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EVALUATION OF AN OTTAWA LIMESTONE AS
CONCRETE AGGREGATE

PART I

by

J.A. Soles, F.E. Hanes and N.G. Zoldners

Mineral Processing Division

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- - -

SUMMARY OF RESULTS

The results obtained from standard physical acceptance tests for abrasion loss (20.5%) and for soundness loss (MgSO_4 - 7.5% in 5 cycles), indicate that the limestone was of acceptable quality for concrete aggregate.

Chemical analyses of samples showed a wide variation in content of silica and alumina at different levels in the quarry. The analyses support evidence from a lithological study that shaly material increases with depth and limits downward development of the quarry.

Durability studies on test specimens of air-entrained concrete incorporating the limestone produced for aggregate showed that after 1000 cycles of freezing and thawing the loss of weight was 0.3% and the expansion 0.025%; these values are well under acceptable limits for durable concrete. The ultrasonic pulse velocity after 1000 freeze-thaw cycles showed an increase of 1.5%, which indicates that the structural properties of the concrete test specimens were not affected during exposure.

Finally, flexural strength of the freeze-thaw test specimens was about the same as that of companion test beams.

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INTRODUCTION

This investigation was undertaken at the request of Francon (1966) Limited, of Montreal and Ottawa, to determine the suitability of crushed limestone produced by its subsidiary, the Ottawa Valley Crushed Stone Ltd., for use as aggregate in concrete mixes.

The company obtained additional data on this aggregate by having chemical analyses made by the Canada Cement Company, Limited, and physical tests made by two independent commercial laboratories in Ottawa and Montreal.

The quarry under investigation has two areas of operation - an old section opened in 1949 and a new section opened in 1967. On March 23, 1966, the authors visited the quarry to examine the lithology and the rock produced in order to gain a knowledge of the quality of the primary materials and the aggregates derived from them. Information collected in this report refers to the coarse limestone aggregate produced in 1966 from the old section; studies on the fine aggregate, manufactured by reducing coarse aggregates, were made earlier by Hanes⁽¹⁾.

LITHOLOGY

The quarry is located in an area underlain by rocks of Ordovician age which has been correlated with rocks of the Black River - Trenton group in Montreal and central to western Ontario. The quarry rock itself is typical of the Ottawa formation, described in detail by Wilson⁽²⁾.

In the higher parts of the quarry, the rock consists of undeformed, thick-bedded (1-6 ft), relatively pure, fine-grained limestone intercalated with thinner layers (0.5 - 2 ft) of darker, impure clayey limestone and partings of shale. Dolomite-rich bands and gritty, siliceous layers are occasionally present in the carbonate strata. Recrystallization of calcite to a coarser grain size is localized and usually associated with fossils, which are scattered and ubiquitous but more abundant in the shaly strata. About 30 feet below the quarry lip, the lithology changes from predominantly massive limestone to thinner beds, accompanied by a gradual darkening of the limestone with depth and an increase in the number of shaly partings. Below about 50 feet, the shale content of the rock may exceed 10%; the limestone is dark with abundant clay and fissile shaly bands up to 4 inches thick. The rock composition

also changes laterally in a similar way because the conditions of sedimentation varied locally. A few narrow, vertical calcite-filled fractures and rare, small igneous dykes cut the section.

The increase in shale content of the rock has curtailed lateral and downward development of the quarry, as the proportion of reject materials increased and the aggregate quality decreased. Only certain areas provide rock which is sufficiently sound for use as concrete aggregate; therefore, selective quarrying would be necessary to produce aggregate of acceptable quality.

MILL PRODUCTS

An examination of the different fractions from the crushing plant indicated that only the minus $\frac{1}{4}$ -inch fraction was of dubious quality, and this had been rejected as unsuitable for aggregate. Less than 2% of shaly material was detected visually in the coarser fractions. The plant manager informed the writers that only 8 - 12% of the mill feed, depending on the shale content of the rock, was being rejected into the fine fraction as material unsuitable for concrete aggregate. This fraction obviously contained much of the friable material from the shaly partings and fissile rock.

Samples of different coarse-aggregate fractions were taken to determine physical properties of the rock and to make concrete test specimens for durability studies.

