Bloating Properties of Shale in the Hilltop Formation in Seminole County

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The manufacture of lightweight aggregate for concrete from clays and shales has become within the past few years an important industry with large capital investment. About two years ago the Oklahoma Geological Survey started an investigation of certain Oklahoma shales of Pennsylvanian age to determine their suitability as raw material for such use. The work was completed and the results published as Mineral Report No. 24. Since that time shale from another formation, the Hilltop, and from the quarry of the Wewoka Brick & Tile Co., in the southwest quarter of section 11, Township 8 N., Range 7 E., in Seminole County has been tested in a manner similar to that used and reported in Mineral Report No. 24 but with slight modifications.

The Hilltop formation in Seminole County was the subject of a geologic investigation by William F. Tanner. His findings were published in the Bulletin of the American Association of Petroleum Geologists.[2] A measured section in the Wewoka Brick & Tile Co. quarry is described. The material used in the tests of the bloating properties was "run-of-mine" stone supplied by that company. The lump shale was run through a jaw crusher, reducing the stone to minus ½ inch sieve*. It was then screend for size and the material that passed the ¼ inch sieve but was retained on the No. 6, being somewhat less than pea size, was selected for the trials. A portion was pulverized for chemical analysis. The analysis shows the material to contain:

[1] Industrial Chemist, Oklahoma Geological Survey. Publication authorized by the Director.

* ½ inch and ¼ inch sieves, U. S. Standard, Coarse Series. No. 6 sieve, U. S. Standard, Fine Series.

CHEMICAL ANALYSIS OF SHALE IN HILLTOP FORMATION

Seminole County

	—%				
SiO_2	62.25	R_2O_3	24.89	K_2O	2.45
Al_2O_3	16.64	CaO	0.85	Na_2O	0.39
Fe_2O_3	5.31	MgO	2.23	ZrO_2	0.07
FeO	1.52	MnO_2	0.05	V_2O_5	0.30
TiO_2	0.85	SO_3	0.03	BaO	None detected
H_2O at 110°C.	2.03	P_2O_5	0.03	L.O.I. above 110°C.	5.65

TOTAL OXIDES—100.65

TESTING PROCEDURE

A portion sufficient to nearly fill was placed in each of seven fire-clay crucibles (Denver Fireclay Co. 100 ml. capacity). All seven crucibles were then placed in an oven and dried at 110°C. for 24 hours. One crucible was put into an electrically-heated furnace and the temperature raised gradually to 1150°C. and maintained at this temperature for one and one-half hours, then removed and allowed to cool. Two crucibles received no further treatment. Two others were subjected to a temperature of 400°C. for one hour. The remaining two were heated at 600°C. for one hour. After cooling in a desiccator the loss in weight was determined.

Loss from room temperature to 110°C.	- 1.93%
Loss between 110° and 400°C.	- 2.02%
Loss between 400°and 600°C.	- 2.61%
Loss between 600° and 1150° C.	- 1.02%

The loss is principally water combined in the clay minerals. However, probably some organic matter was destroyed as well as some oxygen absorbed. The change from ferrous iron to ferric iron could account for 0.17% gain in weight.

The bloating characteristics of the shale were determined as follows: Three crucibles,—one that had been dried at 110°C. only, one that had also been heated to 400°C., and one that had been heated at 600°C.—were introduced quickly into a furnace in which a temperature of 1150°C. had already been attained. The temperature in the furnace dropped approximately 120°C. but regained the loss within 10 minutes. An additional 15 minutes heat at 1150°C. was maintained, then the crucibles were withdrawn as quickly as possible, and allowed to cool.

Another series of three crucibles, similar in all respects to the previous series, were quickly introduced into a furnace in which a temperature of 1210°C. had already been attained. The temperature dropped approximately 120° and regained 1210° in 10 minutes. After holding at this temperature for an additional 15 minutes, the crucibles were quickly removed and allowed to cool.

