DECLASSIFIED
DATE 23/11/78
AUTHORIZED BY RL Currington

CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 62-57

EVALUATION OF BRUCITIC LIMESTONE FROM WAKEFIELD, QUE., FOR USE AS CONCRETE AGGREGATE

by

N. G. ZOLDNERS

MINERAL PROCESSING DIVISION

NOTE: THIS REPORT RELATES ESSENTIALLY TO THE SAMPLES AS RECEIVED. THE REPORT AND ANY CORRESPONDENCE CONNECTED THEREWITH SHALL NOT BE USED IN FULL OR IN PART AS PUBLICITY OR ADVERTISING MATTER.

COPY NO. 11

AUGUST 3, 1962

Industrial Confidential

Mines Branch Investigation Report IR 62-57

EVALUATION OF BRUCITIC LIMESTONE FROM WAKEFIELD, QUE., FOR USE AS CONCRETE AGGREGATE

by

N.G. Zoldners*

SUMMARY OF RESULTS

To evaluate brucitic limestone samples for use as concrete aggregate, two identical test mixes were prepared and two series of test cylinders and beams were moulded.

Mix 225 made with crushed brucitic limestone as coarse and fine aggregate produced abnormal temperature rise and premature stiffening of the concrete. Test specimens of this series showed high expansion and poor durability.

Mix 226 made with the same coarse aggregate but using natural sand as fine aggregate produced good concrete. Compressive strength of this concrete was 25% higher than that of mix 225. The relative dynamic modulus of elasticity of test beams, based on the ultrasonic pulse velocity measurements, after 994 cycles of freezing and thawing was 80% of the initial modulus, an indication of durable concrete.

---

*Head, Construction Materials Section, Mineral Processing Division, Department of Mines and Technical Surveys, Ottawa, Canada.

CONTENTS

TABLES

FIGURES

INTRODUCTION

This investigation was undertaken at the request of the Aluminum Company of Canada, Limited, to determine the suitability of brucitic limestone from Wakefield, Que., for use as concrete aggregate.

Brucitic limestone is being quarried by the company and processed into magnesia and hydrated lime at its Wakefield plant, which is 22 miles north of Ottawa on the Gatineau river. Selective quarrying and processing of the ore resulted in large stockpiles of rejected raw material.

A 600-lb sample of the rejected crushed brucitic limestone, which has been separated into coarse and fine aggregate size fractions, was submitted by Mr. T. Palvio, Development Chemist, for evaluation in concrete mixes.

SCOPE OF INVESTIGATION

Brucitic limestone has been used for concrete work in the Wakefield area but, as far as is known, only as the coarse aggregate or crushed stone. Very little is known on the use of this material in concrete as fine aggregate or sand.

The relatively high magnesia content necessitates the testing of the rock incorporated in concrete both as a coarse and fine aggregate.

The strength development and soundness of the concrete was determined on test specimens under standard moist-curing conditions.

Frost resistance was determined by freezing and thawing tests, conducted according to the ASTM test method C 291-61T by rapidly repeated cycles of freezing in air and thawing in water.

Visual examination of concrete test specimens and the study of weight, length and ultrasonic pulse velocity changes provided necessary information for evaluation of concrete durability and soundness of the aggregate.

BRUCITIC LIMESTONE AGGREGATE

Crushed brucitic limestone separated in screened fractions (Table 5) for coarse and fine aggregate in quantities sufficient for concrete test mixes was supplied by the Aluminum Company of Canada, Limited, from its Wakefield Works.

Petrographic Description (1)

The term "brucitic limestone" is used to designate limestone containing granules of brucite evenly disseminated in a carbonate matrix. Brucite consists of magnesium hydroxide and is a soft mineral with a hardness between that of gypsum and talc. It has a pearly lustre, and most commonly is colourless, white, grey, or pale green. Wetting the specimen makes the contrast between the brucite and the limestone more apparent. Outcrops of brucitic limestone have a distinctively pitted surface caused by the less resistant brucite being removed by weathering agencies and leaving pits in the limestone of the same size and shape as the original brucite granules.

