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# A STUDY OF THE CAUSE OF DETERIORATION OF AN EXPANDED SHALE IN MOIST, CYCLIC FREEZING AND THAWING CONDITIONS

by

JAMES A. SOLES

MINERAL PROCESSING DIVISION

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A STUDY OF THE CAUSE OF DETERIORATION OF AN EXPANDED SHALE  
IN MOIST, CYCLIC FREEZING AND THAWING CONDITIONS

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SUMMARY OF RESULTS

A petrographic study and freeze-thaw tests were made on an expanded shale concrete aggregate to determine the cause of its instability in moist, freezing and thawing conditions.

The investigation indicated that some aggregate particles are permeable and entrap free water, which expands upon freezing and causes spalling of concrete. No deleterious expansible minerals were detected in the bloated aggregate.

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\*Research Scientist, Ore Mineralogy Section, Mineral Processing Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

## INTRODUCTION

At its Cooksville, Ontario plant, Domtar Construction Materials Limited produces lightweight expanded shale aggregate for concrete from shale of the Dundas formation, using the rotary kiln process. Prior to 1967 the particles agglomerated in the kiln, and were crushed to obtain a product of the desired aggregate sizes, trade-named Haydite. In 1967, the process was changed to produce semi-coated Haydite, a product consisting of bloated, mostly discrete particles with a dense skin of low permeability. This product required little crushing and produces more workable lightweight concrete mixes.

The physical properties of semi-coated Haydite differed from those of the crushed Haydite, and the Company required information on its usefulness as a lightweight aggregate. The Mineral Processing Division was requested to assist in determining the durability of lightweight structural concrete made with semi-coated Haydite when exposed to severe weathering conditions involving cyclic freezing and thawing. A series of tests were therefore made by Zoldners and Wilson (1) to obtain compressive and flexural strength data on specimens subjected to accelerated freezing and thawing, in order to compare them with similar strength data obtained on concrete made with standard gravel.

The concrete test specimens developed 'popouts' on the surfaces after about 500 cycles of freeze-thaw; they were related to unsound aggregate particles located near the surface. The present study was made to determine why the semi-coated Haydite aggregate was deteriorating.

## PROCEDURES

Examples of popouts on concrete specimens exposed to accelerated freezing and thawing test conditions were examined under the stereomicroscope, and samples of deteriorated aggregate particles which had caused spalling of the concrete were taken for petrographic study and X-ray diffraction analysis.

A sample of coarse (1 to 2.5 cm) semi-coated Haydite aggregate was selected from the supply provided by the Company. About 500 particles were cemented lightly with epoxy resin onto 1-mm-opening screens fastened in wire baskets (Figure 1), and were subjected to

194 cycles of freezing in air and thawing in water. The particles were examined carefully at 8- to 24-cycle intervals, to detect and follow the progress of any deterioration which occurred. The screen retained fragments greater than 1 mm that broke free of parent particles.

## RESULTS

### Features of the Aggregate

Stereomicroscope examination of deteriorated particles underlying concrete 'popouts' revealed that three types of deleterious aggregate particles were present:

1. Dark grey, rounded, hard-skinned particles which had ruptured diametrically, exposing a highly vesicular, friable interior;
2. brownish red, flattened, hard laminated particles which had broken parallel to the laminations, exposing friable to hard, flaky surfaces;
3. dark grey, laminated particles similar to type 2.

The loose aggregate contained unbroken particles similar to these three types. The flattened particles were rather poorly bloated and dense, and had occasional weak seams containing hard to friable, flaky material. Type (1) particles were highly expanded with large internal vesicles, a thin dense skin and, often, deep cracks.

X-ray diffraction analyses of friable and flaky material from disintegrated aggregate particles in the concrete revealed only the crystalline phases quartz, minor spinel, and rare plagioclase in all three types, and rare hematite in the reddish particles. Petrographic examination of the same materials immersed in index oils revealed that they consisted of glass with varying proportions of mineral fragments, semi-fused dark to opaque masses, and skeletal crystals. Glassy shards predominated in the fragmented material from the vesicular aggregate.





A.



B.

Figure 1. Photographs of mounted semi-coated Haydite aggregate after exposure to 194 cycles of freezing and thawing. Magnification 1.5X.

- A: Vesicular dark particle (V), broken apart and crumbling into shards in the friable interior;
- B: Platy reddish (Pr) and grey (Pg) particles split parallel to original shaly parting. Small flakes ( ← ) have broken from fresh surfaces.

#### Freeze-Thaw Tests

Exposure of the mounted aggregate particles to cyclic freezing in air and thawing in water produced a gradual breakdown of several particles of the three types noted to have caused spalling in Wilson's concrete specimens. The results of the tests are summarized in Table 1. The deterioration progressed from early splitting along laminations and random cracks to piecemeal spalling, either of flakes from freshly exposed surfaces of laminated particles, or of glassy shards from highly vesicular aggregate particles. Figures 1 and 2 show examples of the deterioration products.