TESTS ON AGGREGATES

A. Physical Tests

Physical acceptance tests made on crushed limestone coarse aggregate produced by the Ottawa Valley Crushed Stone Ltd. conform to the following ASTM standard methods of test:

- (a) Specific Gravity and Adsorption of Coarse Aggregate, ASTM Designation C 127-59;
- (b) Resistance to Abrasion of Small Size Coarse Aggregate by Use of the Los Angeles Machine, ASTM Designation C 131-66;

- (c) Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, ASTM Designation C 88-63.

Test Results

The following results were obtained from the physical tests:

(a) Bulk Specific Gravity

Saturated, surface-dry (SSD), av. of two samples - 2.69

Absorption

Average of two samples - 0.40%

(3)

(b) Wear Resistance to Abrasion

Los Angeles machine for a 'B' grading - 20.5%

(A similar test by Paterson Laboratories gave 21.0%)

Approximately 12 lb of material in two sizes, $\frac{3}{4}$ to $\frac{1}{2}$ -inch and $\frac{1}{2}$ to $\frac{3}{8}$ -inch, were used for the abrasion test (3).

(c) Sulphate Soundness Loss (3)

Five cycles in a magnesium sulphate solution, corrected for an assumed blend of coarse aggregate in concrete - 7.5%

Data for individual fractions tested are shown in Table 1 (After Hanes, p.3 (1)).

From a representative sample, four identical portions were prepared by the Mines Branch. Three of these were returned to Canada Cement for Sulphate Soundness Tests by other laboratories. Table 2 shows the results of these tests along with the results of the test made by the Mines Branch (1).

TABLE 1

Percentage Losses in Sulphate Soundness ($MgSO_4$) Test
(5 cycles)

Size Fractions	Grading* per cent	Weights of Test Fractions, g (before test)	Actual Loss, per cent (after test)	Corrected Loss, per cent
-1" + 3/4"	10	1000	7.5	0.75
-3/4" + 1/2"	30	750	9.9	2.97
-1/2" + 3/8"	30	500	7.2	2.16
-3/8" + No. 4	30	3300	5.4	1.62
Total				7.50

* Assumed aggregate grading in concrete mixture

NOTE: After completion of the sulphate soundness test, further shaking on the sieve caused some break-down of the more shaly fragments. Inspection of single particles however indicated that 90 - 95 per cent of the particles remained firm after the sulphate soundness test.

TABLE 2

Sulphate Soundness Percentage Loss Comparison Table

Size Fractions	Loss, Per Cent			
	Dept. Energy, Mines and Resources	Warnock-Hersey	Canada Cement Co.	Paterson & Assoc.
-1" + 3/4" Actual -	7.5	16.3	6.5	7.4
Corrected-	0.75	1.63	0.65	0.74
-3/4" + 1/2" Actual -	9.9	9.3	7.9	5.2
Corrected-	2.97	2.79	2.37	1.56
-1/2" + 3/8" Actual -	7.2	14.3	13.8	11.9
Corrected-	2.16	4.29	4.14	3.57
-3/8" + No. 4 Actual -	5.4	4.8	5.8	7.1
Corrected-	1.62	1.44	1.74	2.13
Corrected Total:	7.50	10.15	8.90	8.00

Average loss of the four tests is 8.64 per cent.

B. Chemical Analyses

The sample of crushed limestone used for physical acceptance tests was also analyzed chemically by the Canada Cement Company. The results of this analysis are shown in Table 3.

Also shown is the average of eight analyses made on samples of two diamond drill (DD) cores, No. 1 and 2, drilled in 1955 at the location of quarry operations in 1966.

C. Discussion

Loss in Abrasion

The Los Angeles abrasion test (4) is essentially an impact test which provides a measure of the hardness and strength of an aggregate.

Data compiled by the U.S. Bureau of Public Roads⁽⁵⁾ show that, of 350 limestone samples tested by the Los Angeles abrasion method, 40 per cent had 22 per cent or less wear. Specifications limits for abrasion loss for coarse aggregate in concrete pavements, set up by the provincial departments of highways, are 28 and 30 per cent in Quebec and Ontario, respectively. ASTM standard specifications for concrete aggregate (C 33-67) specify an abrasion loss of not more than 50 per cent.

Results obtained by Los Angeles abrasion test on the Ottawa limestone aggregate did not exceed 21 per cent; this indicates that it has better than average hardness and strength.

