

Dr. J. Connelley

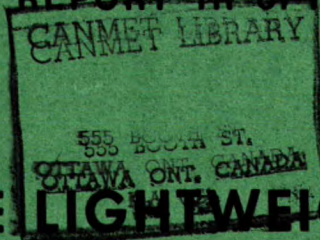
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DEPARTMENT OF MINES AND TECHNICAL SURVEYS

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MINES BRANCH INVESTIGATION REPORT IR 61-116



INVESTIGATION OF SOME LIGHTWEIGHT AGGREGATE PROPERTIES OF UTICA SHALE FROM DELSON, P. Q.

by

H. S. WILSON

MINERAL PROCESSING DIVISION

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CONTENTS

	<u>Page</u>
Summary of Results.....	ii
Introduction.....	1
Scope of the Investigation.....	1
Preparation of Test Sample.....	2
Firing Tests.....	2
Composition of the Raw Shale.....	4
Composition of the Aggregates.....	9
Discussion of Results.....	10
Conclusions.....	12
Acknowledgements.....	13

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OF UTICA SHALE FROM DELSON, P.Q.

by

H. S. Wilson*

SUMMARY OF RESULTS

The sample of shale tested appears to possess bloating properties that would make it a suitable raw material for the production of a coated lightweight aggregate. The temperature at which the shale was fired had a greater effect on the crushing strength of the aggregate than did the rate of cooling.

The amounts of sulphur, water soluble sulphate, and lime impurities in the aggregate, should have no detrimental effect on the strength properties of concrete.

The raw shale is composed of illitic and chloritic clay minerals, quartz, calcite, pyrite, and organic matter.

*Senior Scientific Officer, Mineral Processing Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

INTRODUCTION

Mr. H. V. Henderson, General Production Manager of Cooksville-Laprairie Brick Limited, requested that the Mines Branch carry out an investigation on a sample of Utica shale from the company's quarry at Delson, P.Q. The company plans to produce lightweight aggregate using this shale as raw material. The purpose of the investigation is to obtain information on the bloating characteristics of the shale, and properties and composition of the resulting aggregate.

SCOPE OF THE INVESTIGATION

The specific information requested by the company is as follows:

1. The effect of firing conditions on the bloating properties;
2. Crushing strength and density of bloated clinker and crushed aggregate;
3. Suitability of this shale for coated aggregate as well as the more conventional crushed aggregate;
4. Pyrite burn-out and associated transformations -
 - (a) The sulphur and soluble salts (if any) remaining in the bloated clinker;
 - (b) An estimate of the permissible sulphur level of the aggregate for use in structural concrete;
 - (c) The effects of sulphur and other soluble salts on the concrete and **on the reinforcing steel contained therein;**
5. The lime transformation during bloating and the free lime content in the clinker, if any;

6. Clinker cooling -
 - (a) The effect of water quenching,
 - (b) The effect of forced air quenching,
 - (c) The effect of slow protective cooling by prevailing ambient temperatures,
 - (d) The critical cooling rate, or point, if such exists, in order to determine the temperature at which rapid cooling might be applied;
7. Differential thermal analysis of the raw shale.

PREPARATION OF TEST SAMPLE

The sample of shale, weighing about 1200 pounds, was received in lumps up to about 8 inches in size. It was passed through a jaw crusher and separated on $1\frac{1}{2}$ and $\frac{1}{2}$ inch screens into 3 fractions. The plus $1\frac{1}{2}$ inch fraction was recirculated through a jaw crusher which reduced the size to minus $1\frac{1}{2}$ inches. The investigation was restricted to the minus $1\frac{1}{2}$ plus $\frac{1}{2}$ inch size of material to conform with the company's production plans. The sized shale used in firing tests was dried prior to each test.

FIRING TESTS

A 12 in. I D gas-fired rotary kiln, 12 ft in length was used for the tests. A $2/3$ in. per ft slope on the kiln, which was rotated at 2 rpm, gave a retention time of approximately 20 min.

The company requested that the material be fired, at a temperature that would agglomerate or clinker the shale pieces, to conform with the proposed production process. Firing at this

temperature was unsuccessful. Because of the relatively small diameter of the kiln, pieces of bloated shale formed rapidly into logs which would not discharge from the kiln. Consequently, it was necessary to maintain the temperature at or below the agglomerating temperature.

