied considerably, the percentages of absorption roughly paralleling the porosities of the shale samples. As was the case for porosity, the absorption of the shales had no apparent influence on the resistance of concrete containing these materials to deep-seated deterioration. This is shown by the fact that none of the shales, including the most absorptive samples, caused any deep-seated freeze-thaw failure when used in concrete in amounts up to ten per cent of the coarse aggregate.

Table 9

Vacuum-Saturated Absorption Values for Shale Samples

Source	Size Range (inches)	Absorption (per cent)
2063	3/4-1	1.54
	1/2-3/4	1.60
	3/8-1/2	2.17
		Avg. 1.77
2066	3/4-1	7.07
	1/2-3/4	7.05
	3/8-1/2	7.04
		Avg. 7.05
2068	3/4-1	12.83
	1/2-3/4	12.23
	3/8-1/2	12.63
		Avg. 12.56
2075	3/4-1	3.64
	1/2-3/4	4.06
	3/8-1/2	4.68
		Avg. 4.13
2076	3/4-1	7.82
	1/2-3/4	8.61
	3/8-1/2	7.91
		Avg. 8.11

There does appear to be a relationship between absorption and severity of surface deterioration, however. Shale 2068, which has the greatest absorption, caused by far the greatest amount of popout damage. As was the case for porosity, the influence of absorption on surface deterioration is not so distinct among the other four shales. Shales 2066 and 2076 had considerably greater absorptions than shales 2063 and 2075 (Table 9), but there was little difference in the amount of surface deterioration caused by these four shales. Although the popout damage caused by shales 2066 and 2076 was slightly greater than that caused by shales 2063 and 2075, it was not as great as might be expected in light of the severe damage to concrete containing shale 2068. It is probable that the great difference in surface deterioration between shale 2068 and shales 2066 and 2076 is not entirely due to the greater porosity and absorption of sample 2068, but was at least partially a result of the relative lack of induration of this shale as compared to the others.

Rate of Absorption

The rates of absorption of cherts 2067 and 2077 (Figure 3) apparently had little influence on freeze-thaw durability of these materials. Material in all three specific gravity groups of chert 2067 attained nearly maximum absorption for the test in only five minutes, while the absorption of chert 2077 for the same specific gravity groups for the first five minutes was only about 25 per cent of its total absorption. Although the total porosities and absorptions of these two cherts were similar, the rates of absorption indicate the two materials have considerably different pore systems. Chert 2067 obviously is more

