

OKLAHOMA GEOLOGICAL SURVEY

CARL C. BRANSON, Director

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Lightweight Aggregate
From Certain Oklahoma Shales

by

A. L. BURWELL, Chemist

NORMAN

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from

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by

Albert L. Burwell

Industrial Chemist, Oklahoma Geological Survey

(ABSTRACT)

The increasing demand for lightweight mineral aggregate for use in concrete has led in recent years to the development of several different processes for its production. A wide diversity of raw materials have been tested throughout this country resulting in products equally diverse in appearance, characteristics, and properties. In Oklahoma, no extensive research has been undertaken. The sole commercial installation in the state utilizes an alluvial soil rather than consolidated clay or shale.

Preliminary trials on Oklahoma clays and shales indicate that those of Pennsylvanian age are more likely to respond to the application of high temperatures to yield satisfactory lightweight products, although some material of Cretaceous and Tertiary age show promise. Material of Permian age is less promising.

Seven samples representative of five geologic formations of Pennsylvanian age from northeastern Oklahoma have been tested in laboratory equipment to learn something of their reactions and results when subjected to elevated temperatures. Description is given of the raw materials, the locations from which they were obtained, and their chemical analyses. The density, porosity, and absorption of the products are recorded and the influence of drying and pre-heating are shown. Also, attention is called to changes in the product due to variation in temperature and the duration.

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FOREWORD

Bloating of Clays and Shales

Clays and shales are not minerals of simple and uniform composition. Rather, they are extremely complex and differ widely. Ladoo and Myers (1951) explain that "clay deposits are of two general types; residual and sedimentary; in all cases the clay is of secondary origin, that is, it was formed by the alteration of some other rock."¹ Thus, it is understandable that clay deposits are seldom composed of pure clay minerals. This is especially true of shales, which are in reality bedded clays that have been buried by subsequent deposits of mineral debris and have been more or less consolidated by pressure of the overlying materials. The impurities present in a shale exert a pronounced effect when the shale is processed in an attempt to utilize it.

Clay minerals are considered generally as hydrous aluminum silicates. The clay minerals, kaolinite and halloysite, conform to this description and, if pure, do not contain elements other than silicon, aluminum, oxygen, and hydrogen. Clay minerals which belong to the montmorillinite and illite groups contain in the molecular structure other elements such as sodium, potassium, calcium, magnesium, and iron. Thus, it is plain that the clay minerals themselves may contain elements that will influence their reaction during processing. Further, the extraneous impurities may be portions of the original rock from which Nature made the clay minerals, or it may be material accumulated during transportation from place to place, or it may have been brought into the clay or shale bed by circulating waters.

The bloating properties and characteristics of clays and shales have been studied by numerous persons. It is generally accepted that the ability of a clay or shale to expand or bloat depends upon two factors principally. First, the clay or shale must change from a solid to a thermoplastic state, and this state should exist over a considerable temperature range. Second, a gas or gases must be generated from the clay minerals or the impurities while the mass is in the thermoplastic state. The fact that the mass is plastic enables it to retain the gases formed within itself. Should the viscosity drop and the mass become fluid the gases will escape, and on cooling the mass will shrink to a glassy solid. A summary of research on the subject has been published by Riley (1951).²

The chemical composition of a clay or shale will determine to a great extent, but not entirely, its action when subjected to rise in temperature. The rate at which the temperature is raised as well as the temperature attained and the duration at that temperature are influential. Clay or shale composed largely of kaolinite or halloysite clay minerals must depend upon their molecularly-combined water to yield the gas to bring about an expansion, but the fact that such minerals attain a thermoplastic state only at relatively high temper-

atures mitigate against their utilization. A characteristic of clay minerals of the montmorillinite group is their tendency to melt rather than to become thermoplastic. Even when a plastic state is obtained the temperature range within which it exists is short.

Clays and shales containing clay minerals of the illite group usually do not have a sharp melting point. Most of them when subjected to increasing temperature gradually soften, the mass becoming plastic and viscous. They are more likely to contain in addition to combined water other gas-forming ingredients such as carbonates, sulfides, sulfates, iron, and carbonaceous matter, some of which may exert considerable effect on the temperature at which the mass becomes plastic and upon the degree of viscosity. However, it should be noted that illite-group clays and shales are more prone to weather and to oxidize than some other groups, and that such alterations in many cases lessen their ability to bloat satisfactorily.

The term mineral aggregate has been broadly defined as "an aggregation of sand, gravel, crushed stone, slag, or other material of mineral composition, used in combination with a binding medium to form bituminous and Portland-cement concrete, macadam, mastic, mortar, plaster, etc., or alone as railroad ballast, filter beds and various manufacturing processes, such as fluxing, etc."³ (Woods, 1948, p.1.) Concrete in which the binding medium is a natural cement, synthetic resins, and synthetic rubbers should be included. It is recognized that sand, gravel, and crushed stone are still the principal mineral aggregates and that just so long as such dense material is used the concrete made from them will be high in weight per unit volume. It is only in recent years that the advantages of certain artificial lightweight mineral aggregates have received attention. The first comprehensive report on the subject, including also natural lightweight mineral materials, was published in 1940.⁴ (Foster, pp. 468-73.)

