

PRODUCTION OF LIGHTWEIGHT CONCRETE AGGREGATE
" "
FROM VIRGINIA SHALE

by
George F. Meenaghan
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APPROVED:

APPROVED:

Director of Graduate Studies

Head of Department

Dean of Engineering

Supervisor or Major Professor

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TABLE OF CONTENTS

	Page
I. INTRODUCTION	1
II. LITERATURE REVIEW	3
Structure of Silicates	3
Early Investigation	3
Hexite and Pentite Theory	6
X-ray Study of Silicates	6
Tetrahedral Structure of Silicates	10
Tetrahedral Groups	12
The Action of Heat on Clays and Shales	16
Melting Points of Silicates	16
Temperature - Specific Gravity Relationship	17
Dehydration Temperatures	18
Expansion on Heating	19
Evolution of Heat	20
Other Theories on Action of Heat	21
Causes and Preventions of Bloating or Swelling of Clays and Shales	23
Blistering of Cornish Stone	23
Shale and Clays	23
Prevention of Bloating	25

	Page
Methods of Bloating Clays and Shales	26
Additives that Promote Bloating	26
Natural Bloating Clays and Shales	27
Lightweight Concrete Aggregate	28
Application of Lightweight Concrete	28
Need for Design Information	30
Chemical Constituents	30
Conditions Necessary for Bloating	31
Formation of Small Cells During Firing	32
Commercial Lightweight Aggregates	33
Shalite	33
Haydite	33
Lite-Rock	34
Kanamite	35
Patents	37
Early Patents	37
The Hayde Patent	38
Recent Related Patents	39
III. EXPERIMENTAL	43
Purpose of Investigation	43
Plan of Experimentation	43
Literature Review	44

	Page
Obtaining Shale Samples	44
Bloating Characteristics	44
Determination of the Bloating Temperature of Shale	44
Effect of Prolonged Heating on the Bloating of Shale	45
Effect of Chemical Composition on Bloating	45
Preparation of Load-bearing Test Cylinders	45
Testing of Bloated Shale	46
Materials	47
Apparatus	54
Methods of Procedure	58
Calibration of Thermocouple	58
Calibration of Pyrometer	58
Determination of the Temperature Lag in a Muffle Furnace	59
Code of Identification of Shale Samples	59
Preparation of Shale Samples	59
Determination of Firing Schedule	60
Procedure for Determining the Bloating Characteristics for Different Samples of Shale	60

	Page
Procedure for Determining the Bloating Temperature of Shale	62
Procedure for Determining the Time of Exposure to a Particular Temperature on the Bloating Characteristics of Shale	63
Procedure for Determining the Effect of Rapid Heating on the Bloating Characteristics of Shale	65
Chemical Analyses	66
Comparison of Crushing Strength of Concrete Made with Cinder Aggregate and Concrete Made with Expanded Shale Aggregate	67
Data and Results	69
Firing Schedule	69
Bulk Densities of Unburned Shale	69
Bulk Densities of Burned Shale	69
Rank of Bloating	69
Bloating Temperature	69
Time of Exposure	70
Rapid Heating	70
Chemical Analyses	70
Preparing Test Cylinders	70
Compressive Strength	70
Sample Calculations	86
Bulk Density	88

	Page
IV. DISCUSSION	89
Discussion of Materials, Equipment, and Conditions for Producing a Lightweight Aggregate	89
Shale Tested	89
Shale Sampling	90
Muffle Furnace	91
Sample Preparation	92
Calibration of Thermocouple and Pyrometer	92
Temperature Lag	93
Firing Schedule	94
Bloating Conditions	96
Discussion of Results	97
Non-weathered Liberty Hall Shale	97
Weathered Liberty Hall Shale	99
Martinsburg Shale	100
Brallier Shale	102
Weathered and Non-weathered MacCraday Shale	104
Determination of the Temperature of Bloating	104
Determination of the Effect of Time of Exposure to 2050 °F on the Bloating of Non-weathered Liberty Hall Shale	107

	Page
Determination of the Effect of Rate of Heating on the Bloating of Non-weathered Liberty Hall Shale	108
Effect of Chemical Composition on the Bloating Qualities of Shale	110
Comparative Compressive Strengths of Concrete Made with Cinder as an Aggregate and of Concrete Made with Bloated Shale as an Aggregate . .	114
Recommendations	118
Rotary Kiln	118
Higher Bloating Temperatures	118
Chemical and Petrographic Analyses . . .	118
Compressive Strength Tests	118
Types of Shale	119
Condition of Shale	119
Type of Atmosphere	119
Limitations	120
Shale Samples	120
Muffle Furnace	120
Temperature Range	120
Analyses	121

	Page
V. CONCLUSIONS	122
VI. SUMMARY	125
VII. BIBLIOGRAPHY	127
VIII. ACKNOWLEDGMENTS	138
IX. VITA	139

LIST OF TABLES

		Page
TABLE I.	Procedure Used in Removing Shale Samples from Furnace During Determination of Bloating Temperature	64
TABLE II.	Data Obtained for the Firing Schedule Used to Bloat the Shale Used During the Course of this Investigation	71
TABLE III.	Data Used for Calculation of Bulk Densities of Unburned Shale Samples from Various Deposits Located in Virginia	73
TABLE IV.	Data Used for Calculation of Bulk Densities of Burned Shale Samples from Various Deposits Located in Virginia	74
TABLE V.	Rank of Bloating and Ratios of Bulk Densities of Unburned to Burned Shale Samples from Various Deposits in Virginia	75
TABLE VI.	Data Used for Determining the Temperature at Which Bloating Occurs for Liberty Hall, Martinsburg and Brallier Shales	76
TABLE VII.	Data Used for Determination of Time of Exposure to 2050 °F on the Bloating Characteristics of Non-weathered Liberty Hall Shale	81
TABLE VIII.	Bulk Densities of Non-weathered Liberty Hall Shale When Heated to 2050 °F in One Hour-Forty-Five Minutes Compared to Bulk Densities When Heated to 2050 °F in Twenty Minutes	82

	Page
TABLE IX. Chemical Analyses of Non-weathered MacCrady and Liberty Hall Shales Obtained from Deposits Located in Virginia	83
TABLE X. Grading of Aggregate Used to Make Concrete for Compressive Strength Cylinders Used to Compare Strengths of Cinder Concrete and Bloated Shale Concrete	84
TABLE XI. Compressive Strengths of Concrete Test Cylinders Made with Cinders and Bloated Virginia Shale	85

LIST OF FIGURES

	Page
Figure 1. Structural Formulas	5
Figure 2. Hexite and Pentite Theory	7
Figure 3. Tetrahedral Groups of Silica	13
Figure 4. Typical Chain Structure	15
Figure 5. Topographical Map Showing the Location of the Liberty Hall, Martinsburg and Brallier Shale Deposits	50
Figure 6. Topographical Map Showing the Location of the MacCrary Shale Deposit	52
Figure 7. Firing Schedule Used to Bloat the Shale	72
Figure 8. Relationship of the Temperature at Which Non-weathered Liberty Hall Shale was Removed from the Furnace to its Bulk Density	77
Figure 9. Relationship of the Temperature at Which Weathered Liberty Hall Shale was Removed from the Furnace to its Bulk Density	78
Figure 10. Relationship of the Temperature at Which the Non-weathered Martinsburg Shale was Removed from the Furnace to its Bulk Density	79
Figure 11. Relationship of the Temperature at Which the Non-weathered Brallier Shale was Removed from the Furnace to its Bulk Density	80

Figure 12.	Composition Diagram of Major Oxides Showing the Area in Which Clays Form a Mass Viscous Enough to Insure Good Bloating	112
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I. INTRODUCTION

The use of lightweight concrete is not new, it having been employed in the early days of the Roman Empire when pumice was used as a component of temple roof slabs. Today, the practice has been rediscovered and many types of lightweight concrete are in use. Probably the most notable example is the placing of "Gravelite" lightweight concrete in the upper deck of the San Francisco-Oakland Bay bridge where a \$3,000,000 saving was attributed to the reduction of dead load.

During the past decade, most of the producers of lightweight concrete products have been using by-product aggregates such as cinders, air-cooled slag, and coke, however, today the supply of these by-product aggregates is diminishing rapidly. In Virginia, there are no known naturally occurring lightweight materials available for use as aggregates and steel mill slag cannot be used because of the high transportation cost. On the other hand, there is available an unlimited supply of easily quarried shale.

The producers of lightweight concrete in the state of Virginia are aware of a possible shortage of cinders and also the need of a lightweight concrete and are trying to convert

shale deposits into a suitable substitute for cinders. Preliminary investigations of this Virginia shale have shown that it will expand, or bloat, to a cellular, lightweight product when exposed to a temperature of about 2000 °F but other information concerning the properties of the shale is desired.

The purpose of this investigation was to determine the effect of time, temperature, and chemical composition on the bloating characteristics of MacCrady, Liberty Hall, Martinsburg, and Brallier shales obtained from deposits in the state of Virginia for the production of a lightweight concrete aggregate.

II. LITERATURE REVIEW

The following review of the literature related to shale, its structure, properties, and uses has been made. This review contains only essential information because of the large amount of literature on this subject.

Structure of Silicates

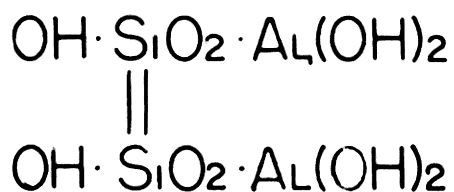
The structure of shale is the same as that of the silicates. For many years chemists, engineers, and other scientists have been making thorough studies of the action of heat on the physical and chemical structure of the silicates. In the following section some of the more important phases of silicate structure are discussed.

Early Investigation. In 1910, Pukall⁽⁶¹⁾ wrote of his experiments with clay substances. He prepared silicates of various metals by fusing silica and sodium carbonate and then precipitating the silicates by double decomposition with metal salts and sodium silicate. An aluminum silicate with the ratio of three parts of silica to two of alumina to six parts of water was produced. From these experiments Pukall proposed the structures for kaolin acid, kaolin (which he

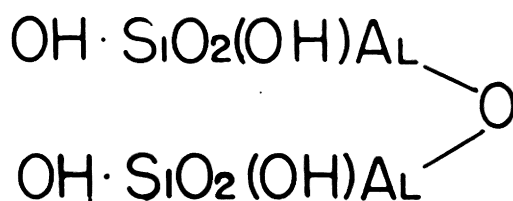
termed dehydrated kaolin acid), and the sodium salt of the acid. These structures are shown in Figure 1, page 5.

Singer⁽⁶⁹⁾, in 1911, proposed the structural formula $(\text{HO.Si.O.OAl}(\text{OH}))_2\text{O}$. He stated that only metasilicic acid may be considered the foundation of all natural and artificial silicates. All possible pyrosilicic acids are derived from $\text{O:Si}(\text{OH})_2$ (metasilicic acid). Singer criticized Pukall's formula unfavorably and pointed out that a double silicon bond, like a double carbon bond, is a source of weakness in a compound rather than one of strength.

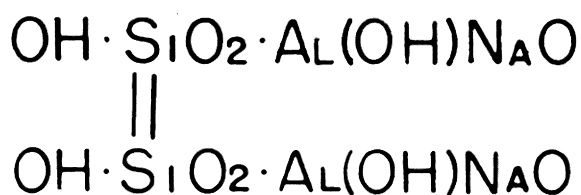
Considerable criticism of Pukall's formula was made by Wegscheider and Manchot⁽⁷⁸⁾. Wegscheider considered complex silicates as salts of polysilicic acids in which the Si groups are linked through oxygen. He made reference to Pukall's formula and pointed out that Manchot had criticized Pukall by stating that combination between Si atoms and SiO₂ combinations take place only in reducing conditions and silicates are formed without reduction. In organic combinations between silicon atoms the products are unstable, especially those containing oxygen. A formula with directly combined silicon atoms would not explain the stable condition of kaolin. If silicon atoms were directly combined in



KAOLIN ACID



KAOLIN



SODIUM SALT

FIGURE 1. STRUCTURAL FORMULAS

PUKALL, W.: DEVELOPMENT IN THE FIELD OF CLAY INDUSTRY,
CHEM.-ZTG., 34, 610-3 (1909); C. A., 4, 3126 (1910).

silicates, the oxygen atoms must also be directly combined. It is known that compounds with directly combined oxygen atoms are unstable (viz H_2O_2). Wegscheider went on to state that regarding silicates as salts of polysilicic acid does not prevent one from regarding those containing aluminum as salts of alumino-silicic acid with oxygen combinations between the silicon and aluminum.

Hexite and Pentite Theory. In 1913, Asch and Asch⁽¹⁾ proposed what they termed the "Hexite and Pentite" theory. They theorized that if five and six molecules of $Si(OH)_4$, respectively, unite together, splitting off water and retaining the quadrivalency of the silicon they form a closed ring as shown in Figure 2, page 7.

Also, by substituting aluminum atoms for the silicon atoms, aluminum hexite and pentite are formed. Thus, when $3H_2O.3Al_2O_3$ is united with $6H_2O.6SiO_2$ the structural formula as shown in Figure 2, page 7, would be obtained.

The Asch and Asch brothers show, by various combinations of aluminum and silicon hexites and pentites, how the structure of a large number of known silicates can be explained.