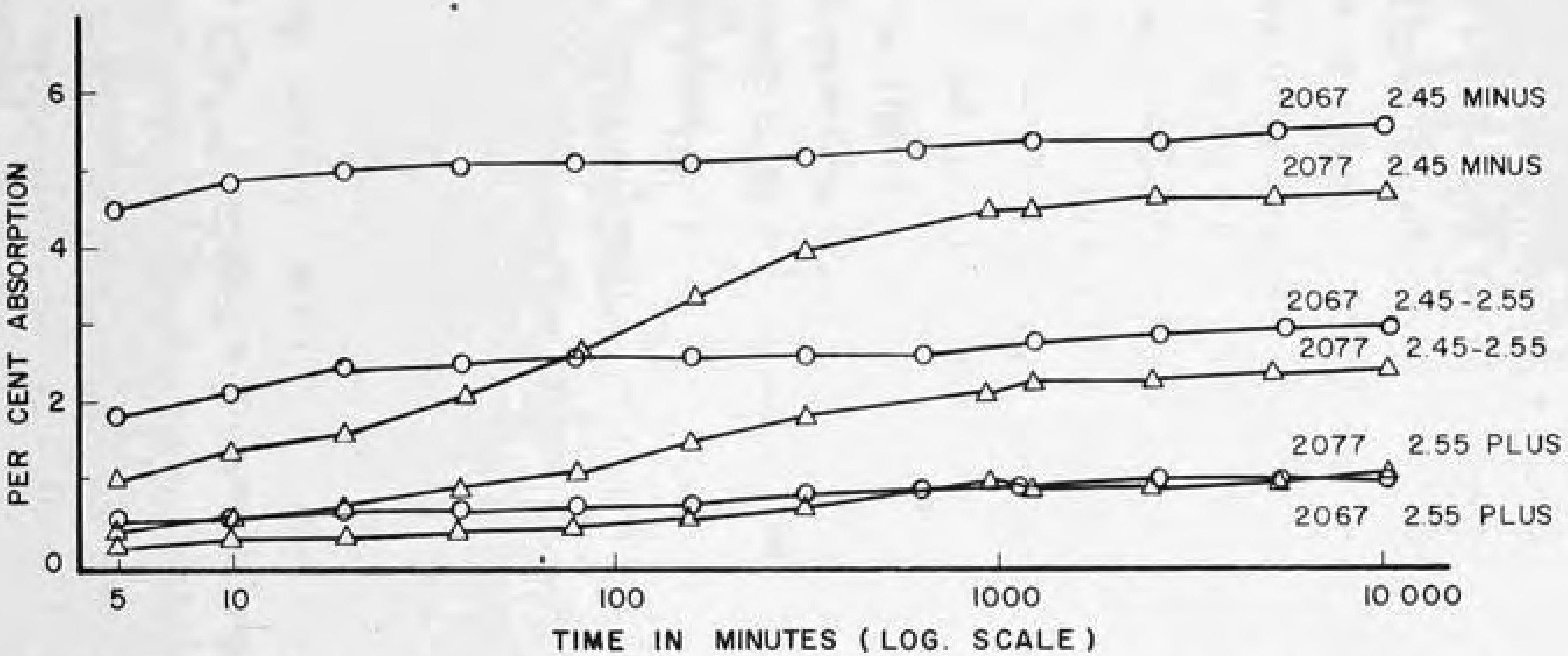

FIGURE 3 RATES OF ABSORPTION FOR DIFFERENT SPECIFIC GRAVITY FRACTIONS OF CHERTS 2067 AND 2077

permeable than chert 2077. This difference in permeability between cherts 2067 and 2077 is reflected in the degrees of saturation obtained by vacuum-saturating these two aggregates (Table 7). At all specific gravity levels chert 2067 had higher saturation coefficients than chert 2077. In spite of this difference in permeability and its resulting difference in degree of saturation, these two cherts resulted in similar deterioration in the freeze-thaw test at all specific gravity levels.

It should be noted that the freeze-thaw tests were conducted under rather severe saturation conditions. The aggregate was vacuum-saturated before mixing the concrete, and the beams were immersed in water for 13 days prior to being subjected to freezing and thawing. T hey were, of course, re-immersed during each thaw cycle. Under such conditions both cherts maintained a high degree of saturation (Table 7). Under actual service conditions, however, the amount of available water would not always be as great as in these laboratory tests and permeability could have a greater influence on freeze-thaw durability.

The rates of absorption of the shales (Figure 4) are directly related to the total absorptions of these materials. Those shales with high total absorptions absorbed water rapidly during the first few minutes of the test, following which water was absorbed at a slowly decreasing rate for the rest of the test. The shales with low total absorptions exhibited a fairly constant increase in absorption throughout the test. As was the case with porosity and total absorption, this greater permeability of certain shales had no influence on the resistance of the shales to deep-seated deterioration. It probably is a factor in surface deterioration, however, since shale 2068 which caused the most popout damage also had the fastest rate of absorption.

