PHYSICO - CHEMICAL STUDIES
of
CONCRETE CHIMNEY BLOCKS

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Reporting on Work Plan 78-CT-40

STATE OF VERMONT
AGENCY OF TRANSPORTATION
MATERIALS & RESEARCH DIVISION

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ABSTRACT

The rapidly rising number of failures of concrete block chimneys, some after only a few months use, prompted this study. Samples were taken from five concrete blocks, one new, two several years old but unused, and two used blocks, one of which had obviously been severely exposed to creosote.

A variety of physical tests, as well as chemical analysis for seven elements usually found in concrete, was undertaken. These included density, loss on drying and ignition, compressive strength, absorption, heat cycling, and exposure to creosote.

Although results are inconclusive, they do lead to two possible mechanisms for the failures and point the way toward more significant experiments.

INTRODUCTION

This study was undertaken at the request of the Attorney General of the State of Vermont in an attempt to ascertain the cause or causes for the rapidly increasing number of failures of chimneys constructed of concrete blocks.

The questions to be answered are:

1. By what mechanism or mechanisms is failure induced?
2. Is a concrete block chimney suitable for the conditions encountered in modern woodburning?
3. If not, can the material be made suitable?
4. Why do some chimneys last indefinitely, while others fail after only a few months?
5. Are the samples taken from chimneys that have failed different in any way from new blocks or unused blocks that are several years old?

Chemical analysis and physical testing were undertaken in an attempt to answer these questions.

PROCEDURES AND RESULTS:

Because of the large variety of tests performed, the procedure and results for each are presented together in the interest of simplicity.

Portions of five concrete blocks were tested. Two of these were several years old, but unused, and were designated O-1 and O-2. Two were used and designated U-1 and U-2. The fifth was new and designated N. Samples of aggregate were also tested separately and were designated A. Separate portions were taken from the inside, center, and outside of the used blocks, leading to the designations U-1-I, etc.

Samples for chemical analysis and ignition loss were powdered, but the powder contained fine, gritty particles believed to be harder portions of the aggregate. Specimens for creosote exposure, compressive strength and heat testing were approximately 1" x 1" x 2" in size.

LOSS ON DRYING AND IGNITION

Powdered samples of 3 - 5 gm. were dried to constant weight in platinum (Pt.) crucibles at 105° C, after which the same sample was ignited to constant weight at 950° C. The dried samples remained gray in color. However, the ignited samples all turned rust-colored.

Data for percentage loss on drying and ignition are given in Table 1. There is no apparent trend in the data, other than the comparatively high loss on ignition of both old samples. This can be due either to limestone aggregates or to a large proportion of chemically bound water.

TABLE 1

Loss on Drying and Ignition of Concrete Block Samples

Sample	Loss on Drying (%) (105° C)	Loss on Ignition (%) (950° C)	Total Loss
U-1-I	1.7	3.5	5.2
U-1-C	2.3	4.4	6.7
U-1-O	1.6	4.9	6.5
U-2-I	1.9	5.2	7.1
U-2-C	1.8	4.7	6.5
U-2-O	1.4	5.3	6.7
O-1	2.4	15.7	18.1
O-2	1.2	13.2	14.4
N	3.0	7.4	10.4
A	0.1	(0.2 gain)	(0.1 gain)

COMPRESSIVE STRENGTH

Three test samples of approximately equal size were sawed from each concrete block. Measurements of the samples were taken and recorded prior to being subjected to compressive force. The average result of three test specimens from each block was considered to be representative of the strength of the block. Table 2 gives the data on compressive strength of specimens 1" x 1" x 2".

There is no distinguishable pattern to the results, but there are indications that compressive strength varies considerably from one block to another.

TABLE 2

Compressive Strength Of Concrete Block Samples

	Average P.S.I.
U-1	3169
U-2	2264
O-1	2716
O-2	2389
N	2947

DENSITY

Densities were determined by weighing in air and in water. The apparent densities as received, and those calculated for the dried and ignited samples are given in Table 3. Although there is a wide variation in the samples as received, there is no significant difference in density after ignition. The overall low density results most likely are due to manufacturing procedures and the use of light weight aggregate.