Mineralogical Analysis*

A thin section of the brucitic limestone aggregate from Wakefield, Que., and a polished section of a concrete beam were used for microscopic study. Staining techniques were used to assist in the identification of minerals.

The aggregate consists of fragments of brucitic limestone up to 3/4 in. size, varying in colour from white to dark grey. The colour darkens with increasing proportions of brucite, dark serpentine, graphite and sulphide. The distribution of the coarse aggregate particles in concrete by the colour and brucite content is approximately as follows:

1. Dark particles, per cent ---------------------- 40
   (> 20% brucite; 5 to 30% serpentine, graphite)
2. Med. grey particles, per cent ------------------ 20
   (5 to 20% brucite; 5 to 30% serpentine, etc.)
3. Light particles, per cent --------------------- 40
   (< 5% brucite; 0-30% serpentine, etc.)

The average (approximate) mineralogical composition, from a study of the minus 3/4 to plus 1/2 in. material and polished sections of the concrete, is given in Table 1.

---

* From the Mineralogical Report, Sample No. CM-134, June 13,1962 by Dr. J.A. Soles, Mineralogist, Mineral Processing Division, Mines Branch.

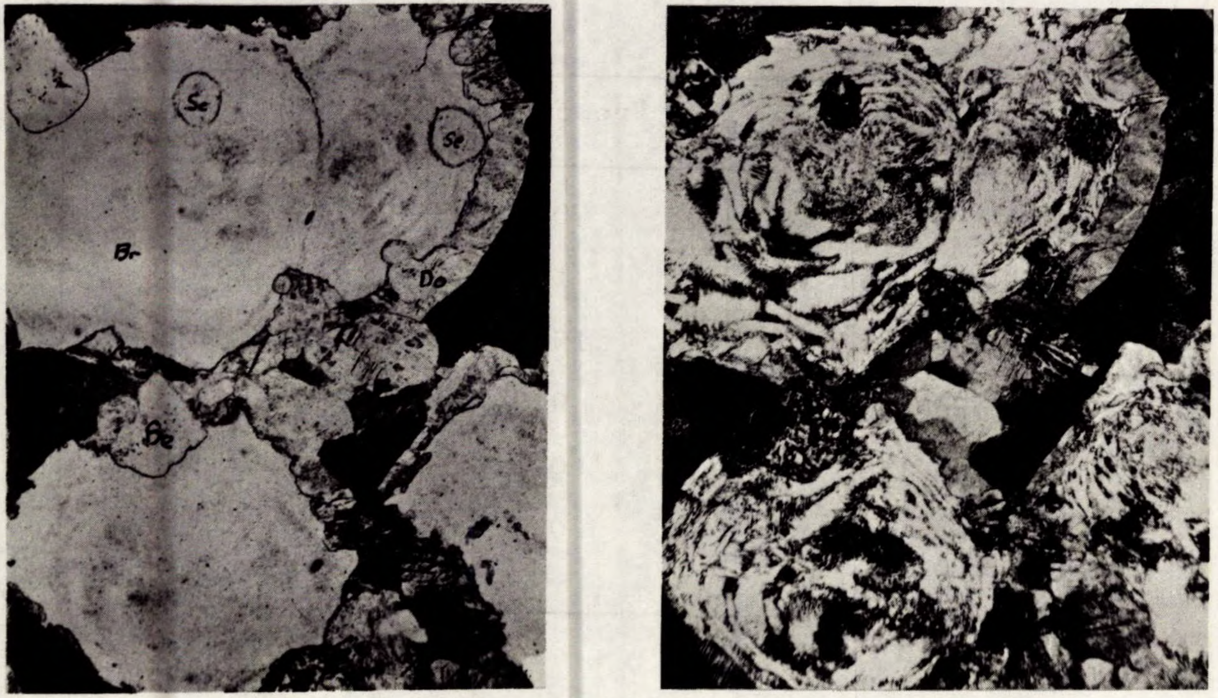
TABLE 1

Mineralogical Composition

Mineral	Proportion, %	Grain Size, mm
Calcite	70.0	0.2 to 4
Dolomite	1.0	0.2 to 2
Brucite	19.4	0.5 to 2
Serpentine	9.0	0.1 to 1
Graphite	0.1	0.01 to 0.1
Fe-sulphide, oxide	0.5	0.1 to 0.5