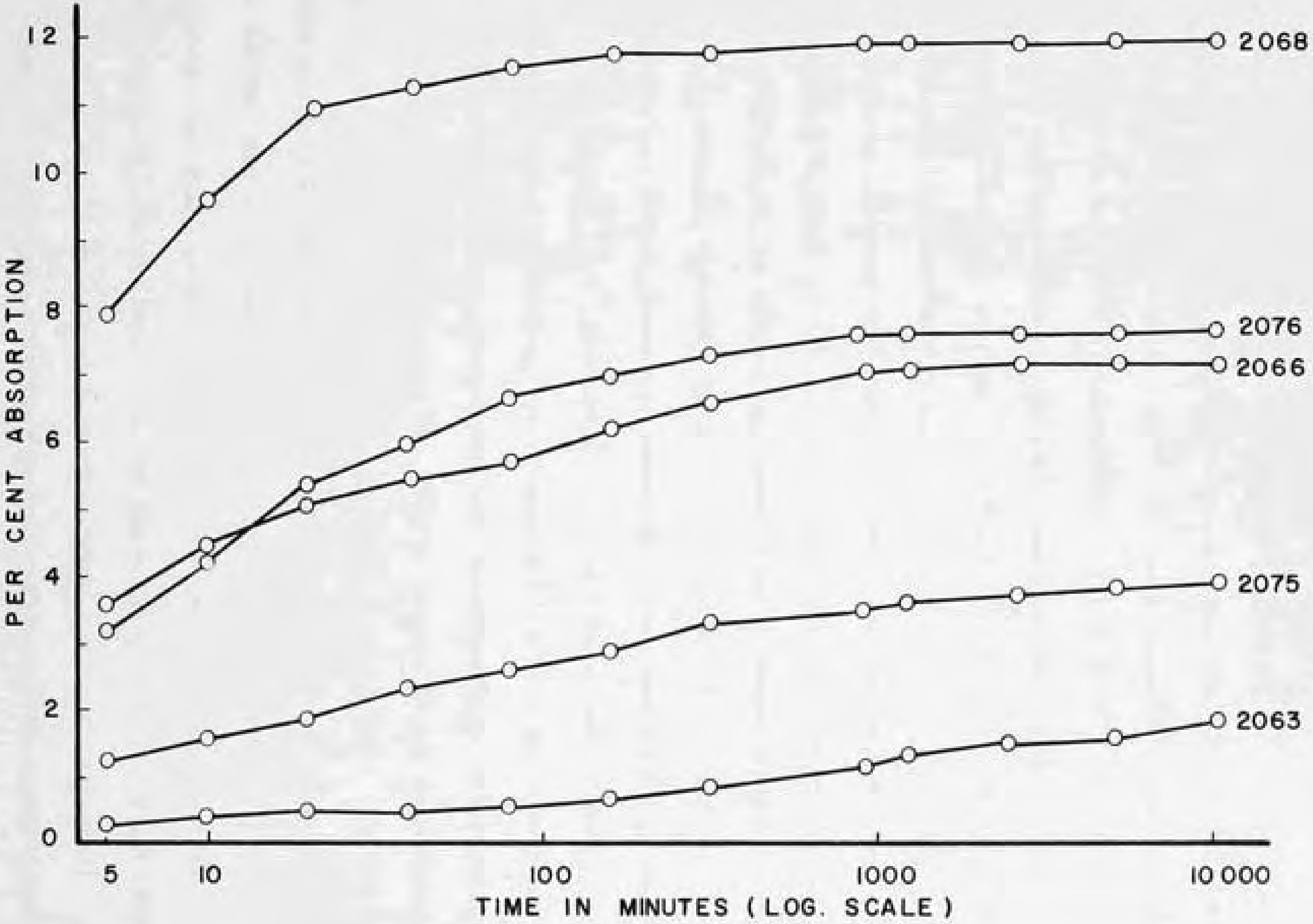

FIGURE 4 RATES OF ABSORPTION FOR SHALE SAMPLES

Mineralogy, Texture and Microstructure

The properties of mineralogy, texture, and microstructure have been grouped grouped together because they were all included in studies utilizing the petrographic microscope. Microscopic petrography has long been a valuable tool in study of the characteristics of rocks. Runner (19) was one of the first to apply petrography to the study of deleterious substances in aggregates. His petrographic investigations were followed by reports of similar studies and description of techniques by Mielenz (20, 21), Rhoades and Mielenz (22, 23), and Mather and Mather (24). In the present study an attempt was made to find a relationship between the results of petrographic studies of deleterious materials and the laboratory freeze-thaw durability of these materials.