At first, the natural materials such as pumice and scoria were the only ones available. Then cinders and coke came into use in a few places. Next, it was only a normal development for the production and marketing of more suitable products, "either by-products of some industrial operation or specially manufactured for use as aggregate.... The special manufacture of lightweight aggregate almost invariably involves the processing of material to produce a vesicular, porous, or exfoliated structure, or to increase the toughness and strength. Materials thus processed include clay, shale, slate, blast-furnace slag, diatomaceous shale, fly-ash, pumice, obsidian, perlite, and vermiculite."⁵ (Davis and Kelly, 1948, pp. 160-1.) The advantages derived from the use of lightweight material are much lighter weight per unit volume of concrete and the greater thermal and acoustical insulating properties of the composition. Probably the greatest advantage comes from the savings in structural and reinforcing steel which is possible due to the lesser weight of the concrete.

Two distinct types of lightweight aggregate are being made from shales and other clay-bearing materials. One type has been designated as *coated aggregate* and is usually made by rapidly firing prepared material in a rotary kiln. The second type is designated as *sinter aggregate*, which may be made also in a rotary kiln but more often is made in a sintering machine. Under ideal conditions the coated aggregate comes from the kiln as smooth, rounded particles the surface of which is vitreous and glassy and the interior cellular. The sinter aggregate comes from the kiln or machine as a porous clinker which requires crushing before use. Aggregate can be made with the sintering machine from a greater variety of raw materials than can be made with the rotary kiln, but the products are definitely different.⁶ (Wilson, 1953, p.iii.) The properties desirable in a lightweight aggregate have been stated as follows:

(a) *High Strength to Weight Ratio*

The product must be light to effect a worthwhile saving in weight at the same time be strong enough to meet strength specifications. Variations in both the strength and weight may be effected by modifying the processing technique. As the strength of a product is increased, the weight is also increased and vice versa. Because of their strong shell and spherical nature, coated lightweight aggregate particles tend to give a product with a higher strength to weight ratio than other types. The actual maximum weights specified for a properly graded aggregate under A.S.T.M. specifications, designated C 130-42 are 76 lb./cu.ft. for the fine aggregate and 55 lb./cu.ft. for the coarse aggregate.

(b) *Good Workability*

The smooth spherical nature of coated clay and shale aggregate particles promotes good workability whereas aggregate particles with sharp corners make a harsh concrete mix that is difficult to work around reinforcing bars, tending to give a honeycomb structure.

(c) *Low Absorption*

An aggregate with a high water absorption, unless it has been pre-soaked, tends to dehydrate the cement which has a harmful effect on the setting of the concrete. Coated aggregates generally have a lower absorption than other aggregates because of the coating covering the cellular interior.

(d) *Uniform Size Gradation*

The product must be composed of aggregate particles of a range of sizes with sufficient fine material to permit working the mix around forms and reinforcing bars. The grading prescribed may be referred to under A.S.T.M. specifications, designation C 130-42.

(e) *Chemical Inertness*

A lightweight aggregate should not contain chemical impurities which might react with the cement or reinforcing bars with deleterious effects.⁷ (Matthews, 1953, pp. 2-3.)

EXPERIMENTAL WORK AT OKLAHOMA GEOLOGICAL SURVEY

Investigation of the expanding and bloating properties of clays and shales at the Oklahoma Geological Survey has been confined mainly to laboratory tests made in assisting interested persons in determining the feasibility of commercial installations for the manufacture of lightweight aggregate. As a result of these tests it has been fairly well established that most of the shales in Oklahoma of Pennsyl-

vian age will bloat to a greater or lesser degree, and that some shales and clays of Cretaceous and Tertiary age will do likewise, but that nothing much can be expected from most clays and shales of Permian age. There may be exceptions that have not been encountered.

This report deals with the results of tests made on certain Pennsylvanian shales taken from five locations in northeastern Oklahoma. At two locations two separate samples were taken representing different portions of the exposures. Four geologic formations are represented in the seven samples. The samples were collected by John H. Warren, assistant geologist on the Survey staff. The locations from which the samples were taken are described by section, township, and range, by county, by name of the geologic formation, and by particulars regarding the deposits and conditions in Table I.

The samples were taken by the "channel" method and therefore represent the entire face of the exposure. As usual, these shales have a laminated structure and individual layers may and in many cases do show considerable difference in composition. It will be understood that the chemical analyses indicate the average composition. These analyses were made in the Survey laboratory, most of them by Thomas E. Hamm, analyst, and are given in Table II.

The predominant clay mineral in all samples appear to belong to the illite-group, although it is probable that other types of clay minerals occur in admixture along with miscellaneous extraneous matter such as might be expected in the mud of an ancient sea-bottom.

EXPERIMENTAL

The samples as received were air-dried, then crushed in a jaw crusher to minus 1 inch size. An aliquot of the crushed material was ground in a disc grinder to pass a 40 mesh sieve. A portion of the ground material was reserved for chemical analyses and the balance for experimentation.

(a) Series

The first series of experiments was prepared for by mixing pulverized shale with 30 per cent its weight of water. After thoroughly incorporating the water, the resulting paste was placed in a bar mold of dimensions 2.5 x 2.5 x 36 cms., and made with a false bottom to facilitate removal of the casting. The casting was cut into approximately 5 cm. lengths and air dried, followed by oven drying at 230°F.