Tests were made at various temperatures to determine the range through which bloating took place. The rate of feed to the kiln was about 50 pounds per hour. Approximately 75 pounds of shale was used for each test. Three methods of cooling the products were employed in order to determine if the rate of cooling affected the resulting strength of the aggregate. The methods were:

1. Water quenching - the aggregate was discharged into a tub filled with constantly flowing cold water;
2. Forced air quenching - the aggregate was discharged into a tub into which air was blown by an electric fan;
3. Slow protective cooling - the aggregate was discharged into a drum which was insulated around the circumference and on the bottom by 2 inches of exfoliated vermiculite and on top by $2\frac{1}{2}$ inches of insulating refractory brick.

A representative sample of about 10 pounds was cut from each product, crushed, screened, and recombined in the following proportion:

75% minus $3/8$ inch plus 4 mesh

25% minus 4 plus 8 mesh

This grading is within ASTM limits for coarse lightweight aggregate. The loose dry unit weight and crushing strength were determined for the graded aggregate. The method used to determine the crushing strength was to compact the aggregate placed in a 3 inch diameter

steel cylinder to a depth of 5 inches. The two pressures required to compact it 1 inch and a total of 2 inches are the figures reported as crushing strength.

The firing temperatures, methods of cooling, unit weights, and crushing strengths are shown in Table 1.

TABLE 1
Firing Tests

Test No.	Temperature (°F)	Method of Cooling	Unit Weight (lb/cu ft)	Crushing Strength (psi)	
				1"	2"
1	1970-2000	Water	32.1	165	920
2	1990-2030	Air	31.6	230	920
3	1980-2000	Slow	33.4	170	870
4	1970-2000	Water	36.6	190	1250
5	1965-2000	Air	37.6	230	1260
6	1970-2010	Slow	37.8	250	1340

COMPOSITION OF THE RAW SHALE

A. Chemical Composition

The chemical composition of the raw shale is shown in Table 2.

TABLE 2

Chemical Analysis of Raw Shale

Constituents	Weight Per Cent
SiO ₂	59.00
Al ₂ O ₃	16.10
FeO	3.65
Fe ₂ O ₃	2.20
TiO ₂	0.74
CaO	1.92
MgO	3.78
Total C	1.19
Elemental C	0.67
CO ₂	1.93
S	1.17
L O I	7.57
Moisture	0.67
H ₂ O	4.66
Quartz	30.46
Na ₂ O	0.93
K ₂ O	3.63

B. Mineralogical Composition

Mineral constituents of the raw shale were identified by means of an X-ray diffractometer and by differential thermal analysis (DTA).

Prior to X-ray examination, the sample of shale was reduced to about 8 mesh, by a jaw crusher. Approximately 75 grams of the minus 10 plus 18 mesh fraction was obtained with a Jones splitter for examination. The fragments of shale were washed and boiled for 24 hr in 1000 ml of distilled water. The minus 1/64 mm fraction was sedimented onto glass slides. The resulting specimens were run twice in the diffractometer under different humidity conditions and once after heating to 600°C (1112°F) for $\frac{1}{2}$ hr. Powder photographs were taken in the diffractometer before and after the specimens were soaked in boiling, 20% hydrochloric acid.

The results indicated that the sample of shale contained illitic clay, quartz, and chloritic clay that showed partial variation of basal spacings with changes in humidity. A minor amount of plagioclase feldspar was also identified.

Another portion of the shale was reduced in size to minus 100 mesh by a jaw crusher, pulverizer, and a porcelain mortar and pestle. About 2 grams of the sample was used in the DTA test.

The DTA curve shown in Figure 1 indicates that this material is a typical Utica shale. The curve was obtained under two different conditions; first in an oxygen atmosphere up to 700°C (1292°F) to concentrate the burn-off of oxidizable material in a narrow temperature range and, second, in a carbon dioxide atmosphere above 700°C to accentuate any peaks due to carbonate decomposition. The sample was heated at a rate of 12°C/min. In Figure 1 exothermic reaction peaks point up and endothermic reaction peaks point down.

The endothermic peak at 120°C (248°F), is caused by the loss of adsorbed moisture from the clay minerals. The endothermic peak at 580°C (1076°F) and the endothermic - exothermic doublet in the 900 to 950°C (1652 to 1742°F) temperature range are caused by a mixture of illite and chlorite. The very intense double exothermic peak between 400 and 500°C (752 and 932°F) is caused by the oxidation of a small amount of pyrite and organic material, the amount being greater than in most Canadian shales. A DTA curve obtained in an air atmosphere indicates that this oxidizable material burns out between 400 and 670°C (752 and 1238°F) at a heating rate of 12°C/min. A small endothermic peak at 800°C (1472°F) indicates the presence of a small amount of calcite.