X-ray Study of Silicates. In spite of the large amount of work done and the apparent logic of some of the theories mentioned, it remained for Bragg and Pauling⁽⁴⁵⁾ to elucidate

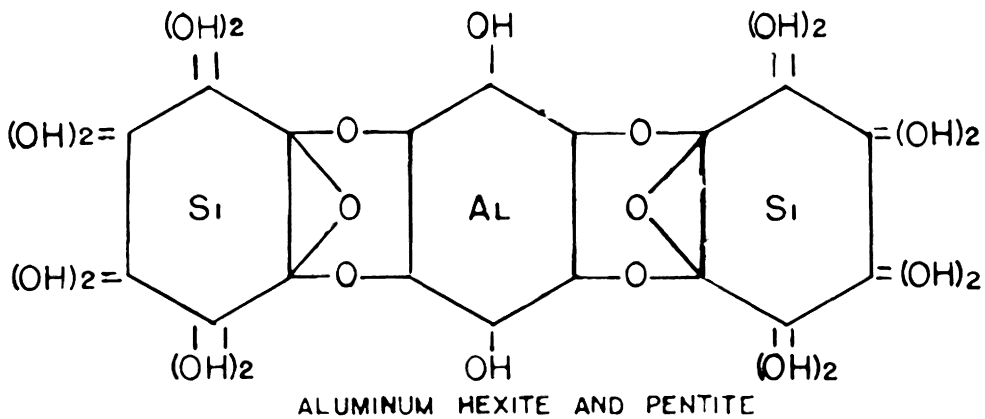
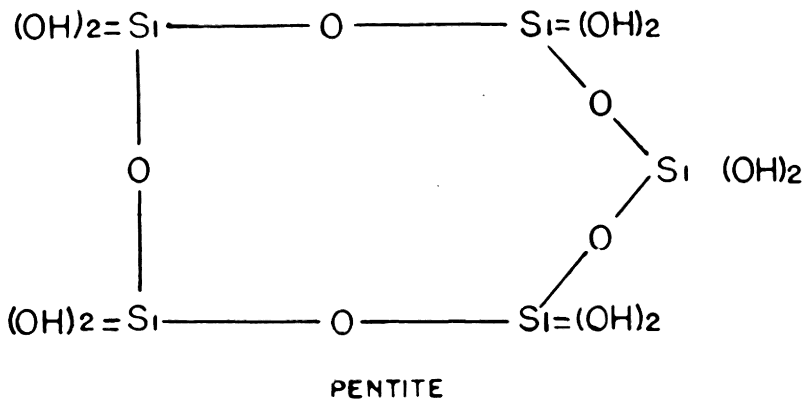
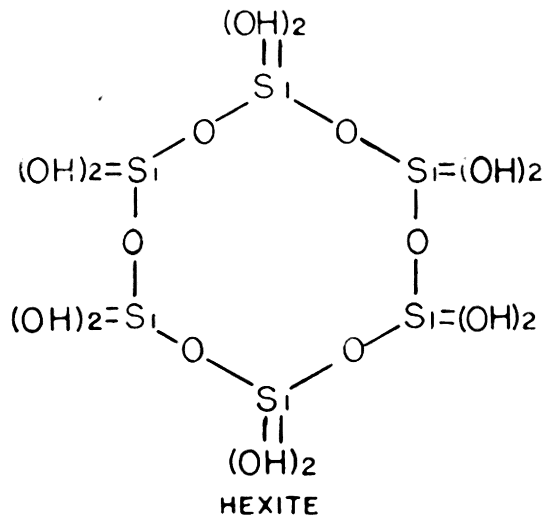


FIGURE 2. HEXITE AND PENTITE THEORY
ASCH, W. AND D. ASCH: "SILICATES IN CHEMISTRY AND
COMMERCE," PP. 30-40. CONSTABLE AND CO., LONDON, 1913.

the different problems involved in explaining the structure of the silicates by x-ray analysis. They showed that the difference between the classes of silicates is directly dependent on the silicon and oxygen arrangement and only indirectly dependent on the remainder of the structure. Occasionally the silicon oxygen arrangement forms a closed group like an ordinary acid radical, but usually the structure is endless and extends continuously throughout a given crystal. The silicon atom, in the silicate, is always associated with four oxygen atoms, tetrahedrally arranged with regard to the silicon. The way in which these tetrahedral groups are related determine the structure of the particular silicate.

Mellor⁽⁴⁵⁾ states that many silicates are derived from various silicic acids. Metasilicic acid is formed by the following reaction:



Metasilicic acid exists as hydrogel and hydrosol; the hydrogel being the gelatinous form and the hydrosol being a solution. If sufficiently dilute, all of the metasilicic acid takes the hydrosol form. Clear solutions containing five per cent colloidal silica can be obtained by dialysis. These solutions

can be concentrated in a flask by boiling until they contain about fourteen per cent H_2SiO_3 , which, on standing a few days, transforms to a hydrogel. The transformation of hydrosol to hydrogel is retarded by small amounts of hydrochloric acid or an alkali hydroxide and accelerated by sodium carbonate. If the clear solution is evaporated in a vacuum at fifteen degrees Centigrade, clear, transparent jelly is obtained which, when dried over sulfuric acid, has the approximate composition of $\text{SiO}_2 \cdot \text{H}_2\text{O}$ or H_2SiO_3 , metasilicic acid. This may also be prepared by dehydrating the gelatinous silicic acid with ninety-five per cent alcohol. When the gelatinous silicic acid is dehydrated with ether and dried, an acid of the composition $\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ or H_4SiO_4 , orthosilicic acid, is obtained. Orthosilicic acid loses water on exposure to air. On the other hand, silica gel of the formula $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ has a remarkable capacity for absorbing water from the air.

For chemical purposes, silicates have been classified on the basis of these silicic acids. Thus, willemite, Zn_2SiO_4 , corresponds to the orthosilicic acid as does olivine, $(\text{MgFe})_2\text{SiO}_4$; zircon, ZrSiO_4 ; and tephroite, Fe_2SiO_4 ; while wollastonite, CaSiO_3 , and enstatite, MgSiO_3 , correspond to the metasilicic acid.

Another series of silicic acids result when two molecules of orthosilicic acid condense into one of ortho-disilicic acid, $H_6Si_2O_7$. This gives rise to a series of ortho-disilicates such as serpentine, $MgSi_2O_7$; barysilite, $PbSi_2O_7$, etc. In like manner, the condensation of two molecules of metasilicic acid gives rise to a series of meta-disilicates. This process is continued for ortho-, meta-, tri-, and polysilicic acids.

Tetrahedral Structure of Silicates. Bragg⁽¹⁷⁾ was the first to show that silicates consist of essentially the relatively larger oxygen-oxygen ions in close packed arrangement, either cubic or hexagonal. The ions are held together by strongly charged metallic ions which fit into the spaces between them. In tetrahedral spaces are to be found the smallest and most highly charged ions, Si^{++++} , Be^{++} , Ti^{++++} , Al^{+++} , Mg^{++} , Fe^{++} , etc. Larger ions, such as Ca^{++} , Na^+ , and K^+ , introduce distortion into the structure. The symmetry of the particular silicate adjusts itself to fit these ions with minimum distortion, with the result that the unit crystal cells are large and complicated because of low symmetry.

Bragg⁽¹⁷⁾ made the following conclusions in regard to the structure of the silicates:

(1) Whatever the silicon to oxygen ratio, silicon is always situated within the tetrahedral group of four oxygen atoms, which is constant in form from crystal to crystal.

(2) The structures are typical coordination structures, four oxygen atoms at the corners of a tetrahedron, six of an octahedron, etc. The whole structure may be regarded as a fabric of which these groups are the stitches, the groups being joined together by sharing oxygen atoms.

(3) The coordination numbers among silicates are
Be⁺⁺ 4; B⁺⁺⁺ 3, 4; Na⁺ 6, 8; Mg⁺⁺ 4, 6, 8; Al⁺⁺⁺ 4, 5, 6;
Si⁺⁺ 4; Ca⁺⁺ 6, 7, 8; Sc⁺⁺⁺ 6; Ti⁺⁺ 6; Mn⁺⁺ 4, 6, 8;
Fe⁺⁺⁺ 6; Zn⁺⁺ 8; Ba⁺⁺ 6, 12; K⁺ 6, 8.

Diopside is a typical meta-silicate of the formula $\text{CaMg}(\text{SiO}_3)_2$. Each silicon atom is surrounded tetrahedrally by four oxygen atoms. Two of these atoms are common to neighboring groups and two are not common with adjacent groups. The tetrahedra are linked by their corners into endless chains parallel to the C axis of this monoclinic

crystal. It is incidental that the SiO_4 groups are cemented sideways by calcium and magnesium ions.

Tetrahedral Groups. In orthosilicate groups⁽⁴⁵⁾ exemplified by olivine, $(\text{MgFe})_2\text{SiO}_4$ and zircon, ZrSiO_4 , the tetrahedral groups are independent like quadrivalent acid radicals. The oxygen atoms each have one negative charge, the oxygen atoms being attached to the silicon by a single valency. These negative charges attract the metallic ions of the crystal. The resulting structure is shown in Figure 3, part A, page 13.

When two of the groups as shown in part A are linked by their common corners, that is, through two groups sharing one atom of oxygen, a grouping of Si_2O_7 results as shown in Figure 3, part B, page 13.

Other groups have been observed in which three tetrahedra, as shown in Figure 3, part C, page 13, are united into a ring through sharing of three oxygen atoms thus forming a group Si_3O_9 , whose effective valence is again six. An example of this structure is the mineral bentonite, $\text{BeTiSi}_3\text{O}_9$. Similarly, rings of six tetrahedra have been found, as shown in Figure 3, part D, page 13. An example of this structure is beryl, $\text{Be}_3\text{Al}_2\text{Si}_6\text{O}_{18}$. A ring composed of four tetrahedra has also

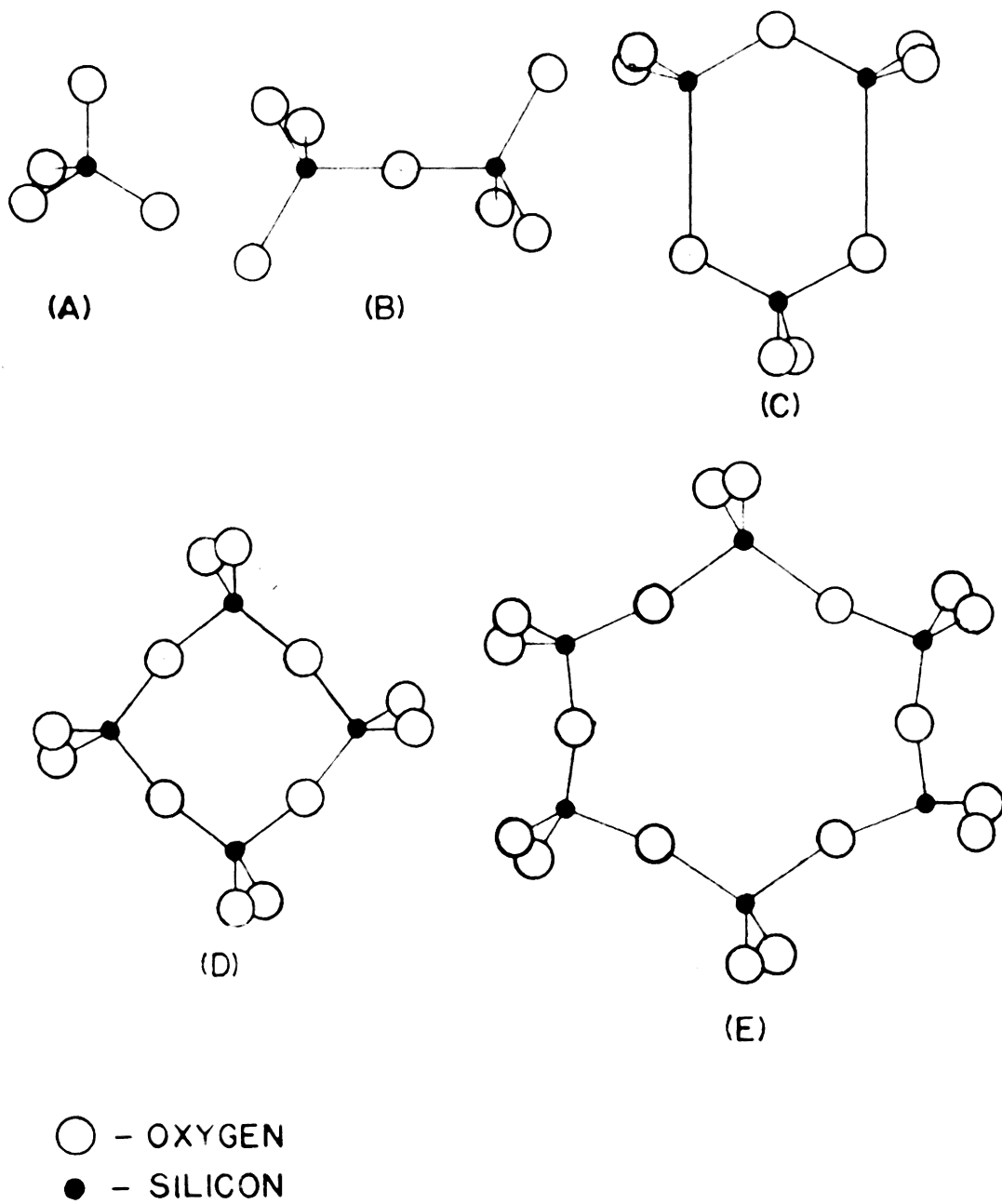


FIGURE 3. TETRAHEDRAL GROUPS OF SILICA

MELLOR, J. W. : "MELLOR'S MODERN INORGANIC CHEMISTRY,"
PP. 684-87. LONGMANS, GREEN AND CO., NEW YORK, N. Y., 1941.

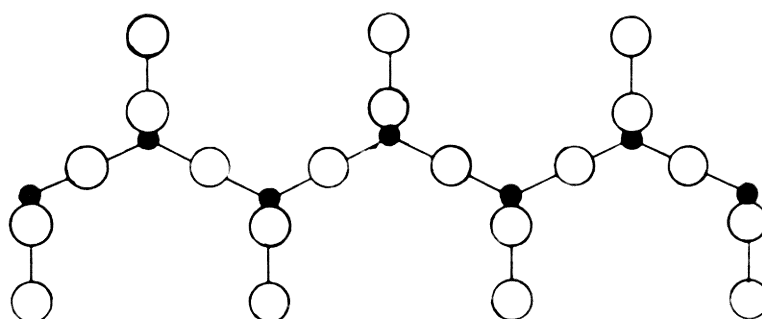
been detected in certain zeolites. This type of structure is shown in Figure 3, part E, page 13.

The silicon and oxygen tetrahedra are also found in chain, band, and sheet-type structures. An example of the chain type structure is pyroxene. This structure is shown in Figure 4, page 15, in which the tetrahedra are joined corner to corner in a row which extends indefinitely in both directions.

Examples of the band type structure exist in the amphiboles in which two chains are bound together by sharing of oxygen atoms. Chains and bands are fibrous as in asbestos.

In like manner, sheets are formed by linking three corners of the tetrahedra, leaving only one free oxygen. Examples of this structure are mica and talc.