Four separate firing tests were run. First, a full set of specimens was placed in an electric furnace with temperature at 230°F. and the temperature was gradually raised over a five-hour period to 1830°F. The specimens were then removed from the furnace and submerged in clean sand until cool. Second: a full set of specimens which had been kept in the oven at 230 F. were quickly removed and placed in an electric furnace which had attained a temperature of

1900°F. This temperature was maintained for 15 minutes. The specimens were then removed from the furnace and submerged in sand to cool, as before. Third: the second run was repeated except that the attained temperature in the furnace was 2000°F. Fourth: this run was a repetition of the second and third except that the attained temperature in the furnace was 2100°F.

The results of treatment in this series are recorded in Figure 1 wherein it will be observed that in comparison with untreated specimens those slowly heated to 1830°F. show considerable shrinkage, whereas those subjected suddenly to high temperatures expanded, the higher the temperature the greater the expansion. It will be observed also that at 2100°F. the specimens became more glassy than at lower temperatures, and in one case at least, the specimen has been close to fluidity, resulting in a product less desirable than had a viscous thermoplastic existed. The bulk density of the specimens, untreated and treated, together with the degree of shrinkage or expansion of those exposed to the heat treatment is shown in Table III. Starting with bulk density in the range from 1.76 to 1.90, the prolonged, slowly increased temperature caused an increase in density to 2.15 in six specimens and to 2.45 in one, and a corresponding shrinkage in volume. The application of sudden high temperature to the specimens yields considerably different products. An appreciable difference in reaction is shown by two portions of the same formation in the Nellie Bly, whereas two portions of the Senora formation above the Henryetta coal differ but little. The results obtained in this series seems to indicate that all seven samples may be successfully bloated if the optimum operating conditions can be determined and adhered to.

(b) Series

From the standpoint of economics to salvage the heat from a bloating-furnace by means of a heat-exchanger and to use this recovered heat in pre-heating the furnace charge seem reasonable and advisable. The State Geological Survey of Kansas had the advantage of a laboratory-size rotary kiln in which to make bloating tests, - equipment of a kind not available at the Oklahoma Survey, and they pre-heat the shale to 1100°F. but do not state the duration of the pre-heating.⁸ (Plummer and Hladik, 1951.) Since it seems probable that the length of time that the shale is exposed to the pre-heating will influence the results of the subsequent bloating, it was decided to pre-heat for 3 hours on the assumption that any change likely at that temperature would have been completed in that time. The full procedure on this series was as follows: The crushed shale was screened and the particles 10 mesh to 1 inch in size were used. Fire-clay crucibles of 100 ml. capacity were filled approximately three quarters full and placed in the drying oven over night at 230°F. The crucibles were moved to an electric furnace in which a temperature of 1100°F. was maintained. After 3 hours at 1100°F., the crucibles were quickly transferred to another electric furnace heated in ad-

vance to a pre-determined temperature for bloating. The temperatures used were 2100, 2150, 2200, and 2300°F. In all cases, when the crucibles were introduced into the bloating furnace the temperature dropped slightly. The time required to re-gain the temperature loss was noted, then the additional time thought desirable was decided upon, and the treatment continued. At the expiration of the firing schedule the crucibles were quickly removed from the furnace and covered with sand until cool. The products were examined and the apparent density, average porosity, and average absorption were determined.

Method used to determine apparent density, porosity, and absorption: The bloated material was weighed dry. Then the material was placed in a wire-screen cage and the cage submerged in water in a beaker. The water was brought to a boil and held at a boil for 30 minutes. The material was allowed to cool submerged in the water and then drained on absorbent paper, and weighed wet. The volume of the dry material was determined approximately by finding the volume of water displaced by the saturated wet material.

Apparent density: dry wt./volume
Apparent porosity: wet wt. minus dry wt./volume
Apparent absorption: wet wt. minus dry wt./dry wt.

The record of the treatments and the results are given in Table IV.

(c) Series

In this series, only one shale was used. Bloating was carried out at 2000, 2100, 2200, and 2300°F. and two crucibles were used at each temperature, one of which contained shale dried at 230°F. for 18 hours and the other pre-heated for 3 hours at 1100°F. in addition to the 230°F. for 18 hours. The duration of the exposure to the bloating temperature was the same in all cases, - 10 minutes to return to temperature and 15 minutes additional. The products were cooled under sand as previously, then examined, and the density, porosity, and absorption determined. Results are shown in Table V. The effect of pre-heating is noted in the reduction in expansion, and the effect of temperature increase on the bloating appears less on pre-heated material.

(d) Series

In this series, only one shale was used. Bloating was carried out at 2100°F. using 4 crucibles. The shale in two crucibles had been dried at 230°F. for 18 hours whereas the other 2 crucibles had been pre-heated at 1100°F. for 3 hours in addition to the 18 hour drying. One of each lot was treated at 2100°F. for 30 minutes and the other two at 2100°F. for 15 minutes. On removal from the furnace, the products were cooled under sand, and the density, porosity, and absorption determined. Results are shown in Table VI. Again pre-heating appears to reduce the ability to expand. The increase in exposure time lowers the porosity and absorption, indicating the extra time gave a less permeable coating.