Petrographic study was carried out using a Leitz Ortholux petrographic microscope with binocular attachments. Thin sections were made from each of the five shale samples and six chert samples. These sections were studied under transmitted light at magnifications of approximately 100X to 400X. Since the complete mineral composition of shales is not easily determined by microscopic analysis, x-ray diffraction and differential thermal analysis supplemented microscopic petrography in study of shale mineralogy.

Petrographic analysis of thin sections showed the cherts to be of generally similar mineralogical character. They are composed primarily of microcrystalline quartz and radial chalcedony. Small amounts of coarser-grained secondary quartz, some calcite, and limonite and carbonate rhombs are also present.

Each of the cherts consists primarily of microcrystalline aggregates of quartz grains usually less than 0.01 mm. in diameter. The secondary quartz occurs as granular masses which apparently replaced carbonate minerals. The individual quartz grains in these secondary masses range in size from less than 0.01 mm. to as large as 0.2 mm. Radiating chalcedony in the chert samples occurs as spherulites, often as much as 0.25 mm. wide.

The carbonate and limonite rhombs range in size from less than 0.01 mm. to as much as 0.1 mm. Although most of the rhombs consisted of carbonate _or_ limonite, some appeared to have a translucent carbonate mineral in the center surrounded by a rim of opaque limonite. The carbonate rhombs probably formed by replacement of crystalline quartz in the original chert (25), and in turn these rhombs are being replaced by limonite. Limonite also occurs as finely disseminated masses scattered throughout the chert.

One chief mineralogical difference was noted among the chert gravels. Cherts from the southern part of Indiana (especially material from the Ohio River) contain considerably more limonite than those from the northern part of the state. This difference in limonite content apparently had no influence on freeze-thaw durability, however, since all six cherts reacted similarly to the freeze-thaw tests.

It is also of interest to note that little difference in mineralogy was found among the different specific gravity groups for each chert source in spite of the fact that the materials in the different specific gravity groups had considerably different freeze-thaw durabilities. Apparently there is little direct relationship between mineralogy and freeze-thaw durability of Indiana cherts.

An important microstructural feature in chert samples from all six sources is the numerous voids observed in thin sections from material in the 2.45 minus specific gravity range, (see Figures 5 and 6). No voids were noted in the 2.45-

Figure 5

Chert 2077 (s.g. 2.45 minus) in plain light.
Limonite (opaque) and carbonate (translucent rhombs in fine-grained quartz matrix.
Note void in process of formation by solution of carbonate from large rhomb near intersection of cross-hairs.

Figure 6

Chert 2063 (s.g.2.45 minus) between crossed nicols.
Large voids surrounded by radial chalcedony and fine-grained quartz matrix.