If three dimensional networks are formed, the tetrahedra are linked by all four corners so that there are no oxygen atoms left carrying charges to attract the positive ions. The result is one of the forms of silica, for example, quartz. In most of the structures already considered, a certain number of silicon atoms may be replaced by aluminum. Whenever this occurs, each tetrahedral unit of the framework acquires a resultant negative charge on account of the lower



○ - OXYGEN
● - SILICON

FIGURE 4. TYPICAL CHAIN STRUCTURE

MELLOR, J. W.: "MELLOR'S MODERN INORGANIC CHEMISTRY,"
PP. 684-87. LONGMANS, GREEN AND CO., NEW YORK, N.Y., 1941.

valency (fewer outer ring electrons) of aluminum. Metallic ions are then incorporated into the interstices of the structure. The feldspars are examples of this arrangement.

The silicates thus, for the most part, form crystals which are not to be thought of as loose aggregations of simple molecules, but as giant molecules in which the whole mass is bound by chemical linkages which extend to the limits of the crystals. The infusibility and insolubility of the silicates is presumably the result of this structure.

The Action of Heat on Clays and Shales

The action of heat on clays and shales will be discussed under the headings of melting points of silicates, temperature-specific gravity relationship, dehydration temperatures, expansion on heating, evolution of heat, and other theories on action of heat.

Melting Points of Silicates. In 1908, Rieke⁽⁶⁴⁾ investigated the fusibility of various lime-alumina-silica mixtures such as $Al_2O_3 \cdot 2SiO_2 \cdot (0.1 \text{ to } 12)CaO$. Pyrometric cones were made and the melting point taken at the temperature at which the point of the cone first touched the bottom of the furnace. The main point of interest, as far as results were concerned,

seemed to be that the melting point decreased as the ratio of lime was increased up to a "certain point." When this "point" was reached the increase in the lime ratio resulted in an increase of the melting point.

Dittler⁽²⁰⁾, in 1911, continued the work on the melting point of the silicates. He investigated artificial diopside which he found had a freezing interval between 2354 °F and 2882 °F. He also investigated other silicates including labradorite which showed a freezing interval from 2192 °F to 2120 °F. He concluded that two processes occur in silicate fusion. First, the transformation from the crystal to the isotropic amorphous state occurred which is not accompanied by noticeable heat reaction, and second, the liquefaction of the amorphous mass followed which is accompanied by a heat reaction.

Temperature - Specific Gravity Relationship. Knote⁽³⁵⁾, in 1911, did some work on relating the temperature of the clays to the specific gravity. His results show that the specific gravity of the clay investigated increased to a temperature of 842 °F and then began decreasing until 1202 °F was reached. At 1202 °F the specific gravity began increasing again and continued to increase to 1832 °F which was the highest temperature attained.

Dehydration Temperatures. Further study by Wallach⁽⁷⁶⁾, in 1913, revealed that when clays were heated slowly from 68 °F to 2102 °F a sudden arrest in the heating curve took place at 212 °F, presumably due to loosely held water being driven off. The second break occurred at 932 °F. He concluded that dehydration was invariably complete at 1112 °F. A slow liberation of heat between 1652 °F and 1832 °F was assumed to indicate the formation of an aluminate.

Rieke's⁽⁶⁴⁾ work showed that a similarity exists between kaolin and high-grade fire clays in that both show a distinct temperature lag at 932 °F and a longer halting period at from 1040 °F to 1076 °F. He found that some ferroginous clays behave similarly to fire clays while others show practically no halting point. The loss of chemical water begins only at 842 °F. The dissociation velocity increases rapidly with the temperature. By heating sufficiently long, practically all of the water may be expelled between 752 °F and 932 °F. The last portions of the chemical water are driven off with difficulty.

Searle⁽⁶⁸⁾ expressed his thoughts on the subject of the effects of heat on clays, probably from the experience of others as he does not mention having done any experimental work. He stated that (1) mechanically combined water is

driven off at 212 °F; (2) as the temperature is raised chemical decomposition occurs; and (3) at 932 °F to 1472 °F a quick evolution of water occurs. These conclusions are more or less in agreement with other investigators, but the basis for his second observation is not shown.

Boudouard and Lefranc⁽⁶⁾ further corroborated the temperatures at which water is given off in the heating of clays, and found that hygroscopic water is evolved continuously and slowly to 842 °F. Liberation of combined water started at 842 °F and was complete at 932 °F. Exception to this was noted in the case of a halloysite which showed the same loss. They investigated seven clays and two halloysites.

Expansion of Heating. Braesco⁽¹³⁾ measured the expansion and contraction of test pieces of clay as they were heated to 1832 °F by means of a telescopic apparatus located on the exterior of the furnace and through which he could view the test pieces as they were heated in the furnace. In all cases, there was first expansion followed by contraction. The temperature at which contraction began for glauconite was 1094 °F. This temperature, according to him, shows no relation to the temperature of dehydration of the aluminum silicates.

Biget⁽⁴⁾, in 1921, concluded that clays and kaolins expand before reaching their fusion temperature but bauxites do not. Mixtures of kaolin and silica show very marked swelling before fusion. This property may be utilized in preparing artificial pumice stone by rapidly heating schist and porcelain to their swelling temperatures.

Evolution of Heat. Wohlin⁽⁸⁰⁾, in 1914, attacked the problem from the standpoint of the temperature at which exothermic or endothermic reactions took place. He used twenty-nine different "clays, bauxites and allied materials." He increased the temperature of his furnace to 1310 °F during the first twenty-five minutes and from there to 2372 °F at the rate of 54 °F per minute for a total heating time of seventy-five minutes. He found that (1) from 1040 °F to 1076 °F clays showed heat absorption; (2) exactly at 1769 °F (at this rate of heating) heat was evolved more or less vigorously the longer the period of heat absorption; and (3) bauxite of the formula $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ showed heat absorption at 1004 °F, while bauxite of the formula $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ showed this at 590 °F. Both varieties often show a considerable evolution of heat due to a change in the form of the Al_2O_3 at 1940 °F. Wohlin⁽⁷⁸⁾ states that this does not occur in

the case of $\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ at 1940 °F, and because of this phenomenon, one can distinguish $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot 2\text{H}_2\text{O}$, $\text{Al}_2\text{O}_3 \cdot 4\text{H}_2\text{O}$, and $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ when present in sufficient amounts.

Houldsworth and Cobb⁽³⁰⁾, in 1925, observed that the changes responsible for the exothermic effects observed at 1742 °F with clays could be completed on continued heating at 1652 °F and that occurring with Al_2O_3 at 1940 °F could be completed at 1832 °F. These changes in clays and in pure Al_2O_3 are distinct and different phenomena. None of the indications of exothermic and endothermic reaction with clays can be attributed to the presence of small amounts of impurities and their possible interaction with the clay. No evolution of heat at 1742 °F was found when mixtures of Al_2O_3 and SiO_2 of the ratio $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ were heated.

Other Theories on Action of Heat. Mellor and Scott⁽⁴⁶⁾, in 1925, reached the conclusion that the dehydration of kaolinite does not take place at a fixed temperature and is completed at temperatures above 932 °F. At temperatures above 932 °F kaolinite decomposes into free silica, free alumina, and water. The exothermic break in the heating curve 1652 °F is associated with a transformation in the form of Al_2O_3 . Sillimanite, similar to the natural mineral,

can form below 2192 °F and probably above that temperature forms solid solutions with $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$. Above 3092 °F, $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ may be formed.

The research staff of the General Electric Company⁽¹⁹⁾, in 1926, attempted to follow by x-ray methods the 932 °F to 1652 °F changes in kaolinite. They found that kaolinite breaks down completely at about 1112 °F, forming a silicate which is stable to about 1652 °F. The silicate presumably is $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$. At higher temperatures mullite is formed. The x-ray patterns of natural sillimanite and mullite prepared by heating andalusite at 2822 °F for five hours are different. The pattern of mullite agrees with those obtained from fired clay mats. The mineral formed in clays at 1832 °F is mullite, not sillimanite. The formation of mullite occurs in all clays examined by heating them for five hours at 1922 °F.

Causes and Preventions of Bloating or
Swelling of Clays and Shales

The causes of bloating or swelling of clays and shales will be discussed under the headings of blistering of Cornish stone, shale and clays, and prevention of bloating.

Blistering of Cornish Stone. Calcott⁽¹⁵⁾, in 1912, investigated the blistering and bubbling of Cornish stone. He said that this phenomenon is found only in mixtures of buff and purple stone and is probably due to unequal fusibility of the two, giving rise to the evolution of gases from the buff stone (the less fusible of the two) after surface vitrification caused by the presence of the purple stone.

Bennet⁽³⁾, in 1941, made the following observation on bloating of Cornish stone. He said that bloating is caused by the expansion of entrapped air when ware is viscous as well as to the decomposition of gas-forming material. "Softer" fluxes cause bloating more than "harder" ones. Fineness of grinding has little effect on the bloating characteristics.

Shale and Clays. Orton and Staley⁽⁵⁴⁾ at the Ohio State University, in 1908, fired shale containing 5.42, 2.87, and 3.0 weight per cent iron, carbon, and sulphur,

respectively, to a number of temperatures and under three sets of conditions. These conditions were: (1) rapid oxidation and normal vitrification, (2) slow oxidation followed by normal vitrification and overfiring, and (3) normal oxidation followed by normal vitrification and heavy reduction without increases in temperature. The conclusions that were made are: (a) iron in clays tends to oxidize to Fe_2O_3 in the absence of carbon or sulphur; (b) the formation of ferrous silicate does not seem to cause the evolution of gases; and (c) sulfur in any form may give rise to the formation of gases at a high temperature by oxidation, dissociation, or replacement by SiO_2 . Since, in the unoxidized portion of the clay, the sulfur content will be less reduced by the action of the heat it will cause more evolution of gases which, in turn, will cause more swelling. Hence, the presence and action of sulfur is probably the most important cause of bloating. Clays high in carbon and iron but low in sulfur content blacken by improper burning but do not swell. Clays low in iron but high in carbon and sulfur content swell without blackening. Clays high in sulfur content but containing neither iron nor carbon, swell on the exterior first.

Begg⁽⁴¹⁾, in 1951, reported that any material used for bloating must satisfy two conditions. First, it must have a chemical composition with the proper balance of fluxing agents, silica, and alumina to allow it to produce a melt of viscosity high enough to trap a gas. This melt should occur at a temperature below 2400 °F for the material to be of any commercial use. Second, the material should contain some substance which either dissociates or reacts with the other constituents in such a manner that the gas is liberated at or above the fusion temperature.

Prevention of Bloating. Kallauner⁽³²⁾, in 1930, gave as the cause of bloating in ceramic products, when heated, the presence of entrapped gases, i.e., water vapor, air, oxygen, carbon dioxide, and sulfur dioxide. He said that in order to prevent bloating the following conditions should be used: (1) the raw materials should be washed; (2) excessive fatness should be removed; (3) carbonaceous substances should burn out at 932 °F; (4) firing should progress slowly; (5) the steam in the kiln space should be reduced to a minimum; (6) sufficient draft should be provided; (7) firing at 1472 to 1652 °F must be carried out in a reducing atmosphere to decompose the

sulphates; and (8) the excess air should be in such quantities that the carbonaceous substances may be burned.

Sheun⁽⁶⁶⁾, in 1937, gave as the cause of blistering in ceramics, (1) the entrainment of air in excessively fat or harsh clay; (2) the reaction of iron oxide and carbonaceous material; (3) the decomposition of iron oxide at high temperatures; (4) formation or liberation of water at high temperatures; and (5) overburning.

Methods of Bloating Clays and Shales

The methods that are used in bloating shales and clays will be discussed under the headings of additives that promote bloating and natural bloating clays and shales.

Additives that Promote Bloating. It has been stated⁽²⁷⁾ that good insulators for furnaces have been made by mixing sawdust, coal, and kieselguhr to clay and firing the mixture. Organic materials such as cut grass, oats, or other cereals, tan bark, coke dust, wood shavings, sawdust, and peat have been mixed with clay and fired for making porous products.

Meyer and Pukall⁽⁴⁷⁾ suggested procuring artificial porosity in ceramic bodies by a chemical reaction between

either the moisture in the plastic mass or constituents of the clay and finely divided metals which are added. These reactions cause the evolution of gases. Metals experimented with were aluminum, magnesium, and silicon.

Loginov⁽⁴²⁾ described the bloating of various Russian clays. Clay cylinders, ten to twenty millimeters in diameter, were prepared from shale crushed to 1/30 millimeter. The firing was done at 2284 °F. Swelling clinkering took place at 2102 to 2156 °F. Various additions to the clay were tried, including gas coal, two per cent; oil shale, five per cent; and swamp ore, four per cent. Swamp ore gave the best results, producing a product weighing 0.56 gram per cubic centimeter.

In 1940, Loginov⁽⁴³⁾ also wrote of preparing the product described and mentioned the additions of three per cent iron oxide and two per cent combustible agent, such as coal or peat, to the Russian clays before firing.

Natural Bloating Clays and Shales. Whitlatch⁽⁷⁹⁾ described the bloating characteristics of a West Tennessee clay with the view of its use for lightweight ceramic products or lightweight concrete aggregate. He stated that the clay fires naturally to exceptionally low densities. Between cones 1 and 2 (2020 and 2050 °F) the

apparent specific gravity ranges from 1.29 to 1.58 and the apparent porosity ranges around forty per cent. Additions to the clay shale of ten to twenty-five per cent lignitic clay gave products of 1.09 and 0.91 apparent specific gravities or sixty to sixty-seven pounds per cubic foot.

Lightweight Concrete Aggregate

In recent years a wide variety of lightweight materials, both natural and processed, have been introduced and marketed for use as lightweight concrete aggregate. The applications, the need for design information, and some of the characteristics of lightweight aggregate will be discussed in the following section.

Application of Lightweight Concrete. The use of lightweight concrete is not new, it having been used in the early days of the Roman Empire when pumice was used as a component of temple roof slabs. Harper⁽²⁸⁾, in writing of the construction of ships with lightweight aggregate during the first World War, said "It was decided to imitate the volcanic rock by baking a clay into a clinker in a kiln. A clay containing vegetable matter was selected. At a temperature of about 2000 °F the

vegetable matter converted to a gas inflating the clinker, leaving the disconnected isolated cells when cooled." Probably the most notable example of lightweight concrete aggregate application is the placing of "Gravelite", a lightweight concrete aggregate, in the upper deck of the San Francisco-Oakland Bay bridge in California, where a huge saving was attributed to the reduction of dead load. Another instance of interest was the addition of six floors to the Argyl building in Kansas City, Missouri, by using "Haydite", an expanded shale aggregate. Neff⁽⁴⁹⁾, of the Armour Research Foundation of the Illinois Institute of Technology in Chicago, reported that tiny glass balloons, about the size of grains of sand, formed from heated clay, will be used as a lightweight aggregate for concrete and plaster in place of ordinary sand or other fillers. This lightweight aggregate has been given the trade name "Kanamite." One of the most important features of this new product is that it has a high fluidity which means that for the first time in building history, contractors can fill forms with concrete pumped through rubber hose. Construction cost can be lowered because of the virtual replacement of shovels and awkward metal hoses now used.