(e) Series

In this series, all seven samples were tested. The samples were placed in the crucibles and dried at 230°F. for 18 hours. In the first run, one crucible of each sample was rapidly transferred to the furnace, at 2100°F., 15 minutes was required to re-gain the temperature, then 10 minutes additional given before removal of the crucibles from the furnace and cooling. In the second run, all factors were as in the first run except the bloating furnace was maintained at 2200°F. The products were examined and the average density, porosity, and absorption determined. Furthermore, individual pieces were selected from each crucible, each piece representing a variation in the product. The apparent densities of these individual pieces are shown for comparison with the average values in Table VII.

OBSERVATIONS

In examining the bloated products it was noted that certain particles differ from the others in the same batch. There were differences in the degree of bloating, in the vitrification, in the cellular structure, and in the color. Evidently some portion of the sample had been more susceptible to the high temperature than others. This condition was observed to some degree in all samples but it was especially noticeable in the products made from shales of the Senora formation. On re-examination of the crushed raw shale from this formation it was seen that a small percentage of the particles were light brown in color and easily distinguishable from the drab gray of the majority. Some of this light brown material was picked out and a chemical analysis made for comparison with the average. As a check on the results specimens of bloated material from the same formation which differ in appearance from the general run were analysed also. The results are given in Table II. Attention is called to the relatively high iron content in both cases, and to recognized influence of iron on the melting point and color of glasses.

The question of the proper temperature and duration of pre-heating should be given further study. In any process where shale is subjected suddenly to a high temperature it seems probable that combined water in the clay minerals is the most prominent and perhaps the sole source of gas to produce the bloating. Thermal analysis and a plotted thermal dehydration curve for each shale would supply helpful information in determining the optimum conditions as to temperature and time of pre-heating. In certain of the experiments it seems obvious that so much of the combined water had been driven off during the pre-heating that there was not sufficient remaining to produce a satisfactory bloat unless a much higher temperature was employed. Even then, the products were not comparable in properties.

In all the experiments so far, the shale particles have been in intimate contact during the bloating operation. As might be expected

note
refer to
Feb. memo of
George S. Bell
noted by Ehlers
p. 95

the bloated particles fused together more or less. In some instances, adhesion was slight and only at points of contact. In other instances, the product had softened, settled, and conformed to the inside of the crucible to such an extent that the mass was virtually monolithic and required crushing in order that it might serve as aggregate. Such products are of the sinter type as shown in Figure 2. It is impossible to make a satisfactory comparison of such products with those of the coated type from figures on porosity and absorption, especially where the coated type material has a tight vitreous sealed coat. Sinter-type products resemble clinker and require crushing. Crushing exposes the inner cellular or porous structure. Depending upon whether the structure is cellular or porous, the fractured surfaces will absorb more or less water when the crushed material is immersed. Usually, the sinter-type aggregate show higher porosity and absorption than the coated type. Further, the crushed products are extremely harsh, and concrete prepared with them is more difficult to handle than concrete in which coated products are used. However, it should be noted that some products which from their appearance may be classed as coated exhibit high porosity and absorption, probably because the coating is not vitreous and tight. For example, the product made from shale Lab. No. 10114 by pre-heating at 230°F. and bloating at 2200°F. appeared to be a coated product, but its absorption was relatively high. When the firing was carried out on an open hearth rather than in a crucible a product was obtained that had low absorption and a well vitrified coating. In contrast, a product made from shale Lab. No. 10117 by pre-heating at 230°F. and bloated at 2100°F. exhibited a high porosity and absorption when processed in a crucible and similar results when processed on an open hearth. These comparisons are shown in Table VII. No explanation is offered for the incongruities. Rather, they are accepted as evidence that each shale deposit is a separate and distinct problem and that minor differences in procedure will cause appreciable changes in the products. The effect of a particle size is shown by the lower temperature at which the brick formed from pulverized shale bloated in comparison with the coarse granular material.

Figure 2 shows a clinker product obtained from Lab. No. 10115 by pre-heating at 230°F. and bloating at 2200°F. The broken pieces as pictured are reduced in size to approximately 50 percent of the actual size.

Figure 3 shows pieces of Lab. No. 10114 before and after processing. The shale was pre-heated at 230°F. and fired at 2200°F. on an open hearth. The pieces as pictured are reduced in size to approximately 30 percent of the actual size.

All photographs were made by Myron E. McKinley, Assistant Geologist.

SUMMARY

The shales of Pennsylvanian age tested and reported on herein all expanded or bloated when subjected suddenly to relatively high temperatures. Generally, the higher the temperature the greater the reaction so long as the mass did not lose its thermoplasticity and viscosity and become too fluid. Evidence is offered that impurities occurring with the clay minerals act as fluxing agents, influencing the temperature at which the thermoplastic state begins and also the range through which the thermoplastic state is maintained. It is evident, too, that shales from different formations react differently, yielding products with different characteristics. Further, similar differences are found from location to location in any formation, and from layer to layer at any location. The differences in the products are apparent in the color, the structure, and in other respects.