Two photomicrographs of the brucitic limestone are given in Figure 1, showing brucite as spherulitic growths of fibres in a medium-grained matrix of calcite. Dolomite occasionally rims brucite, serpentine nodules are ubiquitous, and graphite and sulphides are scattered.

A more detailed description of brucite granules in the limestone from Wakefield, Que., is given by Goudge (1) and Buchanan(2).



(Polarized light)

(Cross-polarized light)

Figure 1. Photomicrograph Showing Concentric Growth of Fibrous Brucite (Br) Granules in Calcite (dark), Nodules of Serpentine (Se), and Dolomite (Do) Peripheral to Brucite.

(Magnification X32)

Chemical Analysis *

A chemical analysis of a representative sample of brucitic limestone from Wakefield, Que., used for the concrete aggregate in this investigation is given in Table 2.

TABLE 2

Chemical Analysis

Chemical Constituents	Per Cent (by weight)
Magnesium Oxide	20.0
Calcium Oxide	35.0
Loss on Ignition	37.0
Silicon Dioxide	5.0
R_2O_3	3.0
Total	100.0

Brucite consists of magnesium hydroxide, $Mg(OH)_2$, and has a magnesia content of 69.1% and water content of 30.9% (1) Analyses of the brucitic limestone from the various deposits in this area are remarkably uniform and show magnesium oxide and calcium oxide to be present in proportions closely corresponding to those of pure dolomite. This supports the theory that deposits of brucitic limestone were formed from dolomite by thermal metamorphism, during which the magnesium carbonate of the dolomite dissociated to form the oxide (periclase) and carbon dioxide, while the calcium carbonate, owing to its higher temperature of dissociation, is not broken down. The periclase so formed is an unstable mineral and changes by hydration to brucite.

$$CaMg(CO_3)_2 \longrightarrow CaCO_3 + CO_2 + MgO \text{ (periclase)}$$
$$MgO + H_2O \longrightarrow Mg(OH)_2 \text{ (brucite)}$$

---

* Chemical analysis was supplied in a letter dated March 7, 1962, by Mr. T. Palvio, Development Chemist, Aluminum Company of Canada Limited, Wakefield, Que.

Specific Gravity and Absorption Tests

Bulk specific gravities, (saturated, surface-dry basis) and absorption percentages of the aggregate samples were determined in accordance with ASTM, Standard Methods of Test C 127-59 and C 128-59. Test results are given in Table 3.

TABLE 3

Specific Gravity and Absorption

Aggregate Sample	Specific Gravity	Absorption %
Coarse Agg. (-$\frac{3}{4}$ in. + No. 4)	2.55	1.43
Fine Agg. (-No. 4)	2.40	1.53

Sulphate Soundness Tests

Brucitic limestone coarse aggregate was tested for soundness in accordance with the ASTM Test Method C 88-59T.

In this test three size fractions of the aggregate were subjected to 5 cycles of alternate immersion and drying, using saturated magnesium sulphate solution.

Test results are shown in Table 4.