TABLE 3

Chemical Analyses of Limestone

Oxide Composition (Ex Alkalies)		Aggregate Sample	DD cores C 55.- 1 & 2 (av. of 8 anal.)
Silica	(SiO ₂)	5.34	6.89
Alumina	(Al ₂ O ₃)	1.58	2.93
Iron Oxide	(Fe ₂ O ₃)	0.88	
Calcium Oxide	(CaO)	50.03	47.89
Magnesium Oxide	(MgO)	1.10	1.71
Loss on Ignit.	(L.O.I.)	39.84	38.88
Total		98.77	98.30

Sulphate Soundness

The sulphate soundness test is one of the most widely used methods of tests for acceptance or rejection of aggregate; but it is also one of the most controversial. It furnishes information helpful in judging the soundness of aggregate subjected to weathering action. The expansion of either sodium or magnesium sulphate salt crystals in the pores of the aggregates is similar to the expansion due to freezing of water. However, research has shown that this test may be related to the freezing and thawing resistance of concrete in only a very general way, hence it provides only a rough indication of potential durability^(6, 7). A high loss usually means that the aggregate is susceptible to damage by freezing and thawing. A very low loss usually, but not always, means that the aggregate is reasonably durable. An attempt to correlate statistically the resistance of coarse aggregate to sodium sulphate with its resistance to freezing and thawing in air-entrained concrete met with some success, but the wide scatter obtained indicated that concrete durability is influenced by factors not measured by the sulphate soundness test⁽⁸⁾.

In the present study, the results obtained in 5 cycles of magnesium sulphate tests (Table 2) gave an average loss of 8.64 per cent, which is well below the specification limits shown in Table 4.

TABLE 4

Specification Limits for Sulphate Soundness Losses

Specifications	Per Cent Loss in 5 cycles of	
	MgSO ₄	Na ₂ SO ₄
1 ASTM Standard Specifications for Concrete Aggregates C 33-57	18	12
2 CSA Standards for Concrete A 23-1960, Sec. 4.5.4.1	15	10
3 Dept. of Highways of Ontario, Spec. for Concrete Pavements (Sec. 502, Coarse Aggregate)	12	-

The results given in Table 2 are in good agreement with soundness loss values obtained in 1964 by Canada Cement Company on rock samples taken from the operating quarry face in the same area where the aggregates under investigation originated. These test results are shown in Table 5.

Chemical Analyses

Because silica and particularly alumina in limestone normally increase in proportion to the amount of shale, the relatively low silica and alumina content of the aggregate, shown in Table 3, suggests that the limestone has a low shale content. However, the analyses of the diamond drill cores give a more accurate picture of the composition of the primary material and support observations from the lithological study that the content of shale may be higher than is indicated by analysis of the aggregate.

The crushing operations have apparently improved the quality of the coarser aggregate sizes by reducing the more friable, shaly rock to rejectable minus $\frac{1}{4}$ -inch material. This study would therefore suggest that it may be hazardous to evaluate a shaly limestone as a potential aggregate source from a chemical analysis of aggregate alone, because it would not

TABLE 5

Soundness Losses on Rock from Quarry Face
(Canada Cement Company Tests)

Sample No.	Location, Map Coordinates	Date of Sample	Soundness Loss, %
77	E 17-19 S 14-15	19/ 5/64	8.1
78	E 19-22 S 14	16/ 7/64	5.5
79	E 19-21 S 15	6/ 8/64	7.7
80	E 17-19 S 15	9/ 9/64	11.4
81	E 19-22 S 15	10/10/64	10.6
			av. 8.7

take into account the partial loss of shaly material during processing. On the other hand, analysis of distinctive lithologic units for alumina and silica would provide invaluable information for predicting the suitability of such rocks as an aggregate material and for establishing approximate boundaries of a quarry.

Although an acceptable soundness loss and favourable chemical composition may indicate a low shale content in the aggregate, the final and most reliable measure of the soundness of an aggregate is obtained from freezing and thawing tests of concrete incorporating the aggregate.

CONCRETE DURABILITY STUDIES

These tests were made to study the effect that the crushed limestone under investigation would have when used as both coarse and fine aggregate. For these studies the ASTM standard method C 291-67 (rapid freezing in air and thawing in water) was used; this required the exposure of concrete test beams to cyclic freezing and thawing. Though this method does not give a quantitative measure of durability, it produces measurable changes in test specimens which may indicate deterioration of concrete.

A. Concrete Test Mix

Aggregates

Both coarse and fine aggregates were prepared from the crushed limestone received from Ottawa Valley Crushed Stone Limited.