A consideration of these facts will serve as a warning in the prospective commercial exploitation of shales as raw material for lightweight aggregate. The shale deposits should be carefully and thoroughly prospected, sampled and tested to determine their extent and the variations in composition and reactivity. The property can then be soundly evaluated, and selectively quarried if that is found desirable. It is probable that most of the shales of Pennsylvanian age in northeastern Oklahoma can be converted to lightweight aggregate, if the proper method and working conditions can be ascertained. Pre-heating may be desirable with certain shales in order to obtain an aggregate with desired character, and with others it may be a distinct disadvantage. If pre-heating is used it will probably be for the purpose of controlling the amount of water which will be liberated at the bloating temperature rather than solely as a fuel conservation means, unless an equally satisfactory product can be obtained with or without pre-heating.

Either coated or sintered aggregate can be produced from the shales tested. The factors that may decide the type product are the kind of fuel available and its cost, capital available, markets and kind of competition. For a coated product it would be good practice to classify the raw shale on particle size and adjust the bloating conditions for the particular size. Pilot plant operation is recommended before building a plant to handle commercial tonnage.

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TABLE I

Lab. No.	Field Sheet	Location	Section-Township-Range-County	Formation	Particulars
10111	0935	SW $\frac{1}{4}$.NE $\frac{1}{4}$	9 15N 16E Okmulgee	Senora	10 feet slightly oxidized, partly weathered shale just above the Henryetta coal.*
10112	0935	SW $\frac{1}{4}$.NE $\frac{1}{4}$	9 15N 16E Okmulgee	Senora	5 feet of shale immediately above Lab.No. 10111.*
10113	0936	SE $\frac{1}{4}$.SE $\frac{1}{4}$	22 15N 16E Muskogee	Boggy	20 feet compact, slightly silty shale from cut on north side U.S. 64 at Yahola hill. Bottom of sample begins at level where shale has been worked for use in highway fill. Top of sample is at a yellow sandy zone.
10114	0937	C SE $\frac{1}{4}$	2 20N 15E Rogers	Senora	16 feet light gray, hard shale above the Broken Arrow coal. The 9 feet above this sample was badly oxidized and weathered.*
10115	0938	SE $\frac{1}{4}$.SE $\frac{1}{4}$.SW $\frac{1}{4}$	17 22N 10E Tulsa	Seainole	6 feet of shale above the Dawson coal in strip-pit. Shale is partly oxidized and weathered.
10116	0939	NE $\frac{1}{4}$.SE $\frac{1}{4}$.NE $\frac{1}{4}$	2 17N 10E Creek	Nelly Bly	25 feet gray, slightly weathered shale which contains several beds of limestone about 1 inch in thickness. Bottom of sample begins at road level on Turner Turnpike.
10117	0939	NE $\frac{1}{4}$.SE $\frac{1}{4}$.NE $\frac{1}{4}$	2 17N 10E Creek	Nelly Bly	Represents 25 feet of shale above Lab.No. 10116. Only 50-60 percent of section represented by Lab.No. 10117 is exposed. Shale is slightly weathered but not oxidized.

above Henryetta

Yahola Hill

above Broken Arrow

above Dawson

Turner Turnpike

*All three samples from the Senora formation contain thin layers of light brown material whereas the main body of the shale is drab gray.

The % 1111 Fe may be calculated off by assuming that Potassium found by chemistry analysis represent 5.9 to 6.0% of the 1111 Fe

TABLE II

Chemical Analyses

Lab. No.	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO ₂	TiO ₂	P ₂ O ₅	CaO	MgO	BaO	ZnO	V ₂ O ₅	CO ₂	SO ₃	S	K ₂ O	Na ₂ O	H ₂ O	LOI	GOI
10111	62.54	16.40	6.93	0.13	0.84	-	0.90	1.91	nil	-	-	1.72	nil	-	2.60	1.01	1.69	7.01	
10112	62.20	18.18	6.51	0.15	0.78	-	0.64	1.90	nil	-	-	0.41	nil	-	2.89	0.89	1.89	6.24	
10113	61.08	17.98	7.23	0.17	0.89	nil	0.66	1.73	nil	0.19	0.07	-	-	0.07	2.14	1.68	0.51	7.08	
10114	61.65	18.85	6.70	0.11	0.89	-	0.40	1.96	nil	-	-	1.46	nil	-	2.95	1.42	0.66	6.10	
10115	54.34	19.30	6.68	0.08	0.93	-	2.95	2.18	nil	-	-	1.75	-	0.01	3.62	1.68	1.70	7.12	
10116	58.00	18.39	7.85	0.06	0.72	nil	0.86	2.18	0.49	-	0.06	-	0.31	-	2.32	1.46	1.70	6.30	
10117	60.78	14.67	5.73	0.09	0.91	-	2.58	2.57	nil	-	-	3.46	-	0.20	2.52	1.44	1.04	7.29	
10127*	40.60	13.34	31.41	0.73	0.47	-	0.70	1.11	-	-	-	-	v. sl. tr.	-	-	0.33	1.95	8.84	
10121#	53.1	18.0	21.4	0.4	0.5	-	1.7	2.2	-	-	-	-	-	-	-	-	-	-	1.5

LOI (loss on ignition)
GOI (gain on ignition)
Total Fe reported as Fe₂O₃.

*Selected light brown pieces from Lab.No.10111, analysis by A.L. Burwell.