Need for Design Information. With the expanding use of lightweight aggregate concrete a demand arises for information descriptive of its behavior. Architects, engineers, contractors, and builders, desiring to use lightweight concrete, require reliable design data as well as knowledge of characteristics which might govern the choice of material for a particular need. Existing building codes and regulations for natural aggregates are not applicable to lightweight aggregates. Recognizing this need for information has resulted in the publication, "Lightweight Aggregate Concrete", issued in 1949 by the Housing and Home Finance Agency in Washington, D. C. To date, this is the only publication which shows the wide variations that may be expected between different types of lightweight aggregates and also their different characteristics from those of sand and gravel.

Chemical Constituents. Klinefelter and Hamelin⁽³³⁾, reported that two or three impurities, or what might be called activating agents, needed for bloating are found invariably and seem to be essential for a good lightweight aggregate raw material. Iron is one of these agents, and typical bloaters contain it in quantities of three to ten

per cent. Since sulfur also seems necessary, iron disulfide probably forms at least a portion of the total iron content. This is a common form in iron-bearing clays and its dissociation at about 2000 °F explains much of the glassy-phase development accompanied with gas formation. Carbon is nearly always cited as essential, but that may be largely because it is invariably found in such clays and because it unites with oxygen to form gases. Lime and magnesium carbonates and sulfates as well as other minerals are nearly always present in small amounts and all must have some effect in the glassy-phase formation as well as the evolution of gas. The early investigators found that the carbon gases had little to do with the actual bloating but that most of the bloating could be attributed to sulfur gases. However, Austin⁽²⁾, in 1942, found a clay which would bloat and yet not give off any sulfur gases.

Conditions Necessary for Bloating. Riley⁽⁶⁶⁾ reported that any material used for bloating must satisfy two conditions. First, it must have a chemical composition with the proper balance of fluxing agents, silica, and alumina to allow it to produce a melt of viscosity high enough to trap a gas. This melt should occur at a temperature below 2400 °F

for the material to be of any commercial value. Second, the material should contain some substance which either dissociates or reacts with the other constituents in such a manner that a gas is liberated at or above the fusion temperature.

Formation of Small Cells During Firing. Cellular formation consists of the generating and entrapping of gases when the material is at or near the point of fusion. The first application of this process was made with molten blast-furnace slag, which was poured on water and manipulated so that steam entrapped to vesiculate the slag. The vesiculated material was then formed into shapes, generally by bonding with cements. A modification consisted of including in the slag a mixture which would generate gas at temperatures above the fusion temperature of the slag and which would be entrapped in the viscous molten mass.

The best known clay fire-expanded material is known as Haydite, so named after the inventor, Hayde. It is strictly a lightweight concrete aggregate. The process used in the manufacturing of these lightweight aggregates is dependent upon heating the material to a molten mass with a high viscosity and then entrapping the gases which are being evolved by the material at that temperature.

Commercial Lightweight Aggregates. In the following section some of the more important types of lightweight aggregates that are being made on a commercial scale will be discussed. These are shalite, haydite, lite-rock and kanamite.

Shalite. This lightweight aggregate is a bloated shale which is made by the Poston Brick and Concrete Company, Springfield, Illinois. This aggregate is made by feeding the raw shale into a rotary kiln in which the peak temperature at the bloating end of the furnace is held at 2050 °F. The shale is brought up to the bloating temperature in three and one-half hours, after which the fired material falls from the end of the kiln in large chunks of loosely-bonded clinkers, dropping into a deep clinker pit for cooling. A stream of water is continually played on the hot clinker to speed its cooling. This clinker is then pulverized by crushing equipment and shipped to concrete block manufacturers. When processed into a lightweight concrete block it weighs approximately 55 pounds per cubic foot.

Haydite⁽²⁹⁾. The lightweight aggregate haydite is manufactured by the Haydite Corporation which has plants in almost all of the states in the country. When a clay

containing a small per cent of carbonaceous material is heated rapidly, a core is formed which makes the brick swell; this defect is turned into a useful account by the Haydite Corporation. Dry shale is crushed and fed into a rotary kiln. Pulverized coal is used as the usual fuel. One flame is directed up the axis of the kiln, and another, strongly reducing, is arranged to strike close to the outlet at a suitable angle. At 1598 °F sintering occurs, and the material gathers into balls which swell when they reach the reducing flame, owing to the expansion of the carbon and sulfur gases. The balls are soft when they leave the kiln and flatten out on falling onto a conveyor, which carries them to a quenching pit. When the material is low in carbonaceous content the Haydite Corporation reinforces the clay with molasses or coal dust. The final product when processed into a lightweight concrete block weighs approximately 50 pounds per cubic foot.

Lite-Rock⁽⁷⁾. Lite-rock is material produced by crushing and burning a shale mined near Banks, Oregon. The mined shale is crushed to a definite size and is then fed into a rotary kiln at temperatures in excess

of 2200 °F. At these high temperatures melting begins and gases are evolved causing expansion of the softened shale by formation of innumerable cells. The outer surface becomes melted completely and upon cooling forms a coating over the inner cellular structure. The size of the crushed aggregate that is fed into the kiln is small enough so as to prevent the recrushing of the shale when it is discharged from the end of the kiln.

Kanamite. Kanamite is the most recent and probably the most important of all of the lightweight aggrates that have been processed on the commercial basis. The development of the lightweight aggregate was announced recently by Armour Research Foundation, Chicago⁽⁶³⁾. The new material consists of tiny hollow ceramic balloons made by "bloating" individual grains of clay in a special furnace. A suitable clay is ground to desired particle size in a hammer mill. The milled clay is screened to obtain the desired fraction, usually 48 to 80 mesh.

Clay particles are fed into a special gas burner by entraining them in a primary air stream delivered by a positive-pressure blower. The burner is mounted at the

top of a vertical cylindrical furnace. This furnace is thirty feet high with an inside diameter of three and a quarter feet.

The time of fall, from clay particle leaving the burner to expanded bubble reaching the bottom of the furnace, is estimated at 0.015 second. The product is picked up pneumatically from the conical furnace bottom and delivered to a bagging machine. The final product weighs approximately 20 pounds per cubic foot, and is chemically inert. Use of kanamite instead of sand in concrete mixes makes for a very fluid mix. This fluidity might mean that concrete can be pumped directly into forms through rubber hose.

Patents

The paragraphs below show a study of the patents obtained on the production of lightweight aggregate concrete.

Early Patents. The first United States Patent 235,961 found pertaining to porous clays was issued to Praetorius⁽⁵⁷⁾, of Breslau, Prussia, Germany, on December 28, 1880. His invention was the preparation of porous earthenware for use in "filtering, absorption, and various kinds of chemical treatment." He prepared the earthenware by mixing iron and lime-free clay with powdered charcoal, fluorspar and feldspar, shaping it to the desired form and firing it in a kiln. The charcoal burned out, resulting in the porous earthenware.

United States Patent 248,094, dated October 11, 1881, was issued to Gilman⁽²⁵⁾, of Eldora, Iowa. His invention was a process for making a porous, earthenware building material. Clay, resinous sawdust, and water were mixed thoroughly in a mill until plastic. The plastic mass was then extruded through iron or steel pipe from eight to twelve inches in diameter to form "logs" about six to twelve feet long. These logs were fired on a "slow steadily increasing fire" until the "vapors" and steam were driven off. The temperature was then raised rapidly until nearly white hot. This consumed the

sawdust and brought the clay to the first stages of vitrification. On cooling, the logs could be sawed into "planks", bored, grooved, planed, or carved with edged tools.

To Lenderoth⁽³⁹⁾, of Ontario, Canada, was issued United States Patent 426,642 on April 29, 1890. His process was essentially the same as the foregoing patents except that he claimed greater strength by the addition of sand to the clay. Also, he saturated the sawdust or other combustible matter with water before mixing.

The Hayde Patent. Probably the most important patent issued for this type of material was to Hayde⁽²⁹⁾, February 12, 1918, United States Patent 1,255,878. In his patent, Hayde specifically stated that clay or shale which was not suitable for making brick could be used even if it contained lime-forming materials or sand. He suggested crushing the material to about four inches and then burning it in a "rotary or other type" kiln at a temperature above 1500 °F preferably to 1700 °F, for about two hours. The clinker formed is allowed to cool slowly in air until it will not disintegrate when sprayed with water. He then suggested immersing the clinker in water to hydrate any lime that

might have been formed. The clinker is then crushed and sized and mixed with cement or other hydraulic material, molded into the desired form and allowed to harden.

Recent Related Patents. In 1918, United States Patent 1,314,752 was issued to Olsen⁽⁵²⁾. This was a patent whereby a mixture of coarse and fine pieces of shale, which had been burned but not clinkered, are used for an aggregate with portland cement to produce a light concrete or may be used like fuller's earth for refining sugar or oil.

A British patent, 132,247, was issued to Olsen⁽⁵³⁾, about the same time which specified shale for use in concrete after being sintered or subjected to a temperature of 2000 °F or over, without thoroughly melting.

Fallon⁽²¹⁾ was issued a patent, United States Patent 1,528,759, on March 10, 1925. His process was for forming porous aggregates for use in making lightweight concrete or other articles. A mass of clay is first fired in a kiln until a temperature of 800 °F is reached. The supply of air is then reduced and steam is passed through the bed of burning fuel into the clay to produce a reducing action thereon. Air is then again supplied to the clay to cause the mass to expand and thereby produce a porous material.

Glass⁽²⁶⁾ secured United States Patent 1,538,482, on March 19, 1925, for a process of producing a hard, cellular clay product. His process consisted of mixing clay or shale with finely divided fuel and burning the mass on a grate by forcing air up through the mass to support combustion.

On May 4, 1926, United States Patent 1,583,521 was issued to Boynton⁽¹²⁾, for a process of making vesicular products by subjecting shales or clays high in iron, sulfur, sodium, and potassium to heat causing them to swell. The expanded shale or clay is then crushed and screened for use as a lightweight concrete aggregate.

Bohlig⁽⁵⁾ obtained Swiss Patent 126,755 in 1927 for making argillaceous clinker by preparing thick layers of calcined clay mixed with bituminous dust and burning the mass to clinkers.

Kress⁽³⁷⁾ was issued United States Patent 1,741,574 on December 31, 1930, for a "method of forming cellular bodies which consists in heating a mixture of clay, bentonite, carbonaceous material, and flux sufficiently to form sealed cells."

United States Patent 1,818,101 was issued to Slidell⁽⁶⁸⁾ on August 11, 1931, for a cellular or bloated clay product. In the process, granular material is fed into a heat zone maintained at bloating temperatures. The material, while in granular form and in the heat zone, is subjected to fusion while confined. The bloated material is extruded under pressure due to its expansion.

Husain⁽³¹⁾ obtained United States Patent 1,897,667 on February 14, 1933, for a cellular fired clay product suitable for fireproofing building block. The product is prepared by generating and releasing a gas in a clay slip of fluid consistency by the interaction of an acid and a carbonate added to the slip.

United States Patent 1,903,821 was issued to Lee⁽³⁸⁾, on April 18, 1933, for a process for bloating earthy materials. In the process, a liquid containing molasses and ferrous sulphate in solution is added to a mass of earthy material which is then heated to first evaporate the solvent portion of the liquid and then cause bloating by fusing the earthy materials and liberating gases through the mass.

United States Patent 2,112,380, issued on March 29, 1938, to Price⁽⁵⁸⁾, describes a process in which siliceous materials having the desired porosity, or attaining it owing to the presence of carbonaceous matter, are mixed with commercial marl of higher melting point and the mixture is burned in a rotary kiln until the comminuted marl coats the surface of the siliceous materials and seals the pores.

McCoy⁽⁴⁴⁾ was issued United States Patent 2,456,207 on December 14, 1948, for a process in which waste slate is used to prepare a lightweight aggregate. The slate is crushed and screened to produce the desired gradation. Fusion between particles is prevented by mixing with the pre-sized slate 10 to 20 per cent of an anti-adhesion agent which has a substantially higher fusion temperature. The agent is screened from the expanded aggregate and reused.

Napier⁽⁵⁰⁾ obtained United States Patent 2,456,643 on December 21, 1948, for a process making a lightweight insulating material. The materials used were portland cement and bentonite. The insulating material prepared compares favorably with 85 per cent magnesia insulating products in density, strength, and hardness, and is superior in its ability to withstand high temperatures and resistance to abrasion and vibration.

III. EXPERIMENTAL

This section contains information related to the experimental phase of this investigation. Subjects discussed include purpose of investigation and plan of experimentation.

Purpose of Investigation

The purpose of this investigation was to determine the effect of time, temperature, and chemical composition on the bloating characteristics of MacCrady, Liberty Hall, Martinsburg, and Brallier shales obtained from deposits in the state of Virginia for the production of a lightweight concrete aggregate.

Plan of Experimentation

The plan of experimentation for this investigation included a review of the literature, obtaining shale samples, determination of bloating temperature of shale, effect of prolonged heating on the bloated shale, effect of chemical composition on bloating, preparation of test cylinders, and testing of the bloated shale.

Literature Review. A search of the literature was made for the purpose of ascertaining what has been done on the use of bloated shale as a lightweight aggregate concrete and to become familiar with the methods used by others for bloating shale.

Obtaining Shale Samples. All samples of shale that were used in this investigation were obtained from deposits located within the state of Virginia. The samples were obtained from road cuts and surface deposits. Figures 5 and 6, pages 50 and 52, show the exact location of these deposits from which shale samples were obtained.

Bloating Characteristics. The samples obtained from various locations throughout the state were exposed to temperatures between 70 and 2200 °F in a muffle furnace. The degree of bloating was determined on each sample by determining the bulk density of the sample before and after firing by means of a mercury volumeter.