TABLE 1

Disintegration of Haydite Aggregate by Freezing and Thawing

Cycles Completed	No. Particles Affected, Type*	Apparent Changes in Particles	
24	1: (p)	Particles rupture primarily along major cracks or laminae	Split along major cracks.
48	3: (2v, 1p)		Same.
64	1: (p)		Splitting of new particles, some fragmentation of old.
74	3: (1v, 2p)		Slight crumbling of interiors of previously split vesicular particles; flaking of previously exposed surfaces of platy particles.
120	4: (2v, 2p)		Continued disintegration of interiors of well expanded vesicular particles, and disruption of flakes from fresh surfaces of platy particles.
194	1: (p)		Only minor changes of particles affected earlier.
Total	13: (5v, 8p)		

\* (v): vesicular type; (p): platy type.



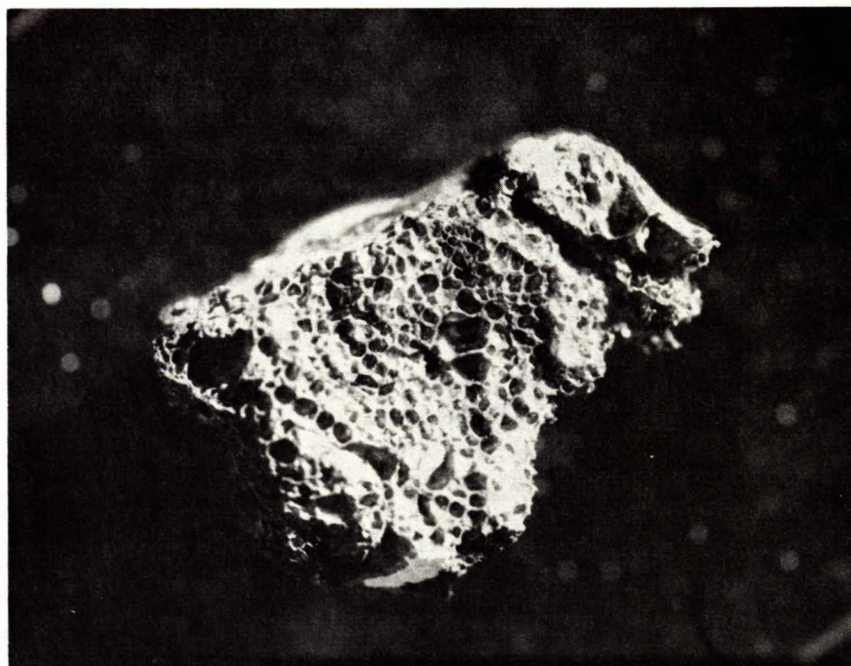


Figure 2. Photograph of broken highly vesicular (Type 1) Haydite particle. Accelerated freezing and thawing disrupts cellular centers, producing glass shards. Magnification X5.

#### DISCUSSION

It appears that the mineralogical composition of semi-coated Haydite aggregate has not been responsible for its deterioration, as the constituents identified are relatively unaffected by cyclic freezing and thawing. No expansible clay minerals are present, even in the flaky laminated material found in poorly expanded, flat aggregate particles; this indicates that the bloating process was probably complete. The degree of expansion of aggregate particles during bloating likely reflects differences in the proportions of expansible minerals in the raw materials (Vrublevskii, 2).

Although no absorptive, deleterious minerals are present, the tests show that the freezing and expansion of absorbed or trapped water does effect the breakdown of the aggregate. Primary rupture takes place along major cracks related to laminations in the particles, and later disruption proceeds either by piecemeal spalling of flakes from the fresh rupture surfaces of flat aggregate, or by crumbling of the highly vesicular centers of well-bloated particles. It appears from these observations that some major cracks are loci of refractory,

non-expansible material which remained permeable after bloating, thereby permitting local entrapment of water for later freezing. The piecemeal spalling of small flakes from freshly exposed surfaces of laminated particles could be an extension of that process, but the granulation of the centers of coarsely vesicular particles required a different process. A possible explanation of their disintegration is that the porous centers have fragile, relatively permeable vesicle membranes which permitted absorption of water after passages were developed.

Zoldners and Wilson (pp. 15, 17) note that concrete made with semi-coated Haydite aggregate has excellent frost resistance, and only particles at the surface deteriorated during freeze-thaw tests. This is consistent with the evidence that no hygroscopic minerals are present in the aggregate, and therefore only readily available water, such as surface water, which could be trapped and frozen in pores would cause deterioration of the concrete. Presumably concrete made with this aggregate would develop surface spalls in freezing and thawing environments only where it was wetted, and would be durable where it was protected from moisture.

#### REFERENCES

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2. Vrublevskii L.E., "On the Causes of Bloating of Clay Rocks", Glass and Ceramics, 19 (1), 20-22 (1962). Transl. from Russian.