The products from all seven crucibles were examined and tested to determine density, absorption, and porosity. The true absorption values were found by placing a weighed amount of the product in a wire-screen cage, submerging the cage and contents in water, bringing to a boil, maintaining at a boil for 1 hour, allowing to cool submerged, draining thoroughly, and

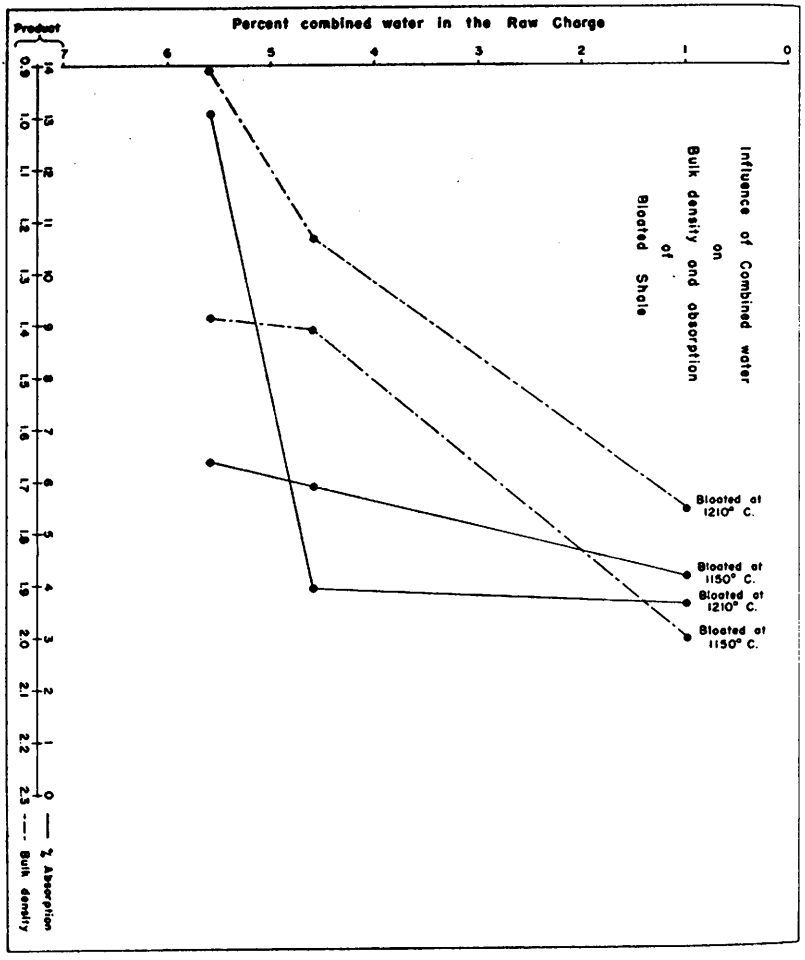

Figure 1. Influence of combined water on bulk density and absorption of bloated shale.

TABLE I.