TABLE 4

Sulphate Soundness Losses

Size Fractions	Original Fractions, %	Fraction Loss, %	Weighed Loss, %
1/2 to 3/4 in.	35	4.5	1.58
3/8 to 1/2 in.	30	5.6	1.68
No. 4 to 3/8 in.	35	6.4	2.24
Totals	100		5.50

The soundness loss of 5.5% obtained in these tests is well below the specification limits set up by the different agencies, as follows:

1. CSA Standards for Concrete
   A23-1960, Sect. 4.5.41------------15%

2. ASTM Std. Specifications for
   Concrete Aggregates C33-59--------18%

3. Dept. of Highways, Ontario
   Specs for Concrete Pavements, Sect. 502---12%

CONCRETE TEST MIXES

Two identical air-entrained concrete test mixes were prepared. One was made with crushed brucitic limestone as coarse and fine aggregate. The other mix was made with natural sand, using the same type of coarse aggregate.

Materials

Crushed brucitic limestone aggregate was supplied in separate sizes by the Aluminum Company.

Natural concrete sand from laboratory stock was used as fine aggregate in the second test mix.

The aggregates were blended to meet ASTM gradings shown in Table 5.

TABLE 5

ASTM Aggregate Gradings

Coarse Aggregate		Fine Aggregate	
Sieve Size	Per Cent Passing	Sieve Size	Per Cent Passing
3/4 in.	100.0	No. 4	100.0
1/2 in.	65.0	No. 8	90.0
3/8 in.	35.0	No. 16	67.5
No. 4	0.0	No. 30	42.5
		No. 50	20.0
		No. 100	6.0

A normal portland cement, produced by the Canada Cement Company Limited, Plant No. 3, Hull, Que., was used in all test mixes. The chemical analysis and compound composition of this cement are shown in Table 6.*

TABLE 6

Chemical Analysis of Cement

Chemical Analysis	%	Compound Composition	%
SiO_2	21.05		
Al_2O_3	5.66	C_3S	47.1
Fe_2O_3	3.03	C_2S	24.9
CaO, (combined)	62.22	C_3A	9.9
CaO, (free)	0.83	C_4AF	9.2
MgO,	2.75	$CaSO_4$	4.2
SO_3	2.47	MgO Ignition loss Alkalies	4.6
Na_2O	0.48		
K_2O	0.56		
Ignition loss	0.81		
Total	99.86	Total	99.9

Design of Test Mixes

Mixes were designed by the absolute volume method, according to the following conditions:

(a) The ratio of fine to coarse aggregate was expressed in absolute volume percentage as 41:59

---

* Chemical analysis of cement by Canada Cement Company, Limited, Plant No. 3, Hull, Quebec.

(b) Cement content was 5 1/2 bags per cubic yard of concrete. Discrepancies of ± 1/10 bag were allowed to meet the desired unit weight and slump.

(c) An air-entraining admixture (Darex) was added in amounts sufficient to produce 6±1% air, as measured by a pressure-type air meter.

Preparation of Mixes

Each mix was prepared as a 2 cu ft batch. Aggregate was soaked in water for 24 hours before use. At the end of this soaking period the excess of water was removed by draining for one hour, and the wet aggregate was weighed to determine the amount of absorbed and free surface water.

A counter-current, Lancaster type 2 cu ft mixer was used. After all materials were added, the constituents were mixed for 2 minutes, allowed to rest in the mixer for 2 minutes, then re-mixed for additional 2 minutes.

The two test mixes were made on two consecutive days, September 13 and 14, 1961.

Properties of Freshly Mixed Concrete

The test mix made with brucitic limestone, both as coarse and fine aggregate, showed abnormal temperature rise and premature stiffening of the concrete. Extra water was added three times to keep the concrete mixture workable until all test specimens were cast in moulds.

Unit weight, slump and air content were determined for each test mix. This data and the computed mix proportions for one cubic yard of concrete are shown in Table 7.