The most unusual feature of Figure 1 is the large exothermic reaction caused by the oxidation of carbonaceous material and pyrite. The amount of these minerals may not exceed 3% but the heat effect is very large.

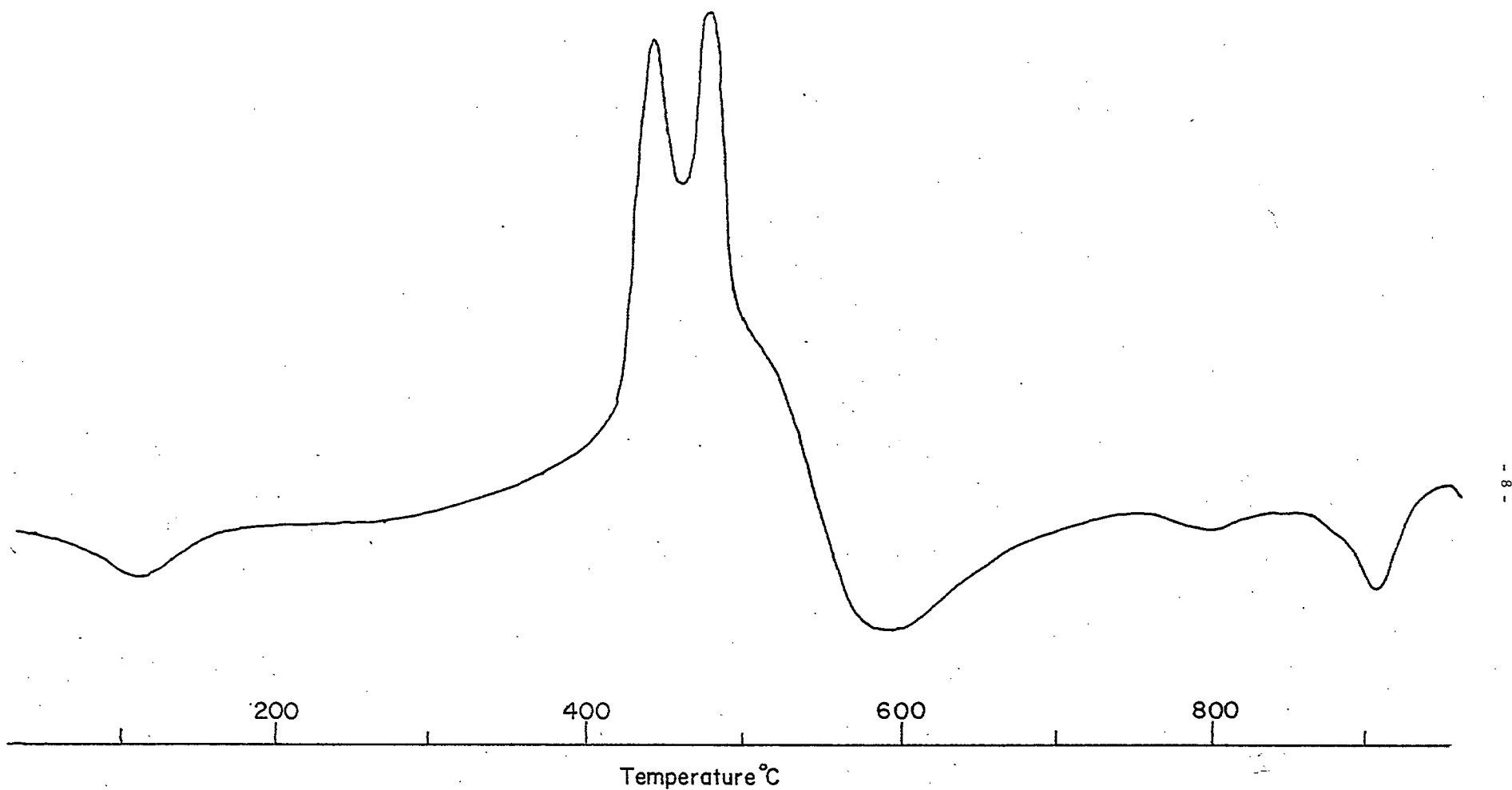


FIGURE I DTA CURVE RAW SHALE

COMPOSITION OF THE AGGREGATES

A. Chemical Composition

The composition of the water-quenched aggregate produced in Test 1 was determined chemically, and is shown in Table 3.