Determination of the Bloating Temperature of Shale. To determine the bloating temperature of shale specimens, ten samples of each shale were placed in the furnace and the temperature raised to 2200 °F over a specified length of time. As the temperature increased, one sample was withdrawn

from the furnace at each of the following temperatures and its bulk density determined: 1500, 1750, 1850, 1900, 1950, 2000, 2050, 2100, and 2200 °F. One sample was used to obtain the bulk density at 70 °F.

Effect of Prolonged Heating on the Bloated Shale. To determine the effect of prolonged heating on the bloating characteristics of the shale, samples of fired shale were placed in the muffle furnace at a temperature where good bloating occurred as previously determined. After this temperature was constant, a sample was removed every five minutes and its bulk density determined. Thus, the effect of exposing the shale to a particular temperature for extended periods of time was determined.

Effect of Chemical Composition on Bloating. The chemical composition of a poor bloating shale and a good bloating shale was determined and the results were compared in an attempt to determine which element or compounds were responsible for bloating.

Preparation of Load-bearing Test Cylinders. A mixture of non-weathered Liberty Hall and Martinsburg shales was used as the aggregate in the preparation of the load-bearing

test cylinders. The above mixture was carefully graded into eight size ranges as shown in Table X, page 84. The ratio of weight of aggregate to weight of cement was three to one. The water to cement ratio was eight-tenths to one by weight. Additional load-bearing test cylinders were made using cinders as aggregate so that a comparison could be made between both types of aggregate.

Testing of the Bloated Shale. Load-bearing tests were made on the bloated shale and cinder aggregate test cylinders. Also, water-cement ratio, molding method, and method of boring were compared for the two different types of test cylinders.

Materials

Information relating to the source and description of all materials used in this investigation is presented in the following paragraphs:

Acid, Hydrochloric. C. P., lot No 10921J. Distributed by Fisher Scientific Co., Silver Spring, Md. Used in the analysis of shale samples.

Acid, Hydrofluoric. C. P., lot No 10715, 52 to 55 per cent HF. Manufactured by Merck and Co., Inc., Rahway, N. J. Used in the determination of silica.

Acid, Sulfuric. C. P., lot No 91946. Manufactured by General Chemical Co., New York, N. Y. Used in the analysis of the shale samples.

Ammonium Carbonate. Powder, lot No 38. Manufactured by General Chemical Co., New York, N. Y. Used to flux the shale samples for the alkali determination.

Ammonium Chloride. Lot No 12311/1. Manufactured by General Chemical Co., New York, N. Y. Used for the precipitation of the calcium in the alkali determination.

Ammonium Hydroxide. Reagent grade of specific gravity 0.90, code 1239, lot No 6133, having an NH_3 content of 28 per cent. Manufactured by the General Chemical Co., New York, N. Y. Used for the precipitation of iron and aluminum hydroxide.

Ammonium Oxalate. Lot No 9340. Manufactured by the J. T. Baker Chemical Co., Phillipsburg, N. J. Used for the precipitation of calcium in the calcium determination.

Calcium Carbonate. Powder, lot No 8343. Manufactured by General Chemical Co., New York, N. Y. Used with ammonium chloride in sintering the shale sample for the alkali determination.

Disodium Hydrogen Phosphate. Lot No 7914, control WRVT. Manufactured by Mallinckrodt Chemical Works, Philadelphia, Pa. Used to precipitate magnesium in the magnesium determination.

Ferrous Ammonium Sulphate. Lot No 9340. Manufactured by the J. T. Baker Chemical Co., Phillipsburg, N. J. Used to standardize the potassium permanganate solution in the determination of iron.

Methyl Orange. Lot No 1111239. Manufactured by General Chemical Co., New York, N. Y. Used as an indicator in the neutralization of the solution from which iron and aluminum were precipitated as the hydroxides.

Potassium Bisulphate. Lot No 40647. Manufactured by Merck and Company, Inc., Rahway, N. J. Used to flux the iron and aluminum oxides preparatory to the determination of iron.

Potassium Ferrocyanide. Lot No 29. Manufactured by General Chemical Co., New York, N. Y. Used in a 0.1N solution in the titration of iron.

Shale Samples. The samples of shale used in this investigation were obtained from deposits located in the state of Virginia. Four different types of shale were used during the experimental work on this thesis. The types of shale used were Liberty Hall, Brallier, Martinsburg, and MacCrady.

Liberty Hall. The exact location of the Liberty Hall shale used in this investigation is shown in Figure 5, page 50. Two forms of this shale, weathered and non-weathered, were obtained. The weathered shale was taken from the surface of a three-foot road cut, while the non-weathered shale was obtained from a depth of approximately one foot below the surface of the road cut. The samples weighed between 10 and 20 pounds and had a black color.

Brallier. The exact location of the non-weathered Brallier shale used in this investigation is shown in Figure 5, page 50. The sample was obtained by digging to a depth of approximately two feet in order to remove the clay and dirt which covered the deposit. No

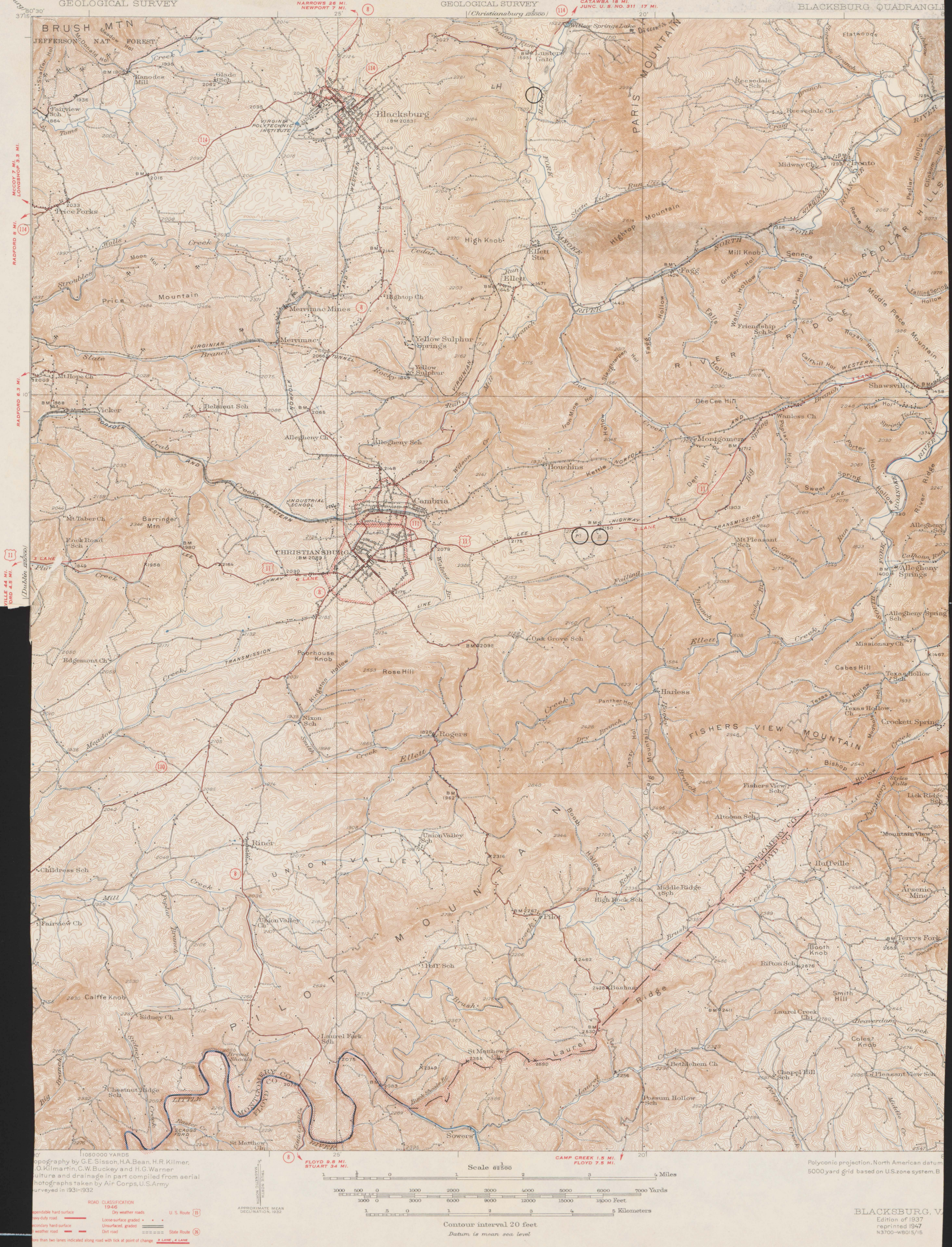


Figure 5. Topographical Map Showing the Location of Liberty Hall, Martinsburg and Brallier Shales

Liberty Hall Shale: $37^{\circ} 14' N, 80^{\circ} 22' W$

Martinsburg Shale: $37^{\circ} 8' N, 80^{\circ} 21' 10'' W$

Brallier Shale: $37^{\circ} 8' N, 80^{\circ} 20' 50'' W$

weathered Brallier shale was found in this deposit. The shale sample had grey color and weighed approximately 30 pounds.

MacCraday. The MacCraday shale that was used in this investigation consisted of two forms, weathered and non-weathered, the location of which is shown in Figure 6, page 52. The weathered shale was obtained from the face of the fifteen-foot road cut deposit and the non-weathered MacCraday shale was taken from a depth of 12 to 18 inches below the surface of the road cut. Both forms of the shale had a brownish-grey color, and each sample weighed approximately 25 pounds.

Martinsburg. The exact location of the non-weathered Martinsburg shale that was used during this investigation is shown in Figure 5, page 50. This sample was obtained by digging to a depth of two feet and removing the shale. The sample removed weighed approximately 25 pounds and had a yellowish-brown color. No weathered Martinsburg shale was found in this deposit.



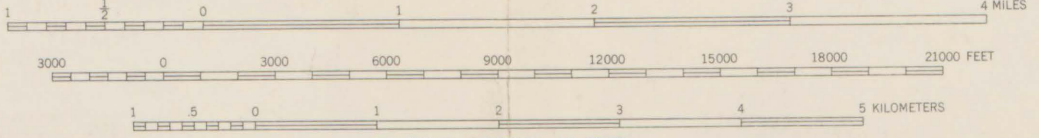
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Topography from aerial photographs by multiplex methods
Aerial photographs taken 1947. Field check 1951

Polyconic projection. 1927 North American datum
10,000-foot grids based on Virginia and West Virginia
coordinate systems, south zones

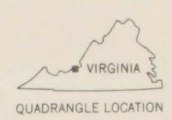
TRUE NORTH
MAGNETIC NORTH
APPROXIMATE MEAN
DECLINATION, 1951

SCALE 1:62,500



CONTOUR INTERVAL 40 FEET
DATUM IS MEAN SEA LEVEL

ROAD CLASSIFICATION
Heavy-duty 4 LANE 6 LANE Light-duty
Medium-duty 4 LANE 6 LANE Unimproved dirt
U. S. Route State Route



WAITEVILLE, VA.-W. VA.
N3715-W8015/15

1951

THIS MAP COMPLIES WITH NATIONAL MAP ACCURACY STANDARDS
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Silver Nitrate. Lot No 11239. Manufactured by General Chemical Co., New York, N. Y. Used to indicate the presence or freedom of chlorides in filtrate washings.

Sodium Carbonate. Anhydrous, granular, lot No 1114. Manufactured by General Chemical Co., New York, N. Y. Used in the fusion of the sample in the determination of silica.

Sodium Chloride. C. P., lot No 514018. Distributed by Fisher Scientific Co., Silver Spring, Md. Used throughout the chemical analysis.

Apparatus

This section contains detailed descriptions of all the equipment used in the course of this investigation:

Balance. Analytical, Chainomatic, capacity 100 gm, graduation 0.0001 gm. Manufactured by Seoderer-Kohlbusch, Inc., Newark, N. J. Used to weigh samples for the chemical analysis of the shale.

Balance. Beam-type, capacity 1600 gm, 1.0 gm increments. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to weigh the shale samples before bloating.

Ceramic Blocks. Ceramic blocks 4 inches long by 4 inches wide by 3/4-inch thick. Obtained from the Ceramic Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used in the furnace as platforms to place the samples of shale on while firing.

Crucibles. Platinum, capacity 20 ml, 33 mm in diameter, 33 mm high. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used in the silica determination.

Crusher. Blake-type with 6-inch corrugated jaws. Manufactured by the Universal Crusher Co., Cedar Rapids, Iowa. Used to reduce size of shale.

Drying Oven. Model number OV-8, serial number 8-270, 110 v, 60 cy, ac, 5 amp. Manufactured by Modern Electric Laboratory, Chicago, Ill. Used in chemical analysis.

Furnace. Hoskins electric muffle, heavy duty, model FH202, 220 v, 60 cy, ac, operating temperature 2000 °F, inside dimensions 10 x 4 x 3 inches, catalog No 10-526. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to bloat the shale samples.

Glassware. Miscellaneous beakers, flasks, burets, evaporating dishes, etc. Used in chemical analysis.

Molds. Cast iron, splitmolds, 6 inches inside diameter, 12 inches high. Manufactured by Walker Machine and Foundry Co., Roanoke, Va. Used to mold the concrete test cylinders.

Pyrometer. Optical, disappearing filament type with potentiometer, model 8621, two range scales, one from 1400 to 2200 °F and the other 1950 to 3200 °F, increments of 5 °F. Manufactured by Leeds and Northrup Co., Philadelphia, Pa. Used to measure temperature in the muffle furnace.

Pyrometric Cones. Research size, 1-1/8 inches long, numbers 020 (1202 °F), 015 (1481 °F), 07 (1814 °F), 03 (2039 °F), and 11 (2417 °F). Manufactured by Edward Orton, Jr., Ceramic Foundation, Columbus, Ohio. Used to verify optical pyrometer and thermocouple readings.

Rheostat. Alternating current, 60 cy, 220 v, for use with FH202 furnace, catalog No 10-529. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to control furnace temperature.

Scales. Ohaus, capacity 30 kilograms, one-gram increments, 22-inch double beam. Manufactured by the Newark Scale Co., Newark, N. J. Used to weigh large shale samples.

Screens. Tyler standard, mesh numbers 3/8 inch, 2, 3, 4, 14, 28, 48, 100, and 200 mesh. Manufactured by the Tyler Screen Co., Cleveland, Ohio. Used to grade cinders and bloated shale samples.