TABLE 7

Concrete Mix Data

	Test Mixes	
	No. 225	No. 226
Type of Aggregate		
Coarse Aggregate------	Bruc. Limestone	Bruc. Limestone
Fine Aggregate ------	Bruc. Limestone	Natural Sand
Mix Proportions (per cu yd)		
Cement, lb ------------	472	474
Coarse Aggregate, lb -	1671	1710
Fine Aggregate, lb ---	1160	1261
Water, lb ------------	417	305
Total Weight, lb/cu yd.	3720	3750
Darex, oz	5.5	4.5
Mix Properties		
Unit Weight, lb/cu ft -	137.75	138.9
Slump, in. -----------	3	3
Air Content, % --------	2	5 1/2

Moulding and Curing Test Specimens

A series of seven 4 x 8 in. test cylinders and nine 3 1/2 x 4 x 16 in. test beams were moulded from each of the two batches of concrete.

Test cylinders were moist-cured until they were tested for compressive strength at the age of 7 and 28 days, and at the end of the freezing-and-thawing test.

Beam specimens were moist-cured for 14 days. Six beams of each batch were placed in the freeze-thaw unit, the three companion beams were kept in the moist-curing room for reference throughout the freeze-thaw cycling period.

TESTS ON STANDARD CURED CONCRETE

Test Procedures and Results

For the purpose of comparison of the two concrete mixes, the tests were conducted to determine the following properties of the standard moist-cured concrete test specimens:

1. Density of concrete
2. Compressive strength
3. Flexural strength
4. Weight change
5. Length change
6. Ultrasonic pulse velocity

Density of concrete was determined after 14 days of moist-curing in saturated, surface dry condition.

Compressive strength measurements of standard moist-cured test cylinders were made at 7 and 28 days' age.

While test beams of series 225 deteriorated rapidly in freezing and thawing, control beams and cylinders stored in the moist-curing room were kept for long-term observation and measurements. Finally these beams were tested at the end of the nine months period.

The three moist-cured control beams of series 226, together with their companion freeze-thaw specimens, were measured for length, weight and ultrasonic pulse velocity at the beginning and at the end of the freeze-thaw cycling, when they were broken in flexure by the third-point loading method (ASTM C 78-59).

Data obtained on the standard moist-cured test specimens were averaged from three test results and are compiled in Table 8.

Discussion of Test Results

The density of hardened concrete series 225, made with all-brucitic limestone aggregate was 1.76 lb lower per cu ft than that of series 226, in which fine aggregate of a higher specific gravity was used.

TABLE 8

Test Results on Standard Cured Test Specimens

Tests	Test Mixes	
	No. 225	No. 226
Density, SSD, lb/cu ft (14 days moist-cured)	139.28	141.04
Cylinder Compr. Strength		
7-day, psi	1735	2365
28-day, psi	2465	3080
135-day, psi	----	3685
270-day, psi	2820	----
Tests on Beams		
Flexural Strength:		
135-day, psi	---	565
270-day, psi	545	---
Weight change, 14-day, %	+0.9	+0.9
135-day,%	+1.5	+1.5
Length change, 14-day, %	+0.0128	-0.0012
135-day,%	+0.0245	+0.0035
Pulse Velocity:		
V_o, 14-day, fps	13020	14290
V_{fin}, 135-day, fps	-----	15020
V_{fin}, 270-day, fps	13710	-----
Velocity change, %	+5.5	+5.1

The 28-day compressive strength of concrete series 226 was about 25 per cent higher than that of series 225. This may be attributed to the difference in water/cement ratios of the two mixes. The higher water consumption in the mix was caused apparently by some chemical reaction and an abnormal temperature rise during the mixing.

No direct comparison of flexural strength of the two concrete mixes is possible becausd test beams were broken at different ages. It is apparent from the test results that a higher strength was shown by test beams of series 226. That is confirmed also by about 10% higher initial pulse velocity (V_o) of series 226 test specimens.

The gain in weight during moist-curing was the same for both series of concrete, i.e. 0.9 and 1.5 per cent after 14 and 135 days, respectively. These fairly high values of weight gain are due to the high absorption of brucitic limestone.