TABLE 3

Chemical Analysis of Water-Quenched Aggregate

Constituents	Weight Per Cent
SiO ₂	64.06
Al ₂ O ₃	16.62
FeO	1.72
Fe ₂ O ₃	5.00
CaO	2.10
MgO	3.70
Na ₂ O	1.00
K ₂ O	3.92
Elemental S	0.69
Elemental C	0.14
CO ₂	n.d.*
Water Soluble SO ₄	0.15
Ca(OH) ₂	n.d.*
L. O I	0.10
	<u>99.20</u>

* not detected

The water soluble sulphate content of the slow-cooled aggregate produced in Test 3 was determined as 0.51 per cent, and only a trace of calcium hydroxide was found.

B. Mineralogical Composition

Mineralogical examination of the two aggregates was made by means of an X-ray diffractometer. It showed the materials to be poorly crystallized, the only minerals identified being quartz and feldspar. The composition of the glassy parts of the aggregates could not be determined mineralogically.

DISCUSSION OF RESULTS

The temperature range, below the agglomerating temperature, through which bloating occurred in the rotary kiln was about 65 degrees occurring between 1965° and 2030°F. The unit weights of graded coarse aggregates produced varied from 31.6 to 37.8 lb/cu ft, depending on the temperature. The maximum unit weight specified by ASTM for aggregate of this grading is 55 lb/cu ft. The tests indicate that a coated lightweight aggregate could probably be produced from this shale.

The firing temperature appears to have had more effect on the crushing strength of the aggregate than did the rate of cooling. Considering the six tests as two series, the rates of cooling did not appreciably affect the crushing strengths in either series. The temperature limits in Tests 1 and 4 (Table 1) were identical, as were the methods of cooling the products. The unit weights and crushing strengths differed, however. The mean temperature in Test 1 was closer to the upper limit than in Test 4. This resulted in a lighter, weaker product from Test 1.

The chemical analysis of the raw shale shown in Table 2 includes both SiO₂ and quartz. The shale sample contains 30.46 per cent free quartz, the total silica content being 59 per cent. This

table also includes moisture and H_2O . The value for moisture refers to free moisture in the sample, and H_2O refers to total water.

The chemical analysis of the water quenched aggregate (Table 3) showed it contained no free lime and only small amounts of water-soluble sulphate and elemental sulphur. The aggregate contains 2.10 per cent CaO , so it must be present as a silicate. Apparently, water quenching of the aggregate leached out most of the soluble sulphate because the slow-cooled aggregate contained a greater amount than did the water quenched aggregate.

It is doubtful if the amounts of soluble sulphate of elemental sulphur contained in these aggregates would have a detrimental effect on the strength properties of concrete or on reinforcing steel. There are no standards limiting the sulphur or water-soluble sulphate contents of aggregates. The limit would depend on such factors as cement composition, impurities in the mixing water, and properties and environment of the concrete. A concentration of 0.5 per cent water-soluble sulphate might cause efflorescence on concrete. The permeability and environment of the concrete could be contributing factors to this effect. A concrete of relatively high permeability exposed to frequent wetting and drying would be more susceptible to efflorescence than one of low permeability or one protected from precipitation.

The mineral constituents of the raw shale as identified by X-ray diffractometer were illitic and chloritic clay minerals, quartz, and a minor amount of plagioclase. Differential thermal analysis showed illitic and chloritic clay minerals, calcite, pyrite, and organic matter to be present.

CONCLUSIONS

The relatively small diameter (12 in.) of the rotary kiln used in this investigation necessitated that the temperature be kept below that at which agglomeration took place because of the formation of logs of expanded material above the agglomerating temperature. It appears possible to produce a coated lightweight aggregate from this shale. Expansion took place below the agglomerating temperature over a temperature range of about 65 degrees. Loose dry unit weights of the graded aggregates, between 31 and 38 lb/cu ft, were well below the limit specified by ASTM.

The method used to cool the aggregate did not appear to be important, in that the rate of cooling did not appreciably affect the crushing strengths of the aggregates. The firing temperature had more effect on the strength than did the rate of cooling. There are no specifications for crushing strength of lightweight aggregate because there is no direct relationship between crushing strength of aggregate and compressive strength of concrete.

Impurities in the aggregates such as elemental sulphur, water-soluble sulphate, and free lime were not in large enough quantities to have any effect on the strength of concrete. Efflorescence might be caused by the amount of soluble sulphate contained in the slow-cooled aggregate, but this defect would also depend on the permeability of the concrete and moisture conditions to which it would be exposed.

Identification of the constituents of the raw shale showed this material to be a typical Utica shale. The clay minerals are illite and chlorite, and other components identified are quartz, plagioclase, calcite, pyrite, and organic matter.

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