TABLE 3

Density of Concrete Block Samples

Sample	Density as received lbs/ft^3	Density after drying lbs/ft^3	Density after ignition lbs/ft^3
U-1*	107 (106.8)	105	100
U-2*	107 (107.2)	105	100
O-1	126 (126.5)	124	103
O-2	118 (117.6)	116	101
N	114 (113.9)	110	102

*These represent an average of inner, center, and outer samples.

ABSORPTION

Water absorption of the blocks was obtained in accordance with AASHTO Designation T33-72, Section 6. As in previous tests, all specimens were of relatively equal size.

The wide range of results, as illustrated in Table 4 below, lends no clue to the cause of failure of materials in the concrete blocks.

TABLE 4

Water Absorption of Concrete Block Samples

	% BY WEIGHT
U-1	14.1
U-2	13.1
O-1	9.3
0.2	13.0
N	16.0

CHEMICAL ANALYSIS

The samples were decomposed by fusion with sodium carbonate (Na_2CO_3) at 1000° C. After cooling, the melt was treated with three normal hydrochloric acid (3N HCL) until decomposition was complete, after which the silica (SiO_2) residue was filtered off, washed with hot .1N HCl and hot H_2O and set aside. The filtrate and washings were evaporated to dryness and baked for 1 hour at 105° C. The residue was treated with 15 milliliters (ml) 3N HCl, followed 10 minutes later by 35 ml. hot H_2O, after which the filtration was repeated.

The combined SiO_2 residues were ignited to constant weight in a platinum (Pt.) crucible at 1050° C. One ml. of 9N sulfuric acid (H_2SO_4) and 20 ml. concentrated hydrofluoric acid (conc HF) were added, after which the contents of the crucible were evaporated to dryness and ignited to constant weight at 1050° C. The difference in weights is the weight of SiO_2 obtained.

A separate sample was taken for the remainder of the analyses. The SiO_2 was separated out as before, the filtrates being caught in a 500 ml. volumetric flask. The residue in the crucible was fused with 0.5 gram (gm.) potassium pyrosulfate ($K_2S_2O_7$) at 500° C, cooled and treated with water to dissolve the melt. This was added to the contents of the volumetric flask, after which the combined filtrates were diluted to 500.0 ml.

Analyses for Ca, Mg, Mn, Fe, and Al were performed on the combined filtrates by the Regulatory Laboratory of the University of Vermont using atomic absorption spectroscopy.

Results for SiO_2 are the average of two samples. Because of the lengthy preparation procedure, the results for the other elements are based on a single sample. The SiO_2 results serve to point out the inhomogeneity of the samples.

Normal precision for a silica analysis by the fusion procedure is 0.2%. The precision of the samples under consideration varied from 2 to 14% which represented up to 20% of the actual values for SiO_2 . Because of the apparent inhomogeneity in the samples, the results of the chemical analysis cannot be conclusive. The analysis for sulfur trioxide (SO_3) was at the limits of detectability and inconclusive. This analysis should be repeated with larger, more homogeneous samples.

Results of the chemical analysis for the samples as received are presented in Table 5. Table 6 gives analytical results based on dried samples (105^{o} C), while the data in Table 7 is based on ignited samples (950^{o} C). Care should be taken in drawing conclusions from Table 7 unless a separate carbonate analysis is undertaken.

Table 8 gives the "proximate analysis" of each sample, with each substance being reported as the oxide. This is standard procedure in many mineral analyses and is included here for completeness. Because of the scanty data, SO_3 is eliminated from the summation. It would have resulted in only a minor correction.

The only datum of possible significance is the elemental analysis of U-1-I. This sample was badly stained with creosote. Analysis shows a much higher amount of SiO_2 and much lower amounts of Mg and Ca than in the remainder of the block or in any of the other samples.

The results could be due to inhomogeneity of the sample. If they are not, one must look for a mechanism by which hot, acidic creosote could attack and deplete the calcium and magnesium content of the block. If this is true, then the creosote would attack the cement portion of the block, causing crumbling.

The sample from the inner portion of the used block, U-2-I, has no creosote staining, and its chemical analysis is similar to that of all the others. Further tests with completely homogeneous samples, whose history of creosote exposure has been more carefully documented, are needed before definitive conclusions can be drawn.