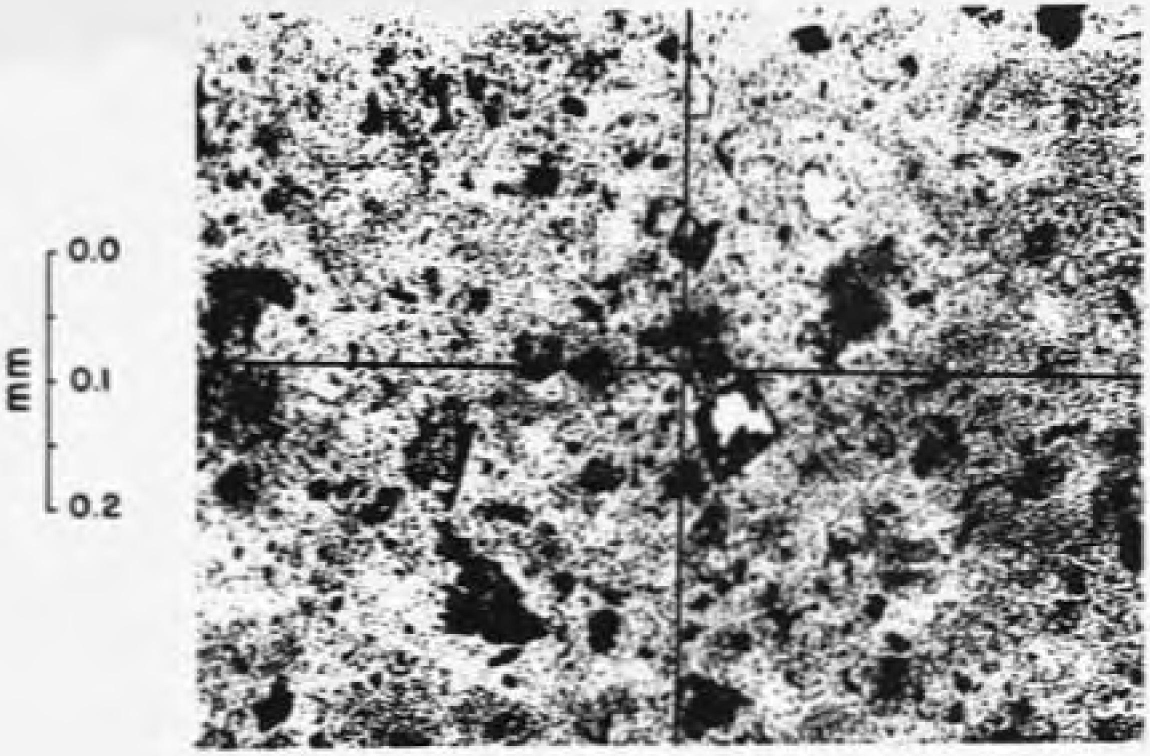

FIGURE 5

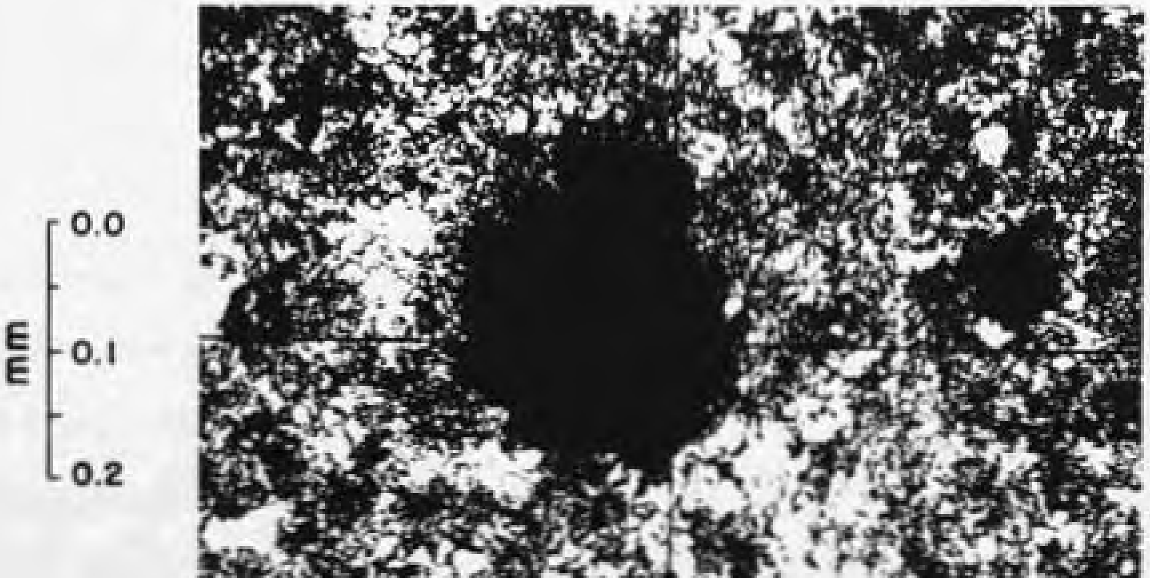

FIGURE 6

2.55 and the 2.55 plus ranges. The voids are all fairly large, since voids less than about 30 microns in diameter cannot be detected easily in thin-section study. They range in size up to 0.4-0.5 mm. in diameter, but most are less than 0.1 mm. in diameter. As noted in the previous section on porosity, the concentration of these voids in the chert with specific gravity less than 2.45 resulted in the relatively high porosity of this lightweight chert, and the high degree of saturation achieved in the lightweight chert fractions is probably also related to the presence of these voids. Voids of this size had previously been recognized in thin sections of lightweight cherts from other states by Wuerpel and Rexford (14) who noted that these voids were related to the lack of durability of the cherts.