The length measurements in series 225 indicated continous expansion of test specimens during the moist-curing period, whereas measurements in series 226 showed shrinkage at 14 days, followed by expansion of test specimens at later ages. The average expansion of concrete test beams after 135 days of moist-curing in series 225 was 0.0245%, whereas in series 226 at the same age only 0.0035%. Linear expansion of concrete series 225 was seven times greater than that of series 226. However, this amount of expansion is not an indication of concrete deterioration under moist-curing conditions. The fact that concrete remains structurally sound is substantiated by the increase of compressive strength and ultrasonic pulse velocity of test specimens during the moist-curing time.

Similar concrete test beams prepared by this laboratory with a shaley limestone coarse aggregate, after 140 days of continuous moist-curing, showed 0.05% expansion without any noticeable effect on the normal concrete strength gain.

The U.S. Bureau of Reclamation has set a tentative limit of 0.07% as a maximum permissable expansion after 300 cycles of wetting and drying for acceptable concrete in exposed structures.

CONCRETE DURABILITY STUDIES

The objective of these tests was to study the durability of concrete made with brucitic limestone aggregate. Although durability cannot be measured directly, prolonged exposure of concrete to repeated freezing and thawing may produce measurable changes in test specimens. These changes may indicate internal deterioration of concrete. Measurements made on the test specimens after repeated freezing and thawing produce data which could be used for evaluating the relative frost resistance or durability of concrete.

Freezing and Thawing Procedure

In this investigation, concrete durability was studied by exposing test specimens to rapidly repeated cycles of freezing in air and thawing in water according to the ASTM test method C 291-61T. One complete cycle, from 40±3 °F to 0±3 °F and back to 40±3 °F, required about 3 hours. The automatic freeze-thaw unit * used in this investigation produces 8 cycles in 24 hours.

---

* Manufactured by the Canadian Ice Machine Company, Ltd., Toronto, Ont.

After 14 days of initial moist-curing, the temperatures of all test beams were reduced to 40±3 °F by placing them in the freeze-thaw cabinet at the thawing phase of the cycle for one hour prior to testing. Initial weights, lengths, and ultrasonic pulse velocity measurements of all beam specimens were taken at this temperature. Six beams of each batch were then placed in the freeze-thaw cabinet and subjected to repeated cycles of freezing in air and thawing in water.

The three companion beams were kept in the moist-curing room for reference purposes throughout the freeze-thaw cycling period.

Test Procedures and Results

The following measurements and tests were made to evaluate the resistance of concrete test beams to accelerated freeze-thaw cycling:

1. Measurement of length changes
2. Determination of weight changes
3. Measurement of ultrasonic pulse velocities
4. Determination of moduli of rupture
5. Visual examination of test specimens.

The freeze-thaw beam specimens were weighed and measured for length changes once a week. At the same time beams were tested for signs of internal deterioration by measuring ultrasonic pulse velocities. All measurements were made at a temperature of 40±3 °F. A visual appraisal was made of the beams for any surface deterioration.

Concrete of series 225 under exposure to freezing and thawing deteriorated rapidly, developing map-cracking on the surfaces of test beams at the end of first days' cycling. During the next day, cracking of concrete resulted in complete disintegration of test specimens. At the end of 16th cycle, when lifted by the end, the beams broke under their own weight.

Great care was used to remove all six test specimens from the freezer chest in an unbroken condition. Figure 2 shows the cracked test beams 6 and 9 of series 225 after 16 cycles of freezing and thawing.

No length measurements of these test specimens were possible because beams expanded beyond the maximum range of the laboratory comparator. Also impossible were the ultrasonic pulse velocity measurements because the contact points on the beam ends had been damaged.

These tests indicate that the durability of concrete series 225 was very poor and it could not resist the destructive action of freezing and thawing.

The three companion beam and cylinder specimens of this series had been kept in the moist-curing room for testing purposes at a later age. The results of these test specimens after 270 days of moist-curing have also been included in Table 8.

The test specimens of series 226 were more durable. Freeze-thaw cycling was continued until the end of 297 cycles, when three beams were measured and broken. The test on the remaining three beams was continued till 994 cycles of freezing and thawing. The final measurements were then taken on the three freeze-thaw and also on the three standard-cured companion reference specimens. The test results on freeze-thaw specimens for both series are compiled in Table 9. For series 225 only initial measurements were made.