TABLE 5

Chemical Analysis - As Received

	SiO_2	Mg	Ca	Mn	Fe	Al
U-1-I	75.6	0.92	1.32	.051	3.53	5.88
U-1-C	58.8	1.27	5.35	.065	3.95	6.32
U-1-O	59.4	1.22	5.37	.064	3.51	6.27
U-2-I	54.6	1.16	5.71	.062	3.46	5.71
U-2-C	68.6	1.21	5.86	.065	3.42	5.73
U-2-O	54.4	1.25	5.96	.061	3.51	5.61
O-1	44.5	3.72	9.99	.065	2.61	3.76
O-2	50.8	3.10	8.77	.057	3.14	4.77
N	55.0	2.16	7.21	.063	3.49	5.48
A	55.4	1.86	1.28	.103	5.30	8.48

TABLE 6

Chemical Analysis - Based on Dried Sample

	SiO_2	Mg	Ca	Mn	Fe	Al
U-1-I	76.9	0.94	1.34	.052	3.59	5.98
U-1-C	60.2	1.30	5.48	.066	4.04	6.47
U-1-O	60.3	1.24	5.45	.065	3.57	6.37
U-2-I	55.7	1.18	5.82	.063	3.53	5.82
U-2-C	69.9	1.23	5.97	.066	3.48	5.84
U-2-O	55.2	1.27	6.04	.062	3.56	5.69
O-1	45.6	3.81	10.23	.067	2.67	3.85
O-2	51.4	3.14	8.88	.058	3.18	4.83
N	56.7	2.23	7.43	.065	3.60	5.65
A	55.4	1.86	1.28	.103	5.30	8.49

TABLE 7

Chemical Analysis - Based on Ignited Sample

	SiO_2	Mg	Ca	Mn	Fe	Al
U-1-I	79.7	0.97	1.39	.054	3.72	6.20
U-1-C	63.0	1.30	5.74	.068	3.75	6.70
U-1-O	63.5	1.30	5.74	.068	3.75	6.70
U-2-I	58.8	1.25	6.15	.067	3.73	6.15
U-2-C	73.4	1.30	6.27	.070	3.66	6.13
U-2-O	58.3	1.34	6.39	.065	3.76	6.01
O-1*	54.8	4.58	12.29	.080	3.21	5.87
O-2*	59.3	3.62	10.25	.066	3.67	5.58
N	61.4	2.41	8.04	.070	3.90	6.12

A - Data not valid due to slight weight gain on ignition

* Data may not be valid due to possibility of limestone aggregates

TABLE 8

Proximate Analysis

	Σ	SiO_2	Total	Total Ign. loss	Total
U-1-I	19.5	75.6	95.1	5.2	100.3
U-1-C	27.2	58.8	86.0	6.7	92.7
U-1-O	26.4	59.5	85.9	6.5	92.4
U-2-I	25.7	54.6	80.3	7.1	87.4
U-2-C	25.9	68.6	94.7	6.6	101.3
U-2-O	26.1	54.4	80.5	6.7	87.2
O-1	31.0	44.5	75.5	18.7	94.2
O-2	31.0	50.8	81.8	14.4	96.2
N	29.0	55.0	84.0	10.4	94.4
A	28.5	56.4	84.9	(.1 gain)	84.8

Σ is sum of MgO, CaO, Fe_2O_3, Al_2O_3

CREOSOTE EXPOSURE

A creosote solution was prepared by extraction with boiling water, using three fresh 300 gm. portions of creosote obtained from chimney cleaning for every 500 ml. of water. Preweighed specimens of each of the concrete blocks were boiled, covered, in the creosote solution for ten days, the solution being replenished as needed, and allowed to cool to room temperature overnight. The specimens were rinsed in hot water, dried, and examined.

There was no effect other than a slight discoloration of the surface. This was not surprising, as the conditions in a hot chimney (temperatures as high as 400° C) are far more severe than those obtainable in a boiling water bath. It is also reasonable to assume that the creosote recovered when a chimney is cleaned is less active chemically than that deposited while wood is being burned, as the more active components of the creosote have had a chance to react and to be exhausted by the time a heating season has passed and the chimney is cleaned.

HEAT TREATMENT

One specimen of each of the five blocks (U-1, U-2, O-1, O-2 and N) was selected. The specimens from the used blocks were taken from the outer portion of the block so as to negate any effect of possible creosote exposure of the innermost portions. The specimens were heated to 450°C every workday for six weeks, for 7½ hours each day, being allowed to cool every night and on weekends.