Coarse aggregate was screened into separate fractions and then re-blended to give equal amounts of nominal 3/4-in., 1/2-in., and 3/8-in. sizes.

The fine aggregate used was produced in a Hazemag impactor from the 3/4-in. aggregate. With the gap set at 1/2 inch and impeller speed at 1500 rpm, a satisfactory graded crushed sand was obtained at a feed rate of 960 lb/hr(1).

Grading of both coarse and fine aggregates used in the concrete mix are shown in Table 6.

TABLE 6

Grading of Aggregates

Coarse Aggregate		Fine Aggregate	
Sieve Size	Accum. % Retained	Sieve No.	Accum. % Retained
3/4-in.	0.0	4	0.0
1/2-in.	33.0	8	10.0
3/8-in.	66.0	16	30.0
No. 4	100.0	30	60.0
		50	83.0
		100	94.0
		Fineness Modulus	= 2.77

Cement

Normal portland cement manufactured by the Canadian Cement Company, Limited, Plant No. 3, Hull, Quebec, was used in all test mixes. The physical properties and chemical analysis of this cement are given in Table 7.

TABLE 7

Physical Properties and Chemical Analysis of Cement*

Physical Tests	Chemical Analysis	%
Time of Setting, hr:min :	Silica (SiO ₂)	20.5
Initial, 3:00; Final, 5:15	Alumina (Al ₂ O ₃)	5.8
Fineness:	Iron Oxide (Fe ₂ O ₃)	2.8
Passing No. 200 sieve	Calcium Oxide, total (CaO)	63.4
- 95.3%	Calcium Oxide, free (CaO)	0.48
Blaine SSA - 2950 cm ² /g		
Compressive Strength, psi:	Magnesium Oxide (MgO)	2.9
3-day: 2670	Sulphur Oxide (SO ₃)	2.6
7-day: 3420	Loss on Ignition (LOI)	0.77
28-day: 4550	Insoluble Residue (IR)	0.41
		99.66

* Data supplied by the manufacturer.

Mix Proportions

A 2-cu-ft batch of air-entrained concrete was prepared using the following mix proportions.

Concrete Mix Ingredients	Batch Weights, lb	Proportion for 1 cu yd, lb
Cement	36.0	495
Fine Aggreg (SSD)	96.0	1325
Coarse " "	132.0	1820
Water (free)	22.5	310
Total	286.5	3940

This mix had a slump of 2½ inches and an air content of 4½ per cent.

B. Preparation and Curing of Test Specimens

From the concrete mix, a set of 6 test beams ($3\frac{1}{2} \times 4 \times 16$ -in.) and 6 test cylinders (6×12 -in.) were moulded. Stainless steel reference plugs for length change measurements were fixed in each end of the beams. After 28 days of initial moist-curing, all beam specimens were placed in the freeze-thaw cabinet at the thawing phase of the cycle for one hour in order to reduce their temperature to $40 \pm 3^{\circ}\text{F}$. At this temperature the initial and following measurements of length, weight, and ultrasonic pulse velocity were taken.

After the initial measurements were made, beams No. 1, 2, and 3 were returned to the moist-curing room for use as reference specimens cured under standard conditions. The remaining three beams, No. 4, 5, and 6, were then placed in the freeze-thaw cabinet and subjected to repeated cycles of freezing in air and thawing in water. The duration of one complete cycle, from $40 \pm 3^{\circ}\text{F}$ to $0 \pm 3^{\circ}\text{F}$ and back to $40 \pm 3^{\circ}\text{F}$, is about 3 hours. The freeze-thaw unit, therefore, automatically performs approximately 8 cycles each 24 hours.

C. Testing of Freeze-Thaw Specimens

The following measurements and tests were made to evaluate the resistance of concrete test specimens to accelerated freezing and thawing:

1. Determinations of weight changes.
2. Measurements of length changes.
3. Measurements of ultrasonic pulse velocities.
4. Determination of flexural strength.
5. Determination of compressive strength.
6. Visual examination of test specimens.

Freeze-thaw beam specimens were weighed and measured for length at approximately 100-cycle intervals. At the same time, beams were checked for evidence of deterioration by measuring ultrasonic pulse velocities and visually inspecting all beam surfaces.

After freeze-thaw cycling, which was continued through 1033 cycles*, both freeze-thaw and standard moist-cured test specimens were subjected to flexural-strength tests.