The 2.45-2.55 and 2.55 plus specific gravity groups contained practically no voids large enough to be recognized in thin section. Since lack of freeze-thaw durability was found only in the 2.45 minus chert, there is a direct correlation between the presence of these voids and the lack of durability of the lightweight chert. Although this contradicts the theories of Blanks (18) and others that freeze-thaw deterioration occurs primarily in voids less than 5 microns in diameter, there is a strong possibility (as demonstrated previously in the section on porosity) that the larger voids are prime factors in the freeze-thaw breakdown of lightweight cherts due to the higher degree of saturation afforded the chert by the larger voids.

Other textural properties such as grain size, and presence of rhombic-shaped grains and replaced fossils, apparently had no influence on the freeze-thaw durability of the chert. These characteristics are similar in cherts of all three specific gravity ranges.

Petrographic, x-ray, and differential thermal analyses of the shales indicate a similarity in their general mineralogic composition, but there is considerable variation in certain characteristics. All the shales consist of detrital mineral grains, primarily quartz, in a very fine-grained matrix of clay minerals or hydromicas. The chief differences shown by the shales are the relative size and abundance of the detrital minerals and the relative amounts of clay minerals and organic material in the samples.

In order to satisfactorily describe the shales and to point out differences in their petrographic characteristics, and yet avoid repetition, a brief petrographic description of a shale with "average" characteristics will be given, and the mineralogies, textures, and microstructures of the strongest, least porous shale (2063) and the weakest, most porous shale (2068) will be compared with those of the average sample.

The average shale is composed primarily of a fine-grained illite matrix enclosing detrital quartz grains up to 0.04 mm. in diameter (0.01-0.02 mm. average). It contains considerable organic matter and disseminated limonite and a small amount of chlorite. Loss on ignition for this shale is approximately 12 per cent. This description fits shales 2066, 2075, and 2076 fairly well.

Shale 2063, the most indurated and least porous of the shales, contained more detrital quartz and less clay mineral than the average shale. Besides being more abundant, the detrital quartz grains are larger than in the other shales, ranging in size up to 0.07 mm. in diameter (0.02 - 0.03 mm. average). The abundance and size of the detrital quartz is sufficient to classify sample 2063 as a silty shale or possibly even a siltstone. This sample also contained more organic material than the other shales as was shown by the 16.7 per cent loss on ignition, the highest of all the shales.

Shale 2068, the softest and most porous of the shales, contains more clay mineral and less detrital quartz than the other shales. The size of the detrital quartz grains is about the same as that of the average shale, but the lower percentage of these grains means that the average grain size of shale 2068 is considerably smaller than that of the average shale. The relatively high percentage of illite accounts for the high porosity of this shale, and the combination of high clay and low quartz content accounts for its lack of induration.

Although the differences in relative percentages of clay minerals and quartz apparently have no effect on the tendency of the shales to resist deep-seated deterioration, these mineralogic differences, which influence the textures and microstructures, apparently do affect the amount of surface deterioration caused by the shales. This is especially true for shale 2068. Its high clay mineral content renders it weaker and more porous than the other shales, and thus it is more susceptible to freeze-thaw deterioration.

SUMMARY OF RESULTS

The following is a brief recapitulation of major findings of the study:

1. Freezing-and-thawing tests of concrete beams containing chert indicated the following:

   (a) There was no difference in degree of deep-seated deterioration caused by any of the different cherts. Thus the source of chert had no effect on freeze-thaw durability.