Testing Machine. Hydraulic compression testing machine, capacity 400,000 lb, located in the Materials Testing Laboratory of Virginia Polytechnic Institute, Blacksburg, Va. Manufactured by the Riehle Testing Machines Division of the American Machine and Metals Co., Inc., East Moline, Ill. Used to test concrete test cylinders.

Thermocouple. An alumel-chromel thermocouple having a range from 100 to 2200 °F. Manufactured by the Weston Electric Instrument Co., Newark, N. J. Used to measure temperature of muffle furnace.

Transformer. Alternating current, 60 cy, 220 v, accessory to FH202 furnace, catalog No 10-529. Obtained from Fisher Scientific Co., Silver Spring, Md.

Volumeter. Speil-Wilson type, mercury balance volumeter, consisting of an Ohaus double beam balance, capacity 20 kilograms, 1.0-gram sensitivity, to which is attached a steel wire cage for immersing and weighing sample in mercury. Volumeter assembled in the shop of the Industrial Engineering Department and located in the Ceramic Laboratory, Ceramic Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used to determine volume of shale samples.

Methods of Procedure

The methods of procedure used in this investigation were as follows:

Calibration of Thermocouple. A chromel-alumel thermocouple was first used to measure the temperature of the muffle furnace. Before the thermocouple was used, it was calibrated against the melting points of tin, lead, and antimony.

Calibration of Pyrometer. A Hoskins chromel-alumel pyrometer was used to check the temperatures of the muffle furnace. Before the pyrometer was used, it was calibrated for temperatures above red heat. The welded or hot end of the pyrometer was covered with a tight winding of pure copper wire, melting point 1949.0 °F, Number 14 B. & S. gage standard melting point metal. The pyrometer was then inserted into a muffle furnace, with the welded end of the pyrometer approximately in the middle of the furnace. The pointer of the attached meter was then corrected to the melting point of the copper wire. This procedure was used again with pure zinc wire as a check. The melting point of zinc wire is 786.0 °F.

)

Determination of the Temperature Lag in the Muffle

Furnace. The temperature lag in the muffle furnace was determined by placing a chromel-alumel thermocouple at the back end of the muffle furnace and moving it forward in one-inch increments along an axis approximately in the middle of the furnace. The distance between the thermocouple head and the back end of the furnace together with the indicated temperature was recorded for each one-inch increment.

Code for Identification of Shale Samples. All shale samples were identified by two letters and a number; the first letter indicated the type of shale, the second letter indicated the condition of the shale and the number indicated the sample number. Thus, Mc-N-1 represents MacCrady shale, non-weathered, sample number one; L-W-3 indicates Liberty Hall shale, weathered, sample number three.

Preparation of Shale Samples. Both weathered and non-weathered samples of shale were used in the tests and the preparation was the same for each. The shale samples were washed with water and cleaned of all extraneous matter with a wire brush. These samples were then broken from the large lumps, approximately two inches in diameter, into small

pieces approximately one-half inch in diameter, in an iron mortar with a pestle.

Determination of Firing Schedule. The rate of heating or firing schedule that was used throughout this investigation was determined by subjecting the four types of shale used to various rates of heating in the muffle furnace until a firing schedule was obtained that could be used in bloating the shale. This firing schedule was then duplicated for all tests.

Procedure for Determining the Bloating Characteristics for Different Samples of Shale. Samples of Liberty Hall, non-weathered and weathered; MacCrady, weathered and non-weathered; Brallier and Martinsburg, non-weathered shale weighing approximately twenty-five grams were broken to approximately 12-mesh in an iron mortar with a pestle. The samples were carefully cleaned of extraneous matter with a wire brush. The samples were then weighed in air on a beam balance. After being weighed in air, the samples were then weighed suspended in mercury. The bulk densities of the samples were calculated and recorded.

The weighed samples were then placed on refractory slabs and placed in the muffle furnace. The furnace was started and

the temperature was raised from 70 to 2200 °F over a period of two hours according to a predetermined firing schedule. Temperature readings were taken for every 50 °F rise in temperature and the time recorded. Up to a temperature of 1500 °F, the alumel-chromel thermocouple was used to indicate the temperature. Beginning at 1500 °F, readings were also taken with the optical pyrometer as a check. Standard pyrometric cones were placed in the four corners of the muffle furnace to indicate the rate of heating. These cones were 020 (1202 °F), 015 (1481 °F), 07 (1814 °F), 03 (2039 °F), and 11 (2417 °F). Thus, three temperature indicating devices were used as a check on temperature.

When the temperature of the furnace had reached 2200 °F, the samples were removed from the furnace, cooled to ambient temperature, and weighed in air. The bulk densities of the burned samples were determined by dividing the volume of the sample as determined on the Speil-Wilson volumeter into the weight in air of the sample. Since the bulk densities of the samples were known both before and after burning, a comparison of the amount of bloating for the various samples could be made.

Samples subjected to this test were designated as explained on page 59. The locations of the shale deposits are shown in Figure 5, page 50 and Figure 6, page 52. The following samples were treated according to the foregoing procedure: L-N-1-2-3-4-5, L-W-1-2-3-4-5, B-N-1-2-3-4-5, Mc-N-1-2-3-4-5, Mc-W-N-1-2-3-4-5, and M-N-1-2-3-4-5. The numbers one through five were used in order that the five samples of each type of shale could easily be identified.

Procedure for Determining the Bloating Temperature of Shale. Four tests were made to determine the temperature at which bloating occurred. In these tests only one type of shale was tested each time. The shale designated as L-N, Liberty Hall non-weathered shale, was used for the first test; shale L-W, Liberty Hall weathered shale, was used for the second test; shale M-N, Martinsburg non-weathered shale, was used for the third test; and shale designated as B-N, Brallier non-weathered shale, was used for the fourth test. Forty 25.0 ± 1.0 -gram samples were prepared and weighed for four tests. The samples of each type of shale were numbered from one to ten. Sample number one was not fired but was used to determine the bulk density of the shale by means of a mercury volumeter at 70 °F.

The remaining nine samples were placed in the furnace on individual refractory slabs. The furnace was then adjusted so as to reach a temperature of 2200 °F in two hours. Samples were removed from the furnace as the furnace reached the indicated temperature, as shown in Table I, page 64, and allowed to cool in air to ambient temperature. They were weighed in air and then in mercury, from which data their bulk densities were calculated. By comparing the bulk densities of the samples removed at the various temperatures, the temperature at which the major part of the bloating occurred could be ascertained.

Procedure for Determining the Time of Exposure to a Particular Temperature on the Bloating Characteristics of Shale. Eleven 25.0 ± 1.0 -gram samples of Liberty Hall, non-weathered shale were prepared for firing. These samples were thoroughly cleaned and numbered according to their position in the muffle furnace, from one through eleven. The temperature of the muffle furnace was raised to 2050 °F over a period of two hours. The furnace was then adjusted so as to maintain this temperature throughout the tests. One sample was removed every five minutes. Thus, sample number one was removed when the temperature reached 2050 °F; sample number two was removed

TABLE I

Procedure Used in Removing Shale Samples from Furnace
During Determination of Bloating Temperature

Sample No	Temperature at Which Sample Removed From Furnace °F
1 ^a	--
2	1500
3	1750
4	1850
5	1900
6	1950
7	2000
8	2050
9	2100
10	2200

^a Sample number one was not fired but was used to determine the bulk density of the shale at 70 °F.

five minutes later; sample number three was removed ten minutes later, and so on, until sample number eleven was removed fifty minutes after the temperature reached 2050 °F. The samples were cooled in air to ambient temperature and weighed, and their bulk densities determined by means of the mercury volumeter. By comparing the bulk densities of the samples removed from the furnace after various times of exposure to 2200 °F, the effect of the time of exposure on bloating could be determined.

Procedure for Determining the Effect of Rapid Heating on the Bloating Characteristics of Shale. For this test, non-weathered Liberty Hall shale was selected since it already had shown good bloating properties. It was decided to use two furnaces for this test rather than just one because it was feared that the thermal shock of injecting the unburned shale into a furnace at 2050 °F would cause disintegration and loss of the sample. Accordingly, two electric muffle furnaces were used. In one furnace a constant temperature of 1600 °F was maintained and in the other a constant temperature of 2050 °F was maintained.

Nine samples of non-weathered Liberty Hall shale were prepared and numbered one through nine. Samples four, five, and six were placed in one of the furnaces and the temperature

was raised from 70 to 2050 °F in a period of one hour and forty-five minutes, thereafter immediately removed. Samples one, two, and three were placed in the other furnace which had previously been brought to a temperature of 1600 °F. The samples were allowed to remain in this furnace at 1600 °F for ten minutes and were then immediately transferred to the furnace maintained at 2050 °F, and allowed to remain for ten minutes at this temperature. In this way, samples one, two, and three were brought from 70 to 2050 °F in twenty minutes, while samples four, five, and six were brought from 70 to 2050 °F in one hour and forty-five minutes. The bulk densities were determined for all six samples and a comparison made to determine the effect of quick heating on the bloating characteristics of the shale. Samples seven, eight, and nine were not fired but were used to determine the bulk density of the unburned non-weathered Liberty Hall shale. This was done with a mercury volumeter.

Chemical Analyses. Chemical analyses were made on non-weathered, burned and unburned, Liberty Hall shale, and also on non-weathered, unburned, MacCrady Shale. The analyses included determination of loss on drying at 212 °F, loss on temperature ignition at 800 to 900 °F, loss on high

temperature ignition at 1700 to 1800 °F, silica, combined iron and aluminum oxides, iron oxide, (aluminum oxide by difference), calcium, magnesium, and combined alkalies.

The samples selected were chosen because the Liberty Hall shale showed excellent bloating qualities and the MacCraday shale exhibited rather poor bloating qualities. Thus, by comparing the chemical analyses of the two shales, it was thought that the component or components responsible for the bloating could be determined.

The samples were prepared for the analyses by selecting samples of 25.0 ± 1.0 grams and grinding them in a mortar until the entire sample would pass through a 200-mesh Tyler standard screen. Standard methods of analyses were used throughout (56).

Comparison of Crushing Strength of Concrete Made with Cinder Aggregate and Concrete made with Expanded Shale Aggregate. Commercial cinders, as used by Concrete Products Company, Incorporated, Christiansburg, Virginia, were used for making the cinder concrete. Expanded shale, consisting of a mixture of non-weathered Liberty Hall and Martinsburg shale, was used for the expanded shale aggregate. The aggregates were carefully graded into eight size ranges.

These size ranges and the percentages of the total aggregate in each range are shown in Table X, page 84. Tyler standard screens were used for grading the aggregate.

The ratio of weight of aggregate to weight of cement was three to one in both instances. The water to cement ratio was eight-tenths to one by weight. Both mixes were prepared in as much the same manner as was possible by hand mixing. The mixed concrete was molded into six-inch diameter by twelve-inch deep cylinders by filling the mold half full and tamping fifty times with a ten-pound tamping rod; then filling to three-quarters full and tamping 100 times; then filling the mold and tamping seventy-five times; then filling the mold a second time and tamping fifty times; filling the mold a third time and tamping fifty times; and finally striking the top off with a smooth plasterer's trowel.

The cylinders were placed in a curing autoclave in which the steam pressure was maintained at 150 pounds per square inch, gage for a period of eight hours⁽⁶³⁾. The cylinders were then removed from the curing autoclave and capped with plaster of Paris for testing. The cylinders were tested in the compression testing machine of the

Materials Testing Laboratory of Virginia Polytechnic Institute
twenty-four hours after they were molded.

Data and Results

The experimental data and results obtained during this investigation are given in tabular form.

Firing Schedule. Data obtained for the firing schedule used to bloat the shale used during this investigation are presented in Table II and Figure 7, pages 71 and 72, respectively.

Bulk Densities of Unburned Shale. The data and results obtained for the bulk densities of the unburned shale are presented in Table III, page 73.

Bulk Densities of Burned Shale. The data and results obtained for the bulk densities of the burned shale are presented in Table IV, page 74.

Rank of Bloating. The rank of bloating and the ratios of unburned to burned bulk densities are presented in Table V, page 75.

Bloating Temperature. Data used for determining the temperature at which bloating occurs are presented in Table VI, page 76, and Figures 8, 9, 10, and 11, pages 77, 78, 79, and 80, respectively.

Time of Exposure. Data used for determining the effect of time exposure on the bloating qualities are presented in Table VII, page 81.

Rapid Heating. The results obtained by rapid heating of the shale are presented in Table VIII, page 82.

Chemical Analysis. The results of the chemical analyses that were performed during this investigation are presented in Table IX, page 83.

Preparing Test Cylinders. The data recorded while preparing the cinder and shale concrete cylinders are presented in Table X, page 84.

Compressive Strength. The data and results of the compressive strengths of the shale and cinder concrete test cylinders are presented in Table XI, page 85.