Test Results

The initial and final values of weight, length, and pulse velocity for standard-cured and freeze-thaw test specimens, as well as the per cent changes in these properties, are shown in Tables 8 and 9.

Also shown are the final flexural strengths of standard-cured and freeze-thaw test specimens after completion of 1033 cycles.

Visual examination revealed no significant external deterioration of the freeze-thaw test specimens after exposure to 1033 cycles. Only slight surface scaling in isolated small areas and crumbling of a few corners had occurred suggesting that freeze-thaw cycling had not damaged the test specimens.

Discussion of Results

The weight loss of freeze-thaw test specimens is negligible (av. 0.3%); this conforms with the conclusion from visual examination.

The length measurements indicate the test beams expanded slightly, averaging about 0.025 per cent. This is well below the generally accepted maximum expansion limit of 0.10 per cent for durable concrete⁽⁹⁾.

The standard-cured reference beams showed slight weight increases (av. 0.2%) and some shrinkage (av. 0.002%).

Table 9 provides data on changes in pulse velocity before and after freeze-thaw cycling. The ultrasonic pulse velocity increased during exposure to freezing and thawing for an average gain of 0.7% and 1.5% after 1033 cycles. At the same time for the companion standard-cured specimens, the pulse velocity after 154 days showed an average increase of 3.5%, which is more than double that recorded for the freeze-thaw test specimens.

* 1033 cycles took 154 days to complete; some time was lost due to equipment being non-operational.

TABLE 8

Weight and Length Changes of Beam Specimens

Tests		No.	Initial (28-d)	Final (154-d)	Change	Per Cent Change
Weight Change	Reference (Standard cured)	1	1b 18.550	1b 18.592	1b +0.42	% +0.2
		2	18.807	18.863	+0.56	+0.3
		3	18.456	18.491	+0.35	+0.2
	Freeze-Thaw Specimens		0 cycles	1033 cycles		avg -0.2
		4	18.925	18.882	-.043	-0.2
		5	18.394	18.311	-.083	-0.5
		6	19.306	19.272	-.034	-0.2
						avg 0.3
	Reference (Standard cured)	1	Gauge dial readings			
		2	.0759	.0755	-.0004	.003
		3	.0286	.0284	-.0002	.0015
Length Change*	Freeze-Thaw Specimens		0 cycles	1033 cycles		.002
		4	.0454	.0451	-.0003	avg .002
	Freeze-Thaw Specimens	4	.0465	.0507	+0.0042	0.03
		5	.0841	-	-	-
		6	.0608	.0633	+0.0025	0.02
						avg 0.025

* Gauge length 13.6 in.

These results indicate that improvement produced under standard curing conditions, as evidenced by the reference beams, was not achieved on the freeze-thaw specimens. Normally, exposure to freezing and thawing tends to retard the normal curing of concrete. However, the cycling in this test caused no serious deterioration of the beams. That the concrete did not deteriorate after 1033 cycles is also indicated by the flexural strengths of the test beams. Table 9 shows that both standard moist-cured and freeze-thaw test specimens broke at about the same flexural strength, i.e., 830 and 840 psi, respectively.

TABLE 9

Pulse Velocity Changes and Flexural Strength of Beams

Test Series	No.	Ultrasonic Pulse Velocity, fps					Beam Strength	
		at 28 days	at 70 days	% Change	at 154 days	% Change	Load, lb	Flexural psi
Standard-Cured Reference Specimens	1	14,125	-	-	14,600	+3.4	4120	880
	2	14,320	-	-	14,790	+3.3	3540	780
	3	14,210	-	-	14,700	+3.7	3900	835
Average		14,210	-	-	14,700	+3.5	3850	830
Series	No.	at 0 cycles	at 330 cycles	% Change	at 1033 cycles	% Change		
Freeze-Thaw Test Specimens	4	14,270	14,300	0.2	14,400	+0.9	3900	835
	5	14,140	14,300	1.1	14,420	+2.0	3860	825
	6	14,180	14,300	0.9	14,400	+1.6	3980	850
Average		14,200	14,300	0.7	14,410	+1.5	3910	840

CONCLUSION

The results of physical acceptance tests and concrete freezing and thawing tests show that crushed limestone taken from the quarry production line is satisfactory for use as aggregate in exposed concrete.

However, in order to assure production of a good quality concrete aggregate, selective quarrying should be carried out to eliminate unsatisfactory, shaly material.

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