#Selected glossy black pieces in the bloated product from Lab.No.10114.

All analyses, except 10127, by Thomas E. Hamm.

H₂O is ~~6.0%~~ ^{6.0%} based at 110°C.
LOI is " was about 110°C "

1100°C
1090°C

TABLE III

Expansion Tests on Shales of Pennsylvanian Age

Test pieces prepared from minus 10 mesh shale and 30 percent water; cast in mold 2.6 x 2.6 x 3.6 cms. Cut into portions approximately 5 cms. in length, and dried at 230°F.

Lab. No.	10111	10112	10113	10114	10115	10116	10117
Approx. bulk density of oven dried pieces.	1.83	1.90	1.76	1.76	1.81	1.88	1.88
Pieces into furnace at 230°F. Temperature raised gradually to 1830°F. Cooled under sand							
Shrinkage in volume	23.0%	11.5%	11.5%	11.5%	11.5%	11.5%	11.5%
Bulk density	2.45	2.15	2.15	2.15	2.15	2.15	2.15
Pieces from oven at 230°F., into furnace at 1900°F. for 15 minutes. Cooled under sand							
Expansion in volume	50.5%	59.0%	20.0%	27.5%	27.5%	59.0%	35.0%
Bulk density	1.25	1.20	1.60	1.50	1.50	1.20	1.40
Pieces from oven at 230°F., into furnace at 2000°F. for 15 minutes. Cooled under sand							
Expansion in volume	95.5%	116.0%	95.5%	95.5%	160.0%	126.5%	59.5%
Bulk density	0.97	0.88	0.97	0.97	0.73	0.84	1.19
Pieces from oven at 230°F., into furnace at 2100°F. for 15 minutes. Cooled under sand							
Expansion in volume	210.0%	210.0%	160.0%	185.0%	semi-fluid	330.0%	185.0%
Bulk density	0.60	0.60	0.73	0.67	-	0.44	0.65

14.22

18.85

TABLE IV
Bloating Tests

Shale particle size from 10 mesh to 1 inch. Processed in covered fire clay crucibles after pre-heating at 1100°F. for 3 hours. (a) time required to regain temperature in furnace after introducing the crucibles. (b) time held at bloating temperature.

Lab. No.	Bloated at °F.	Duration (a) (b) minutes	Average Apparent Density	Average Porosity % by vol.	Average Absorption % by wt.	Comments
1150°C 1170°C 1200°C 18	10111	2100 12 8	1.99	6.8	3.4	light brown: some slight evidence of bloating.
		2150 10 8	2.00	3.5	1.6	drab brown: some slight evidence of bloating.
		2200 8 12	1.76	5.2	2.9	light drab; slight evidence of bloating.
	10112	2100 7 13	1.65	3.9	2.3	red. brown: fair bloat
		2200 5 15	1.81	20.3	11.3	drab brown: most pieces bloated.
		2300 - 15 -	1.31	4.8	3.6	some cream color; slight expansion and bloat.
	10113	2100 7 8	1.66	12.2	7.4	red. brown: most pieces bloated.
		2200 10 8	1.67	7.8	4.7	drab brown: most pieces bloated.
		2300 4 8	1.48	9.2	6.2	deep dark brown: most pieces bloated.
10114	2100 7 15	2.29	1.8	0.8	brown: very little bloat.	
	2200 9 15	1.78	0.0	0.0	dark brown: no evidence of bloating.	
	2300 10 15	1.66	2.9	1.7	dark brown: some bloated pieces.	
1150°C 1200°C 1260°C	10115	2100 7 8	1.99	2.2	1.1	red. brown: very little bloat.
		2200 7 8	1.03	5.3	5.1	choc. brown: fine cellular bloated product.
		2300 11 4	1.04	10.0	9.6	dark choc. brown: glassy; large cells; over-bloated.
10116	2100 7 8	1.43	16.4	11.5	red. brown: most pieces bloated.	
	2200 10 8	1.22	16.3	13.4	brown: thick coated, cellular bloat.	
	2300 4 8	1.19	28.7	24.0	dark brown: most pieces bloated.	
10117	2100 7 8	1.45	21.4	14.8	red. brown: most pieces bloated.	
	2200 10 8	0.98	27.6	28.1	drab brown: bloated.	
	2300 - 15 -	1.23	23.6	19.2	drab: spongy bloat.	

TABLE V
Bloating Tests

Pre-heat at 230°F. vs Pre-heat at 1100°F.

Lab. No.	Pre-heated at °F (x)	Bloated at °F (y)	Duration in minutes (a) (b)	Average Apparent Density	Average Porosity % by vol.	Average Absorption % by wt.	Comments
17	10111	230	2000 ^{1090°C} 10 15	1.41	21.8	15.4	Yellowish color; few swollen pieces.
		230	1100 2000 10 15	2.07	10.2	4.9	Light brown: very little bloat.
		230	2100 ^{1150°C} 10 15	1.12	20.0	20.3	Lt. brown: coated cellular product.
		230	1100 2100 10 15	2.33	6.0	2.6	Few pieces slightly swollen.
		230	2200 ^{1200°C} 10 15	1.00	2.0	2.0	Cellular product, partly sintered.
		230	1100 2200 10 15	2.15	5.1	2.4	Dark brown: very little bloat.
		230	2300 ^{1260°C} 10 15	0.81	26.7	33.0	Cellular, mostly over-bloated.
		230	1100 2300 10 15	1.52	9.4	6.2	Few pieces swollen slightly.