TABLE 9

Test Results on Freeze-Thaw Test Specimens

Tests	Test Mixes	
	No. 225	No. 226
1. Flexural Strength		
Stand. moist-cured, psi- - - -	546 (270d)	565 (135d)
After 297 cycles (37d), psi- -	*	570
" 994 " (135d), psi- -	*	463 (-18.0%)
2. Weight Change		
After 994 cycles, %- - - - - -	*	-0.3
3. Length Change		
After 297 cycles, %- - - - - -	*	+0.026
" 994 " , %- - - - - -	*	+0.064
4. Pulse Velocity		
Vo, 0 cycles, fps- - - - - - - -	13,020	14,290
V_{fin}, 297 cycles, fps- - - - -	*	14,160(-0.9%)
V_{fin}, 994 " , fps- - - - -	*	12,780(-10.6%

* No measurements made because test was discontinued after 16 cycles of freezing and thawing.

Visual examination of the test specimens of series 226 after 994 cycles of freezing and thawing showed that test beams were in a fairly good condition with few "pop-outs and some corner damage only (Figure 3).

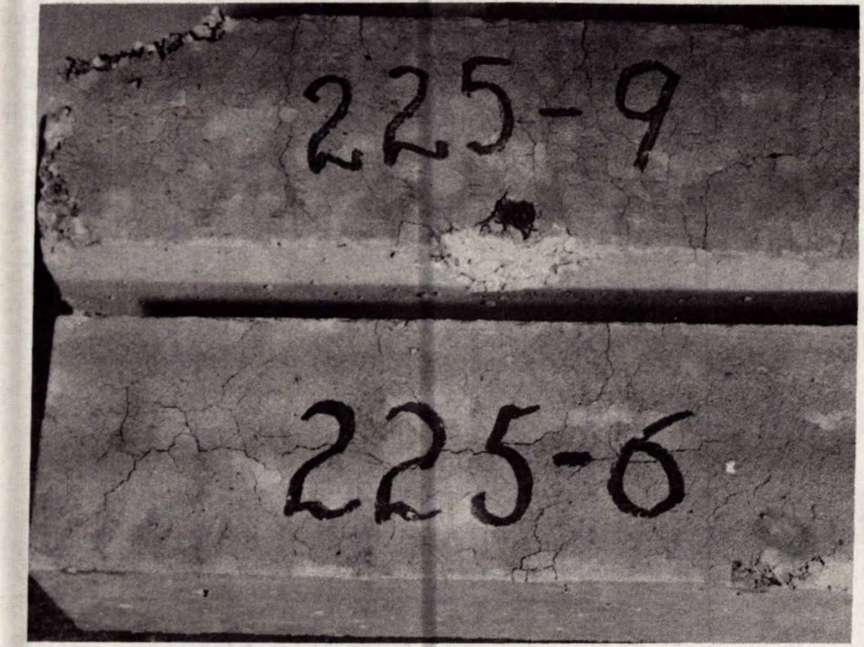


Figure 2. Deteriorated Test Beams of Concrete Series 225 Showing Extensive Cracking After 16 Fr.-Th. Cycles