TABLE II

Data Obtained for the Firing Schedule Used to Bloat the
Shale Used During the Course of this Investigation

Time	Thermocouple Temperature	Pyrometer Temperature
min	°F	°F
4.0	500	500
5.0	650	650
6.0	800	800
7.5	900	900
9.0	1000	1000
11.0	1100	1100
14.0	1200	1200
16.5	1300	1300
21.0	1400	1400
25.0	1500	1520
30.0	1600	1650
37.0	1700	1760
46.0	1800	1850
57.0	1850	1960
71.5	2000	2100
96.0	2100	2200

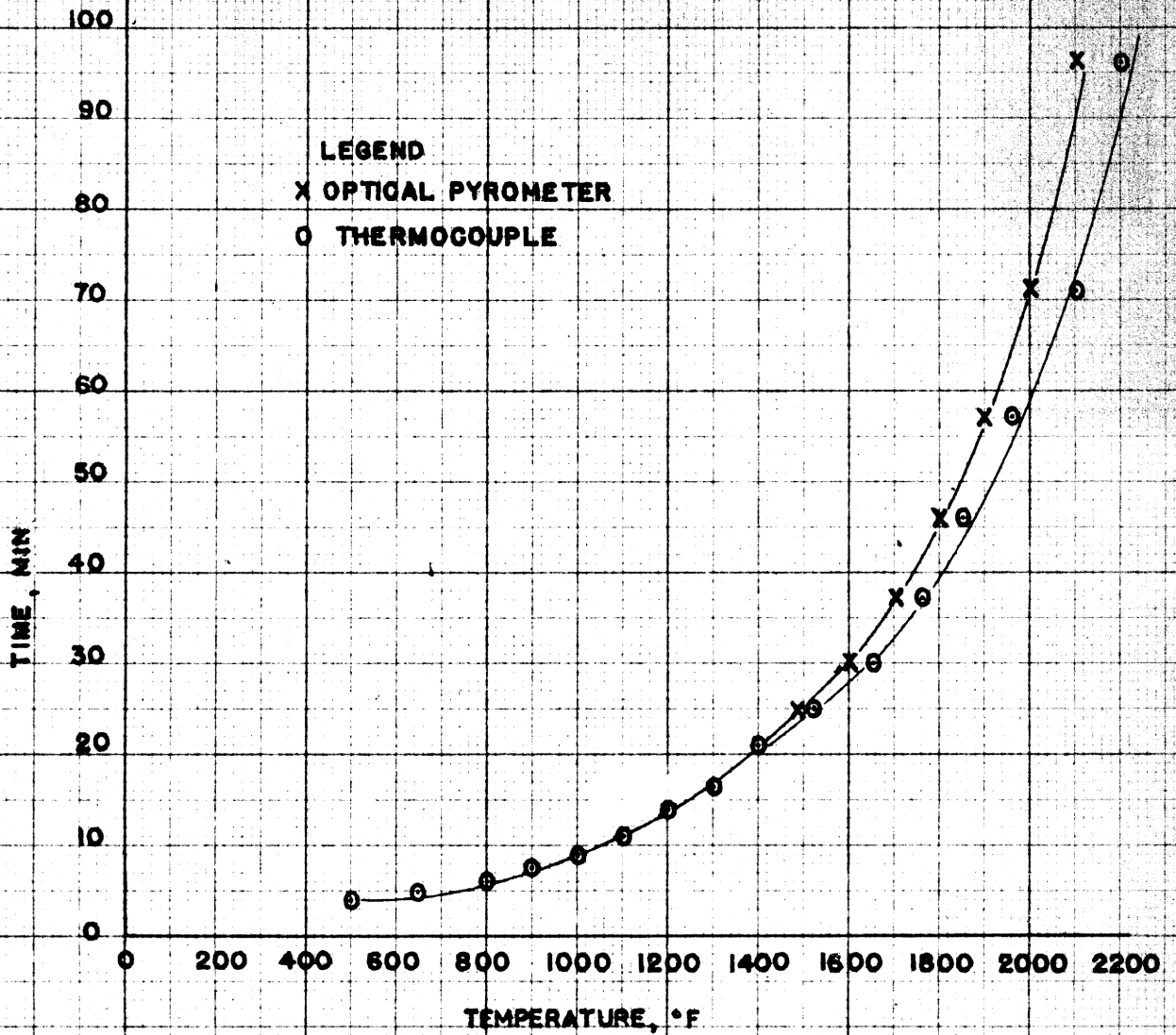


FIGURE 7. FIRING SCHEDULE USED TO BLOAT THE SHALE CONSIDERED IN THIS INVESTIGATION

TABLE III

Data Used for Calculation of Bulk Densities of Unburned Shale
Samples from Various Deposits Located in Virginia^a

Sample Number ^b	Weight in Air gm	Weight in Mercury gm	Volume of Sample cc	Bulk Density gm/cc
L-N-1	25	521	10.66	2.356
L-N-2	25	525	10.77	2.323
L-N-3	25	531	10.89	2.302
L-N-4	25	522	10.68	2.349
L-N-5	25	517	10.64	2.354
L-W-1	25	478	9.94	2.523
L-W-2	25	501	10.33	2.427
L-W-3	25	483	10.01	2.494
L-W-4	25	493	10.20	2.454
L-W-5	25	480	9.95	2.516
B-N-1	25	513	10.50	2.383
B-N-2	25	501	10.33	2.422
B-N-3	25	495	10.22	2.448
B-N-4	25	511	10.49	2.394
B-N-5	25	521	10.65	2.355
Mc-N-1	25	531	10.89	2.301
Mc-N-2	25	547	11.10	2.253
Mc-N-3	25	539	10.95	2.388
Mc-N-4	25	549	11.14	2.249
Mc-N-5	25	533	10.85	2.307
Mc-W-1	25	524	10.81	2.315
Mc-W-2	25	531	10.89	2.303
Mc-W-3	25	527	10.75	2.323
Mc-W-4	25	522	10.68	2.344
Mc-W-5	25	519	10.12	2.475
M-N-1	25	522	10.68	2.347
M-N-2	25	527	10.75	2.327
M-N-3	25	516	10.59	2.368
M-N-4	25	530	10.86	2.300
M-N-5	25	525	10.77	2.321

^a The location of shale deposits are shown in Figure 5, page 50, and Figure 6, page 52.

^b See code on page 59.

TABLE IV

Data Used for Calculation of Bulk Densities of Burned Shale^a
Samples from Various Deposits Located in Virginia^b

Sample Number ^c	Weight in Air	Weight in Mercury	Volume of Sample	Bulk Density
	gm	gm	cc	gm/cc
L-N-1	24.6	1625	29.33	0.844
L-N-2	23.4	1262	23.12	1.015
L-N-3	23.5	1960	34.84	0.675
L-N-4	24.0	2510	44.20	0.544
L-N-5	24.1	1158	21.32	1.130
L-W-1	23.2	807	15.35	1.515
L-W-2	22.8	624	12.21	1.866
L-W-3	22.9	595	11.75	1.950
L-W-4	22.4	796	15.09	1.484
L-W-5	22.6	690	13.24	1.705
B-N-1	24.6	526	10.72	2.291
B-N-2	25.0	501	10.33	2.422
B-N-3	24.8	505	10.38	2.389
B-N-4	25.0	511	10.49	2.394
B-N-5	25.0	521	10.65	2.355
Mc-N-1	25.0	531	10.89	2.301
Mc-N-2	25.0	547	11.10	2.253
Mc-N-3	25.0	539	10.95	2.288
Mc-N-4	25.0	549	11.14	2.249
Mc-N-5	25.0	533	10.85	2.307
Mc-W-1	25.0	524	10.81	2.315
Mc-W-2	25.0	531	10.89	2.303
Mc-W-3	25.0	527	10.75	2.323
Mc-W-4	25.0	522	10.68	2.344
Mc-W-5	25.0	519	10.12	2.475
M-N-1	22.8	1428	25.80	0.885
M-N-2	23.1	1191	21.90	1.055
M-N-3	23.6	1150	21.22	1.110
M-N-4	23.9	1414	25.65	0.932
M-N-5	22.6	1487	25.78	0.878

^a Temperature of bloating 2000 °F.

^b Location of shale deposits are shown in Figure 5, page 50, and Figure 6, page 52.

^c See code on page 59.

TABLE V

Rank of Bloating and Ratios of Bulk Densities of Unburned to Burned Shale Samples from Various Deposits in Virginia^a

Rank of Bloating ^b	Sample Number ^c	Bulk Density Unburned gm/cc	Bulk Density Burned gm/cc	Ratio of Unburned to Burned Bulk Density
1	L-N-4	2.349	0.544	4.320
2	L-N-3	2.302	0.675	3.410
3	L-N-1	2.356	0.844	2.790
4	M-N-1	2.347	0.885	2.660
5	M-N-5	2.321	0.878	2.647
6	M-N-4	2.300	0.932	2.470
7	L-N-2	2.323	1.015	2.288
8	M-N-2	2.327	1.055	2.209
9	M-N-3	2.368	1.110	2.130
10	L-N-5	2.354	1.130	2.080
11	L-W-1	2.523	1.515	1.664
12	L-W-4	2.454	1.484	1.651
13	L-W-5	2.516	1.705	1.472
14	L-W-2	2.427	1.866	1.305
15	L-W-3	2.494	1.950	1.278
16	B-N-1	2.383	2.291	1.040
17	B-N-3	2.448	2.389	1.024
18 ^d	--	--	--	1.000

^a Location of shale deposits are shown in Figure 5, page 50, and Figure 6, page 52.

^b Temperature of bloating 2000 °F.

^c See code on page 59.

^d Samples B-N-2-4-5, and all MacCrady shale did not bloat at 2000 °F.

TABLE VI

Data Used for Determining the Temperature at Which Bloating
Occurs for Liberty Hall, Martinsburg and Brallier Shales

Sample Number	Temperature of Furnace °F	Bulk Density			
		Liberty Hall Shale ^a gm/cc	Liberty Hall Shale ^b gm/cc	Martinsburg Shale ^a gm/cc	Brallier Shale ^a gm/cc
1	70	2.36	2.47	2.32	2.39
2	1500	2.22	2.41	2.28	2.31
3	1750	2.38	2.40	2.28	2.30
4	1850	2.23	2.38	2.30	2.26
5	1900	2.30	2.39	2.14	2.22
6	1950	2.10	2.30	2.22	2.20
7	2000	2.04	2.24	2.24	2.17
8	2050	1.74	2.20	2.04	2.08
9	2100	1.40	1.63	1.78	1.98
10	2200	0.84	1.19	0.99	1.80

^a Indicates non-weathered shale.

^b Indicates weathered shale.

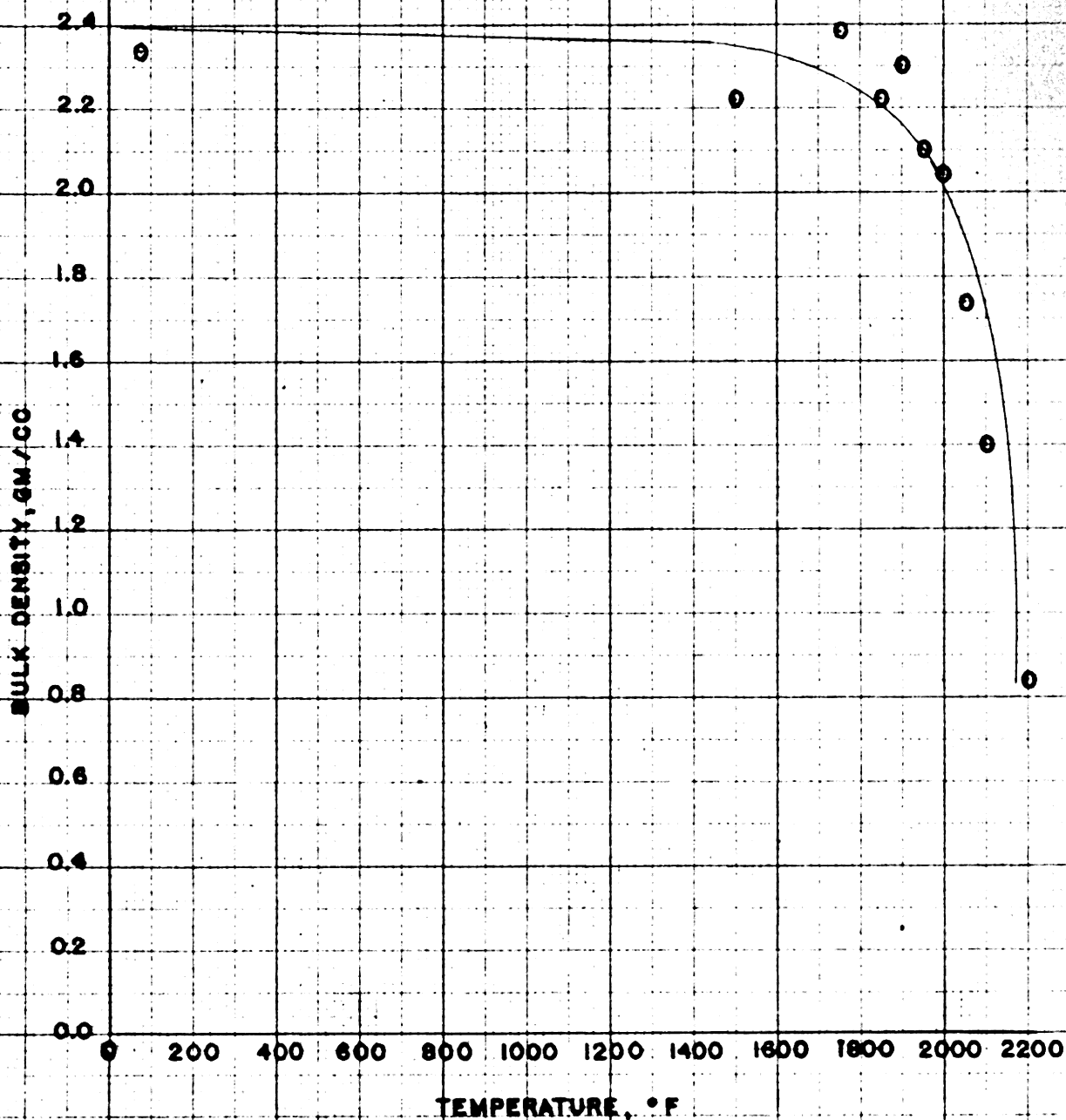


FIGURE 8. RELATIONSHIP OF THE TEMPERATURE AT WHICH NON-WEATHERED LIBERTY HALL SHALE WAS REMOVED FROM THE FURNACE TO ITS BULK DENSITY