Note: (x) pre-heated at 230°F. for 18 hours.

(a) time required to regain temperature drop.
(b) time at temperature after regaining temperature drop when charging furnace.

(y) same plus 3 hours additional at 1100°F.

TABLE VI

Effect of Time on Character of the Bloat

Lab. No.	Pre-heated at °F.	Bloated at °F.	Duration in minutes	Average Apparent Density	Average Porosity % by vol.	Average Absorption % by wt.	Comments
10116	1100	2100	15	2.43	13.6	5.6	Unsatisfactory bloat; porous.
	1100	2100	30	2.49	4.0	1.6	Unsatisfactory bloat; less porous.
	230	2100	15	1.19	24.0	28.7	Drab brown: coated, cellular; not sealed.
	230	2100	30	1.11	5.0	4.5	Lt. red. brown: coated, cellular; sealed.

Shale sized to 6 mesh to 1 inch.

TABLE VII

Bloating Tests

Lab. No.	Bloated at °F.	Duration		Average Apparent Density	Average Porosity % by vol.	Average Absorption % by wt.	Apparent Density Individual Pieces			Comments
		(a)	(b)				(1)	(2)	(3)	
✓ 10111	2100*	15	10	1.33	6.1	4.6	1.74	1.18	0.85	Coated and tight seal.
10112	2100*	15	10	1.41	3.9	2.3	1.99	1.38	0.76	Coated and tight seal.
10113	2100*	15	10	1.54	22.8	14.7	1.57	1.43	1.18	Coated but not sealed.
10114	2100*	15	10	1.00	3.3	3.2	1.67	1.02	0.69	Coated and partly sealed.
← 10115	2100*	15	10	0.85	24.6	26.9	0.96	0.86	0.56	Sinter; required crushing.
10116	2100*	15	10	1.24	21.3	17.3	1.92	0.95	0.91	Coated but not sealed.
✓ 10111	2200*	15	10	0.80	7.7	9.2	0.78	0.81	0.63	Coated and partly sealed.
10112	2200*	15	10	1.10	4.3	3.8	0.94	0.98	2.77	Coated and sealed.
10113	2200*	15	10	0.77	18.4	20.1	0.75	0.77	0.81	Sinter; cellular; crushed.
10114	2200*	15	10	0.97	7.7	9.4	0.80	1.14	0.92	Coated; partly sealed.
← 10115	2200*	15	10	0.58	7.8	12.5	0.69	0.59	0.56	Sinter; cellular; crushed.
10116	2200*	15	10	0.71	10.9	15.0	0.87	0.72	0.70	Sinter; cellular; crushed.
10117	2200*	15	10	0.95	12.3	12.4	0.58	0.78	1.11	Coated but not sealed.
10114	2200#	15	10	0.78	4.4	5.7	-	-	-	Coated and sealed.
10117	2100#	5	10 ^Z	1.21	20.5	16.9	0.43	-	-	Coated but not sealed.

Note: (a) time required to regain temperature drop.

(b) time at bloating temperature after regaining drop.

(*) processed in fire clay crucible.

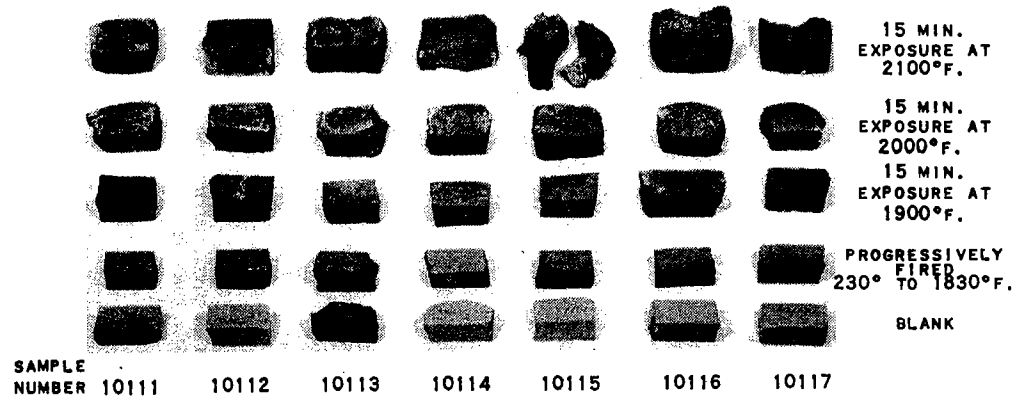
(#) processed on open hearth.

(Z) At expiration of bloating time the furnace was opened and plugs removed to hasten cooling. Furnace temperature dropped to 1800°F. in 5 minutes; to 1620°F. in 10 minutes; to 1500°F. in 15 minutes; and to 1000°F. in 1 hour.