BLOATING PROPERTIES OF SHALE IN HILLTOP FORMATION, SEMINOLE COUNTY, OKLAHOMA

Test No.	Preliminary Treatment	Bloating Temperature (Fired in Clay Crucibles) Except VIII	Lbs. Per Cu. Yd. Bulk	Density Apparent Bulk		Absorption % By Wt. *	Absorption % By Wt. #	Porosity % by Vol.
I	Dried at 110°C.	1150°C. Temp. Dropped to 1030° Back to 1150° in 10 Min. Hold for 15 Min.	1300	1.52	1.38	2.6	6.3	9.6
II	Dried at 110°C.	1210°C. Temp. Dropped to 1090° Back to 1210° in 10 Min. Hold for 15 Min.	780	1.04	0.91	7.1	13.1	13.6
III	Dried at 110°C. Pre-heated at 400°C. for 1 Hr.	1150°C. Temp. Dropped to 1030° Back to 1150° in 10 Min. Hold for 15 Min.	1300	1.52	1.41	2.5	5.4	8.2
IV	Dried at 110°C. Pre-heated at 400°C. For 1 Hr.	1210°C. Temp. Dropped to 1090° Back to 1210° in 10 Min. Hold for 15 Min.	1260	1.29	1.23	2.0	3.9	5.1
V	Dried at 110°C. Pre-heated at 600°C. for 1 Hr.	1150°C. Temp. Dropped to 1030° Back to 1150° in 10 Min. Hold for 15 Min.	1870	2.24	2.05	3.6	4.2	9.3
VI	Dried at 110°C. Pre-heated at 600°C. for 1 Hr.	1210°C. Temp. Dropped to 1090° Back to 1210° in 10 Min. Hold for 15 Min.	1620	1.88	1.76	0.6	3.6	6.8
VII	Dried at 110°C.	Temp. Raised Gradually From Room Temp. to 1150°C. Hold for 1½ Hrs.	1870	2.17	2.15	--	0.3	0.6
VIII	Dried at 110°C.	Bloated on Open Hearth. In at 1230°C. Temp. Dropped To 1150°. Back to 1160°C. in 5 Min. Remove from Furnace.	600	--	0.8	4.4	19.3	15.3

* Cold Water Immersion for 1 Hr.
# Immersed, Brought to Boiling, Hold 1 Hr. Cooled Submerged.

weighing. The absorption obtained by immersing in cold water for 1 hour was also found. The water displaced by the thoroughly saturated granules was measured and the weight of the granules in water was taken. From the information obtained, calculations were made of bulk density, apparent density, absorption, and porosity. The results are given in Table 1, and shown graphically in Figure 1.

CONCLUSIONS

It is evident that increase in the temperature of pre-heating decreases the bloat; which is to say that the lower the amount of combined water in the raw material the less gas is formed and consequently a smaller bloat is obtained, other factors being constant. The only apparent advantage to be derived from pre-heating lies in the greater strength of the product. Even in the cases where the greater strength is desired, it can be best obtained by pre-heating at 400°C. or less

Bloating at the higher temperature where the mass is more thermoplastic makes better use of the available water for expansion, but the product of most clays will be more fragile.

Examination of the products shows occasional granules of light gray color which are identified as pieces of partially calcined limestone. These particles are highly argillaceous and contain considerable unaltered carbonate. Tanner had reported the presence of limestone nodules and thin layers in his publication on the Hilltop formation. What influence, if any, these limey particles would have on concrete, either before or after the "set", should be investigated.

REVIEW

From the results in these tests it appears possible that a superior product might be obtained by using a bloating temperature slightly in excess of 1150°C. but with a shorter exposure than 15 minutes. Therefore, a trial was made using oven-dried granules, but introducing the raw material onto an open hearth rather than a crucible. This procedure should yield results more nearly comparable with those which would be obtained if the processing were done in a rotary kiln.

The dried granules were quickly introduced into the furnace wherein a temperature of 1230°C. had been attained. The temperature dropped to 1150° but in 5 minutes time it was at 1160°C., whereupon the product was quickly withdrawn from the furnace and allowed to cool. The expanded granules were olive-drab in color. The exterior of the granules was smooth and there had been only slight adhesion of particle to particle. The interior was cellular. The properties of this product are shown as Test No. VIII in Table 1.

Other than the presence of a small amount of limestone, which might be eliminated by selective quarrying, the Hilltop shale from this particular location appears to offer commercial possibilities as a source of raw material for the manufacture of lightweight aggregate.

LITERATURE CITED

1. Burwell, A. L. 1954. Lightweight Aggregate from Certain Oklahoma Shales. Mineral Report No. 24. Oklahoma Geological Survey.

2. Tanner, William F. 1953. Hilltop Formation, Upper Pennsylvanian. Seminole County, Oklahoma. Bulletin of the American Association of Petroleum Geologists. Vol. 37, No. 8 pp. 2046-2055.