   (b) The only combination of variables resulting in severe deep-seated deterioration was ten per cent of 2.45 minus chert. This combination resulted in deep-seated failure of all beams containing chert from each of the six sources. In addition, six per cent of 2.45 minus chert caused moderate deep-seated damage in a few cases.

2. Durability factors for the shale beams indicated that no deep-seated deterioration occurred in beams containing two to ten per cent of any of the five shales studied. The data included no extremely low durability factors as were found for beams containing ten per cent of 2.45 minus specific gravity chert. Only a few had durability factors below 90, and these few values were seemingly randomly distributed throughout the data.

3. Study of surface deterioration of concrete beams containing chert showed that freezing and thawing caused significant popout damage in beams containing 2.45 minus specific gravity chert. Very few popouts were caused by chert having specific gravities of 2.45-2.55 and 2.55 plus.

4. The greatest amount of surface deterioration of the beams containing shale was caused by shale 2068, the most porous and most absorbent of the shales. Shale 2068 caused considerable popout and pitting damage at all four percentage levels, but, as would be expected, the amount of deterioration increased with increasing percentage of shale. The other four shales tested caused no surface deterioration when included in concrete in amounts up to and including four per cent. At the six and ten per cent levels, shales 2066 and 2076, which were more porous and absorbent than shales 2063 and 2075, caused slightly more surface deterioration than the latter.

5. The study of air voids in concrete by means of the linear traverse technique demonstrated that machine-mixed concrete beams with high durability factors had air-void spacing factors lower than 0.01 inches, and hand-mixed beams with low durability factors had spacing factors higher than 0.01 inches. These results support Powers' theory that concretes with spacing factors lower than 0.01 inches are well protected from freezing and thawing deterioration, while those with spacing factors greater than 0.01 inches are poorly protected.

6. Study of the size distributions of pores for cherts 2067 and 2077 indicated a marked increase in percentage of total voids volume consisting of pores larger than 5 microns in diameter with decreasing bulk specific gravity of the chert. For example, in the case of chert 2067, the voids larger than 5 microns in diameter constituted only 20 per cent of the total pore space in the 2.55 plus chert, 25 per cent in the 2.45-2.55 material, and 50 per cent in the 2.45 minus range. Conversely, the voids less than 5 microns in diameter constituted a decreasing percentage of total voids volume with increase in total porosity and resulting decrease in bulk specific gravity.

7. Although the total porosities and absorptions of cherts 2067 and 2077 were similar, their rates of absorption indicate that these two cherts have considerably different pore systems. Chert 2067 was more permeable than chert 2077. Material in all three specific gravity groups of chert 2067 attained nearly maximum absorption after only five minutes of immersion, while the absorption of the same specific gravity groups of chert 2077 for the first five minutes was only about 25 per cent of its total absorption.

The rates of absorption of the shales were directly related to the total absorptions. Those shales with high total absorptions absorbed water rapidly during the first few minutes of immersion, following which water was absorbed at a slowly decreasing rate for the rest of the test. The shales with low total absorptions exhibited a fairly constant increase in absorption throughout the test.

8. Petrographic analysis of thin sections showed the cherts to be of generally similar mineralogical character. They were composed primarily of microcrystalline quartz and radial chalcedony. Small amounts of coarse-grained secondary quartz, some calcite, and limonite were also present. One chief mineralogical difference was noted. Cherts from the southern part of Indiana (especially from the Ohio River) contained more limonite than those from the northern part of the state. This limonite occurred both as rhombs and in amorphous form. No differences in mineralogy were noted among the three specific gravity groups for the chert samples.

The shales also presented a similarity in their general mineralogic compositions, but showed considerable variability in certain characteristics. All the shales consisted of detrital mineral grains, primarily quartz, in a very fine-grained matrix of clay minerals or hydromicas. The chief differences shown by the shales were the relative size and abundance of the detrital mineral grains and the relative amounts of clay minerals and organic material in the samples.