2100°F
1150°C2200°F
1204°C

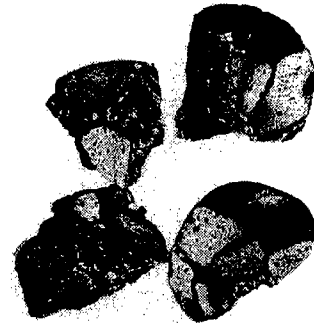
FIGURE 1

BLOATING TESTS



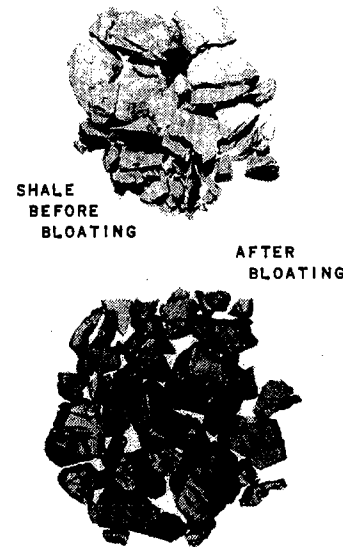
SHALE AIR DRIED, CRUSHED AND PULVERIZED, PORTIONS MIXED WITH 30% WATER BY WEIGHT, CAST IN MOLD, AIR DRIED AND OVEN DRIED AT 230°F.

FIGURE 2



SINTER-TYPE AGGREGATE,
1/2 ACTUAL SIZE

FIGURE 3



COATED AGGREGATE,
3/10 ACTUAL SIZE

Bloating Properties of Shale
in the
Hilltop Formation in Seminole County

Bloating Properties of Shale in the Hilltop Formation in Seminole County

ALBERT L. BURWELL¹

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 II, 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 screened 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:

CHEMICAL ANALYSIS OF SHALE IN HILLTOP FORMATION

Seminole County *Lab. No. 10037*

—%—

SiO ₂	62.25	R ₂ O ₃	24.89	K ₂ O	2.45
Al ₂ O ₃	16.64	CaO	0.85	Na ₂ O	0.39
Fe ₂ O ₃	5.31	MgO	2.23	ZrO ₂	0.07
FeO	1.52	MnO ₂	0.05	V ₂ O ₅	0.30
TiO ₂	0.85	SO ₃	0.03	BaO	None
H ₂ O	2.03	P ₂ O ₅	0.03		detected
	at 110°C.			L.O.I.	5.65
	<i>88.60</i>		<i>3.19</i>		above 110°C. <i>886</i>
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

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.

88.6
3.19
88.60
100.65

CO₂ 0.00
76

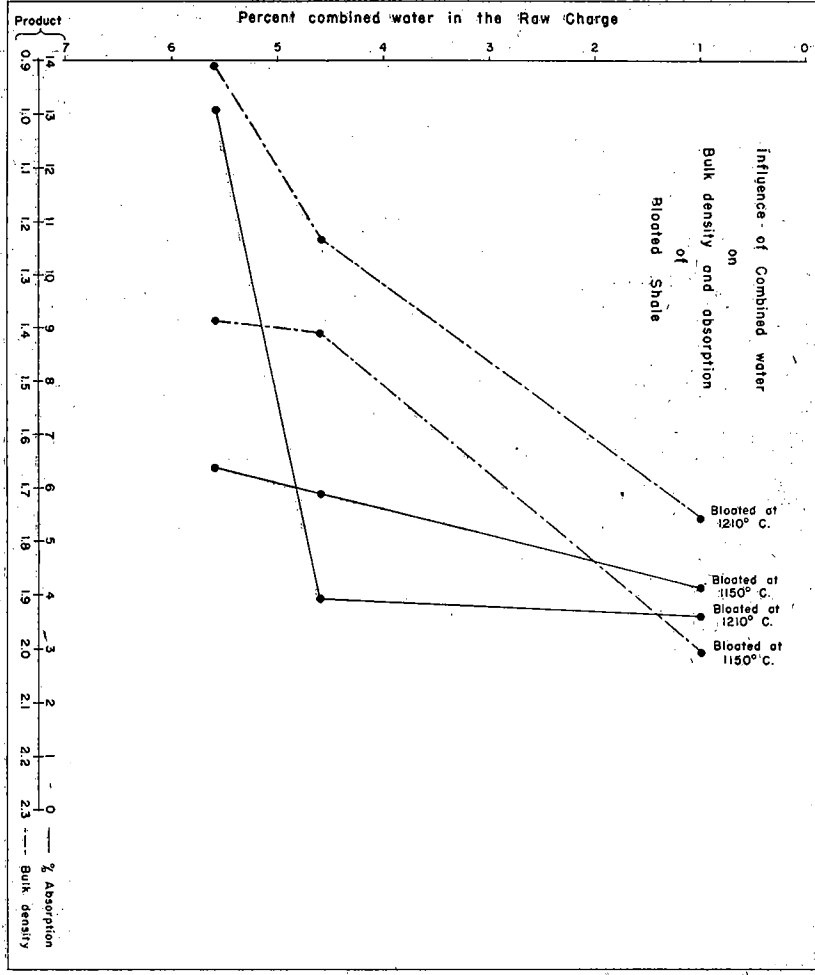


Figure 1. Influence of combined water on bulk density and absorption of bloated shale.

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. #	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	2.6	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	7.1	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	2.5	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	2.0	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	3.6	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	0.6	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
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	19.3

* 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 limy 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.