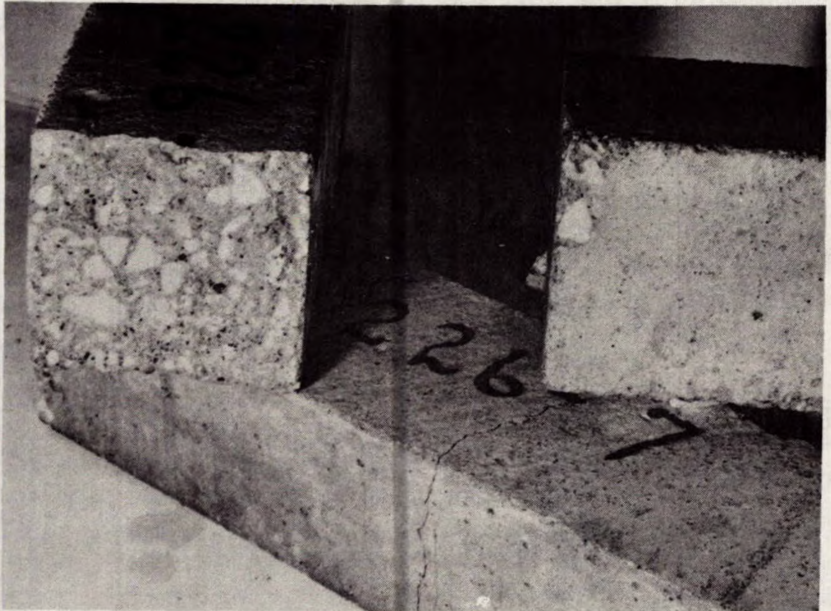


Figure 3. Test Beams of Concrete Series 226 Showing Good Shape After 994 Fr.-Th. Cycles.

Discussion of Test Results

The rapid deterioration of concrete specimens of series 225 in freezing and thawing indicate the poor durability of concrete made with crushed brucitic limestone sand. The abnormal rise in temperature during the mixing required addition of more water and more of the air-entraining agent (Darex A.E.A.). However, the air content in the mix finally measured was only 2% which is not sufficient for the durability of concrete.

The origin of the exothermic reaction causing flash set of cement with brucitic limestone sand should be studied further.

The test specimens of series 226 were in excellent conditions after 297 freeze-thaw cycles. Even after 994 cycles test beams were still in good shape with the flexural strength reduced by only 18% and length increased by 0.064%.

Paul Klieger(3) has set 0.07% as the limit of expansion for durable concrete after 300 cycles of freezing and thawing. That will be equivalent approximately to a 40 per cent reduction of the dynamic modulus of elasticity.

Ultrasonic pulse velocity values obtained on concrete test beams before and after freeze-thaw cycling were used to compute the relative dynamic moduli of elasticity, which are directly proportional to the squares of pulse velocities(4). The numerical value of relative dynamic modulus of elasticity, in per cent was calculated by the following equation:

$$P_c = \frac{V^2_{fin}}{V^2_o} \times 100$$

Where: P_c = relative dynamic modulus of elasticity in per cent.
cycles of freezing and thawing
V_o = initial ultrasonic pulse velocity.
V_{fin} = final ultrasonic pulse velocity in concrete specimens after C cycles of freezing and thawing.

The relative dynamic modulus of elasticity P_c for concrete test beams of series 226 after 994 cycles of freezing and thawing averaged to

$$P_{994} = 80\%$$

This is another indication of good durability of concrete series 226.

CONCLUSIONS

1. The sample of brucitic limestone from the Wakefield area, when incorporated as coarse aggregate in air-entrained concrete, showed good strength performance and durability when used with natural sand as fine aggregate.

2. The use of sand manufactured from brucitic limestone seems to impart undesirable properties to concrete, and further investigation work is recommended.

REFERENCES

1. MF. Goudge, "Brucite", The Geology of Canadian Industrial Mineral Deposits, Sixth Commonwealth Mining and Metallurgical Congress, p. 61-69 (1957)

2. R.M. Buchanan, "A Microscopic Examination of Brucite Granules in Limestone from Wakefield, P.Q.", Mines Branch Investigation Report IR 58-159 (1958)

3. P. Klieger, "Studies of the Effect of Entrained Air on the Strength and Durability of Concretes Made with Various Maximum Sizes of Aggregate", Proceedings, Highway Research Board, Vol. 31, p. 177, Table 15 (1952)

4. K.B. Woods, "Application of Pulse Velocity Tests to Several Laboratory Studies of Materials," Bulletin 206, Highway Research Board, p. 19 (1959).