They were examined every Monday morning, with no change in appearance being found. Weight losses were consistant with, and somewhat lower than, previously determined losses on ignition, as complete release of chemically bonded water requires much higher temperatures. The specimens were then heated to 950°C every day for five days. There was still no visible change in appearance. They were removed from the furnace and allowed to stand for one week in the hot and humid laboratory of a typical mid-August. At the end of this time, two of the samples, O-1 and O-2, were reduced to rubble. The remaining three were cracked and had lost all their strength.

These results, together with the results of loss on ignition, would seem to point to the uptake and loss of water on standing and heat treatment as a possible failure mechanism for the blocks. The old blocks have had a chance to take up and chemically bind large amounts of water. Loss of all of this water would possibly tend to shrink or weaken the crystal lattice. Taking water up rapidly on standing could possibly re-expand the lattice, causing failure. The new block has not had enough time to take up water, and the used blocks have been heat treated periodically, which would explain why these blocks withstood heat treatment better that the old, but unused, blocks.

It is obvious that further work is needed in this area. Compressive strength tests in addition to visual observation and weighing should be done as a function of both length and temperature of treatment.

CONCLUSIONS AND RECOMMENDATIONS

The analyses done were not able to detect a difference between used blocks that had failed, new blocks and old unused blocks. The results obtained to date would indicate two possible failure mechanisms for the blocks. The first is chemical attack of the cement portion by hot creosote. The second is the loss and gain of chemically bound water by alternately heating to high temperature and cooling. This could cause alternate shrinkage and swelling of the crystal lattice. The enormous physical stresses resulting can lead to very rapid failure.

From the data obtained, it was impossible to determine whether concrete blocks are suitable or can be made suitable for the construction of chimneys. It was also not possible to determine why some chimneys have longer life than others.

Perhaps the most important conclusion is that this is a preliminary study whose major significance is in pointing the way toward further work. This work should include heat treatment with compressive strength tests as a function of time and temperature and creosote exposure under conditions approaching those obtained in a hot chimney.

The history of each sample needs to be thoroughly documented and the samples should be completely pulverized and homogeneous. The analysis for SO_3 should be attempted with larger samples, as SO_3 is often an indication of cement content. Finally, chemical analysis should be done at least in duplicate in order to be able to place more credence in the results.

APPENDIX
LIST OF CHEMICAL TERMS

Al	Aluminum
Ca	Calcium
Fe	Iron
HCl	Hydrochloric Acid
HF	Hydrofluoric Acid
H_2SO_4	Sulfuric Acid
$K_2S_2O_7$	Potassium Pyrosulfate
Mg	Magnesium
Mn	Manganese
Na_2CO_3	Sodium Carbonate
Pt	Platinum
SiO_2	Silicon dioxide (Silica)
SO_3	Sulfur trioxide
N	Normal, a measure of concentration

HD 565 3/78

Prepared By: D. Brown
Date: 10/18/78
Sheet 1 of 1

STATE OF VERMONT
AGENCY OF TRANSPORTATION
MATERIALS & RESEARCH DIVISION

RESEARCH INVESTIGATION

Work Plan No. 78-CT-40

Subject Concrete Chimney Blocks

Investigation Requested By Consumer Protection Division of Attorney General's Office Date September 28, 1978

Date Information Required As soon as possible

Purpose of Investigation To try and determine the cause of early rapid deterioration of certain concrete chimney blocks

Proposed Tests or Evaluation Procedure All tests will be performed on: 2 deteriorated blocks from actual chimneys, 2 unused blocks over 2 years old, 1 new block recently made

Physical Tests	Chemical Tests	
1. Unit wt. of all blocks	to be run on blocks & aggregate	
2. Water absorption	Silico	Magnesium
3. Effect of freezing & thawing	Iron	Sulfar Trioxide
4. Effect of high temp. on compressive strength	Aluminum	
5. Effect of creosote saturation on comp. strength	Calcium	
	Loss on ignition	

Proposal Discussed With D. Brown, R. Fraser, B. Noble, P. Corti

Projected Manpower Requirements 1 Tech, 1 Chemist

Investigation To Be Conducted By P. Corti, B. Noble

Proposed Starting Date October 5, 1978 Estimated Completion Date A.S.A.P.

Approval/Disapproval by Materials Engineer R. F. Nicholson 10/19/78

Comments by Materials Engineer

Materials Division
Agency of Transportation