9. The textures and microstructures of the cherts were all similar. Each chert consisted primarily of microcrystalline aggregates of quartz grains usually less than 0.01 mm. in diameter with granular masses of secondary quartz, radiating masses of chalcedony, and carbonate and limonite rhombs. The only notable structural difference in the cherts was that the 2.45 minus fraction of each sample contained numerous voids large enough to be identified in thin sections between crossed nicols. These voids, which averaged less than 0.1 mm. in size, but ranged in size up to 0.4-0.5 mm., did not occur in the 2.55 plus and 2.45-2.55 specific gravity groups.

The textures and microstructures of the shales varied considerably. Although all the shales consisted of a fine-grained matrix enclosing detrital quartz grains, the relative amounts of these materials and the sizes of the detrital particles varied enough to influence strongly the strength and hardness of the different shales. All the shales showed preferred orientation of grains.

10. Heavy-liquid separation of the chert samples showed that all six cherts had similar specific gravity distributions. About 50 per cent of each sample fell within the 2.45-2.55 bulk specific gravity range (saturated surface-dry basis). The 2.55 plus material constituted from 15-30 per cent of each sample, and about 20-30 per cent of each sample had a specific gravity lower than 2.45.

Although the bulk of material in each of the shale samples fell in the 2.05-2.45 specific gravity range, the shale samples showed greater variability in their specific gravity distributions than did the cherts. The shales varied in specific gravity from samples 2075 and 2063 with over 70 per cent heavier than 2.25 to shales 2068 and 2066 with less than ten per cent heavier than 2.25.

CONCLUSIONS

Based on the results of this study, the following conclusions seem justified. Since this study was restricted to certain Indiana cherts and shales subjected to specific methods of test, the conclusions can logically be applied only to similar cherts and shales under similar conditions. However, in some cases, field behavior of the cherts and shales may be inferred from these conclusions.

1. For a wide variety of cherts, the source of the chert has no effect on its freeze-thaw durability in concrete.

2. Chert exhibits a definite relationship between its bulk specific gravity and durability in concrete exposed to freezing and thawing. Apparently only chert with a bulk specific gravity of less than 2.45 (saturated surface-dry basis) will cause either deep-seated or surface deterioration of air-entrained concrete in which it is used.

3. The freeze-thaw durability of concrete containing chert apparently is not as dependent on pores in the chert less than 5 microns in diameter as has been postulated by Sweet (1). Instead chert durability is apparently based on a more complicated interrelationship between total porosity, size of pores, absorption, and degree of saturation. Pores larger than the 5-micron size specified by Sweet permit easier passage of water into immersed aggregates, result in relatively high degrees of saturation, and contribute to freeze-thaw deterioration of lightweight chert. Microscopic studies of polished sections show that these larger pores make up about half the void volume in 2.45 minus specific gravity chert.

4. The petrographic characteristics of the cherts influence the freeze-thaw durability of these materials only in the relationship of these characteristics to porosity of the cherts. For example, although mineralogy of the cherts has no direct effect on their freeze-thaw durability, the presence of carbonate rhombs, which have weathered out to form voids, has lessened the durability of some chert particles.

5. Many shales will not cause deep-seated deterioration of air-entrained concrete beams subjected to laboratory freezing and thawing when included in these beams in amounts up to ten per cent. The inherent structural weakness of these materials may account for this.

6. Different shales cause considerably different degrees of surface deterioration of air-entrained concrete exposed to freezing and thawing. Some shales cause considerable popout damage when included in concrete in amounts as low as two per cent of the coarse aggregate. Other shales cause little damage when used in amounts up to ten per cent.

7. The durability of the shales studied apparently is related primarily to the porosities and absorptions of these materials; the most porous and most absorbent casuing the greatest amount of surface deterioration of concrete in which these materials are used. However, the strength and induration of the shales, as determined by relative amounts of clay minerals and detrital quartz present, also influence the ability of these materials to cause surface deterioration, the softer, weaker materials being less resistant than the harder, stronger ones.

8. As theorized by Powers (10), concretes with air-void spacing factors lower than 0.01 inches are well-protected from freezing-and-thawing deterioration, while those with spacing factors greater than 0.01 inches are poorly protected.

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