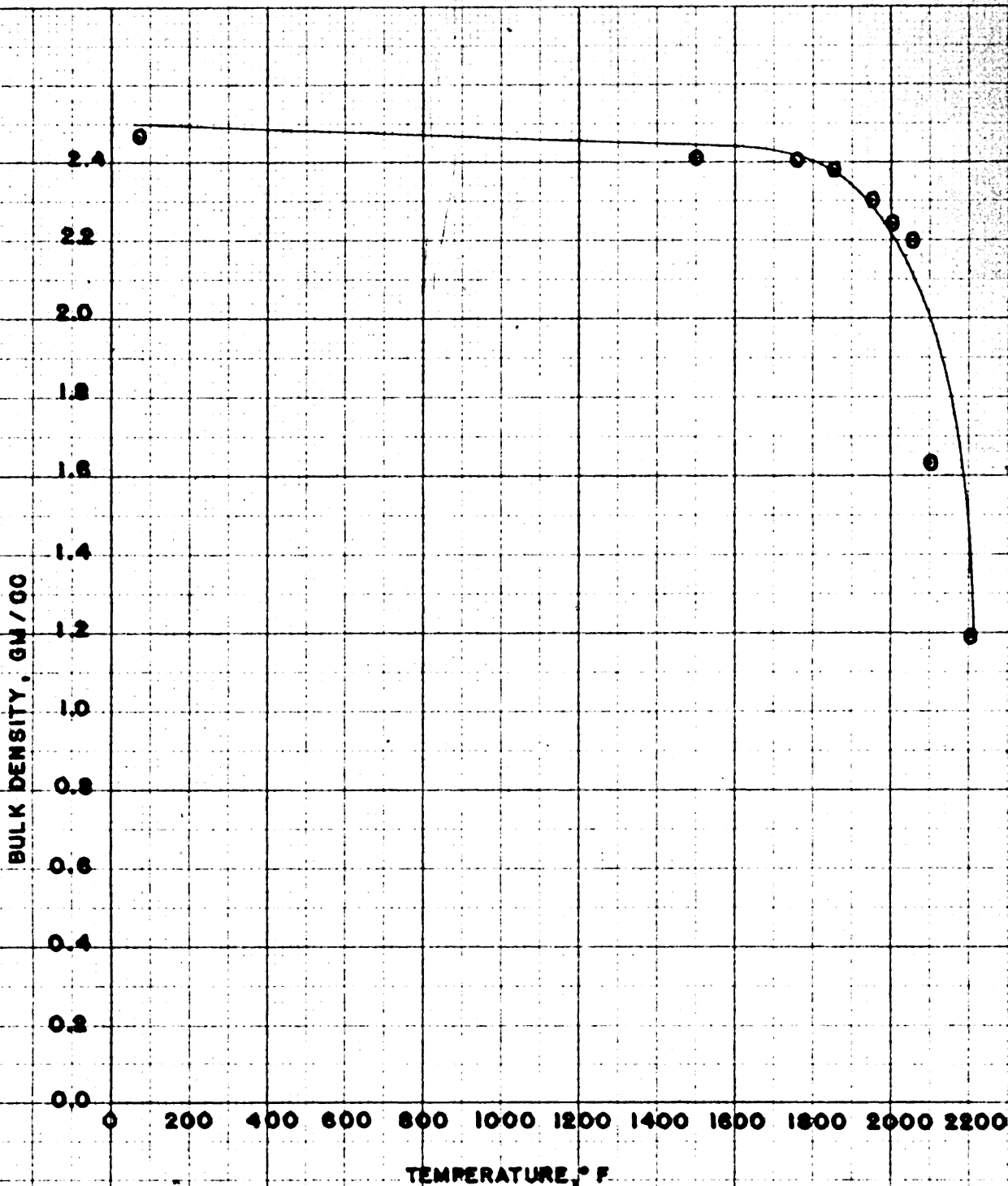


FIGURE 9. RELATIONSHIP OF THE TEMPERATURE AT WHICH WEATHERED LIBERTY HALL SHALE WAS REMOVED FROM THE FURNACE TO ITS BULK DENSITY

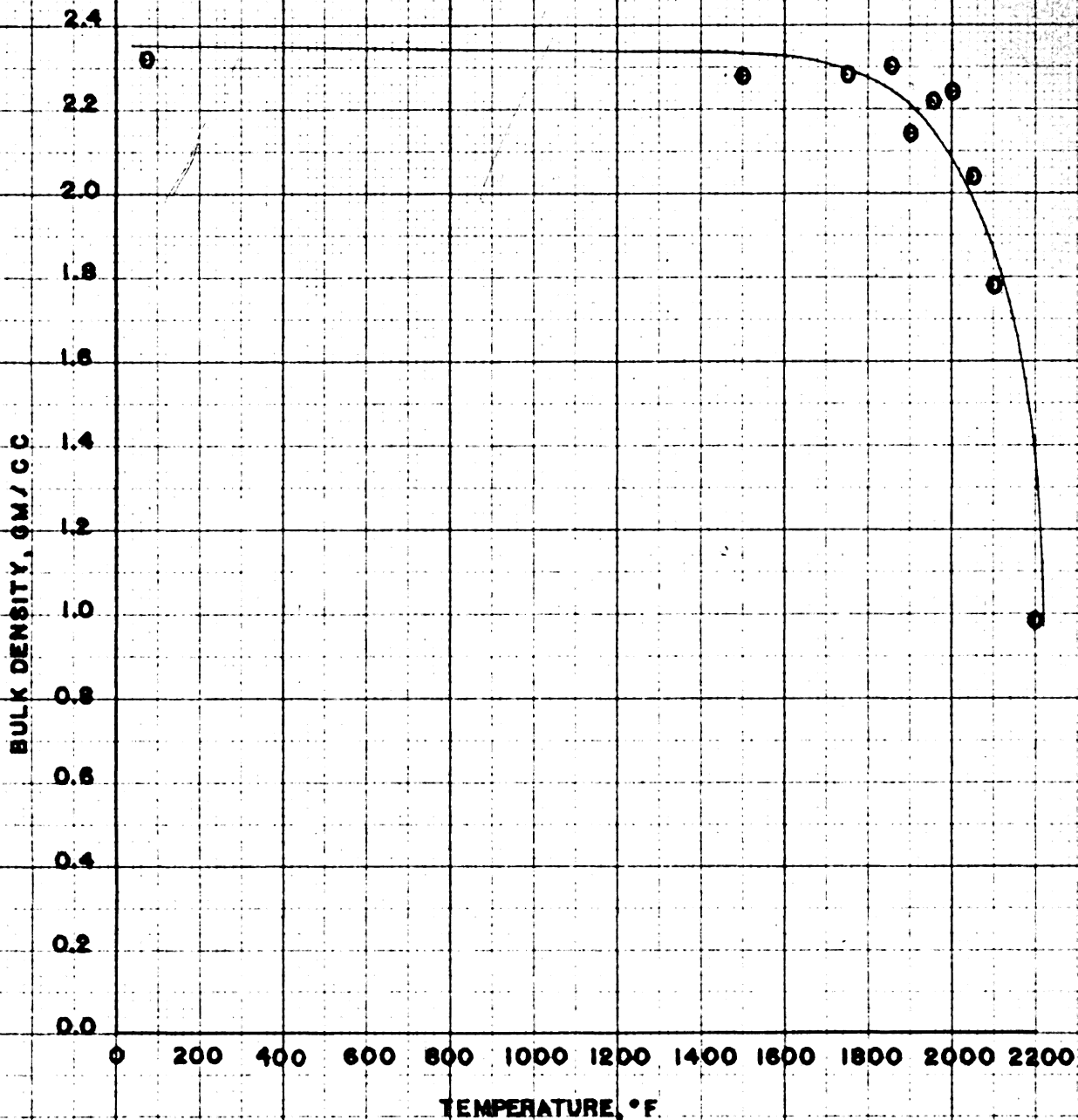


FIGURE 10. RELATIONSHIP OF THE TEMPERATURE AT WHICH THE NON-WEATHERED MARTINSBURG SHALE WAS REMOVED FROM THE FURNACE TO ITS BULK DENSITY

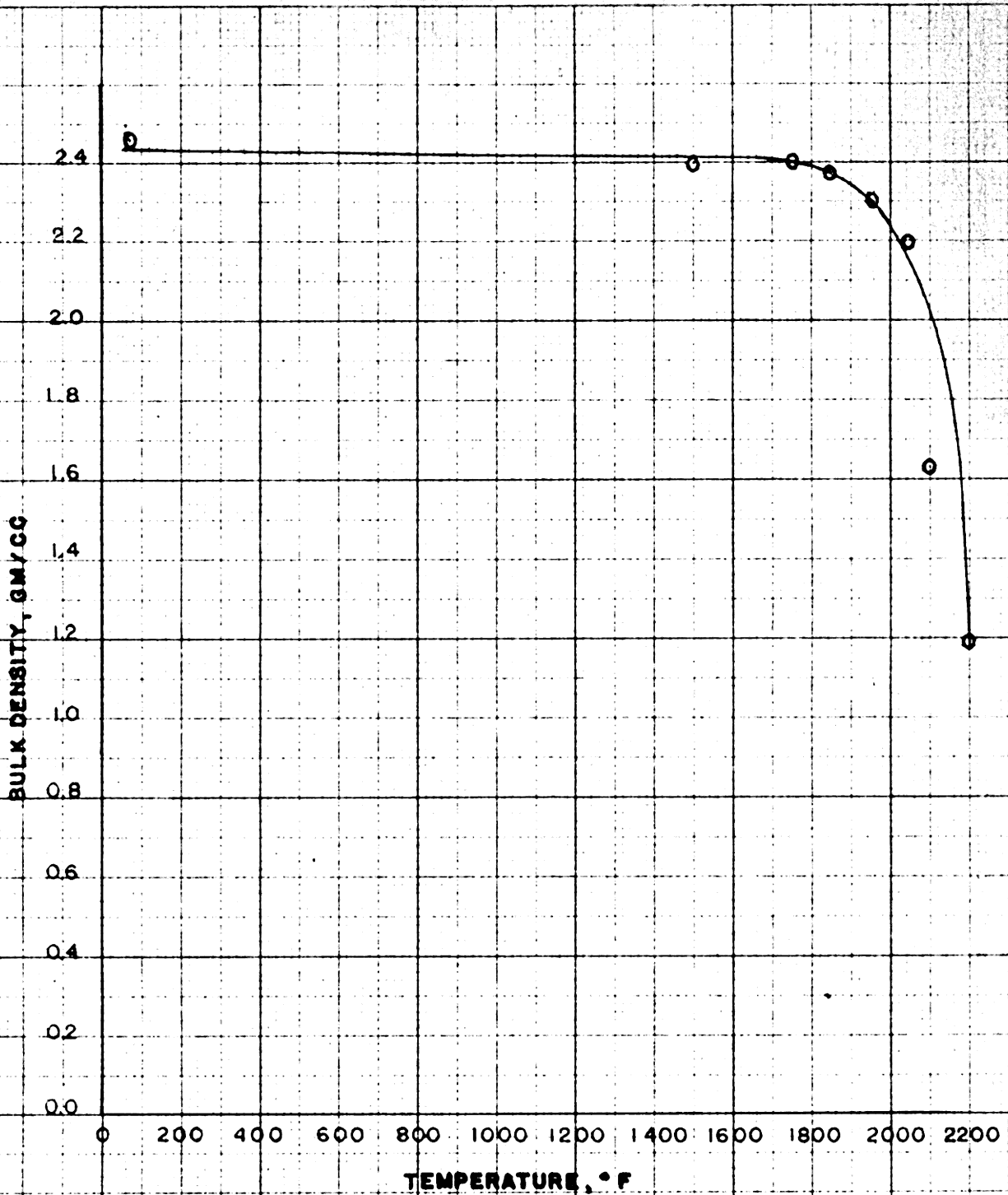


FIGURE II. RELATIONSHIP OF THE TEMPERATURE AT WHICH NON-WEATHERED BRALLIER SHALE WAS REMOVED FROM THE FURNACE TO ITS BULK DENSITY

TABLE VII

Data Used for the Determination of Time of Exposure
to 2050 °F on the Bloating Characteristics of
Non-Weathered Liberty Hall Shale

Time Elapsed	Weight in Air		Weight in Mercury		Bulk Density	
	Before	After	Before	After	Before	After
min	gm	gm	gm	gm	gm	gm
0	25.0	24.5	537	--	2.327	--
5	25.0	24.3	531	2100	2.303	0.601
10	25.0	24.4	523	1468	3.310	0.851
15	25.0	24.5	521	1538	2.358	0.744
20	25.0	24.6	523	991	3.310	1.215
25	25.0	24.5	527	1374	2.327	0.991
30	25.0	23.9	529	1414	2.300	0.932
35	25.0	24.2	533	793	2.307	1.484
40	25.0	24.4	528	1137	2.315	1.115
45	25.0	24.5	525	1621	2.323	0.845
50	25.0	23.2	526	807	2.334	1.515

TABLE VIII

Bulk Densities of Non-Weathered Liberty Hall Shale When Heated to 2050 °F in One Hour and Forty-five Minutes Compared to Bulk Densities When Heated to 2050 °F in Twenty Minutes

Sample Temperature Raised from 70 to 2050 °F in Twenty Minutes		Sample Temperature Raised from 70 to 2050 °F in One Hour Forty-five Minutes		Unburned Shale	
Sample Number	Bulk Density gm/cc	Sample Number	Bulk Density gm/cc	Sample Number	Bulk Density gm/cc
1	0.469	4	0.581	7	2.725
2	0.629	5	0.639	8	2.685
3	<u>0.474</u>	6	<u>0.648</u>	9	<u>2.745</u>
Average	0.524	Average	0.623	Average	2.718

TABLE IX

Chemical Analyses of Non-Weathered MacCraday and Liberty Hall
Shales Obtained from Deposits Located in Virginia^a

Components wt %	MacCraday Shale	Liberty Hall Shale	Liberty Hall Shale
	Unburned	Burned ^b	Unburned
Loss on Drying at 212 °F	0.25	0.00	0.65
Loss on Heating at 800-900 °F	0.26	0.00	0.91
Loss on Heating at 1700-1800 °F	0.70	0.00	1.51
Silica	60.22	51.50	48.63
Ferric Oxide	1.66	4.62	3.75
Aluminum Oxide	35.30	29.45	29.16
Calcium Oxide	--	4.28	--
Magnesium Oxide	--	5.72	--
Calcium Carbonate	Trace	--	5.75
Magnesium Carbonate	Trace	--	7.63
Undetermined	<u>1.61</u>	<u>4.43</u>	<u>2.01</u>
Total	100.00	100.00	100.00

^a Location of shale deposits are shown in Figure 5, page 50.

^b Temperature of bloating, 2000 °F.

TABLE X

Grading of Aggregate Used to Make Concrete for Compressive
Strength Test Cylinders - Used to Compare Strengths of
Cinder Concrete and Bloated Shale Concrete

Tyler Standard Screen No	Differential	Cumulative
Passing	Amount Retained	Amount Retained
Retained on	on Screen	on Screen
	wt %	wt %
-	3/8	6.34
3/8	4	40.60
4	8	15.28
8	14	9.94
14	28	7.95
28	48	4.97
48	100	6.97
100	Pan	7.95
		100.00

TABLE XI

Compressive Strengths of Concrete Test Cylinders Made with
Cinders and Bloated Virginia Shale^a

Sample Number	<u>Cinder Concrete</u>			
	Weight	Cinder Concrete Density	Ultimate Load	Unit Strength
	lb	lb/cu ft	lb	lb/sq in
C1	18.38	93.60	58,000	2051
C2	18.33	93.35	60,000	2122
C3	<u>18.35</u>	<u>94.54</u>	<u>54,000</u>	<u>1910</u>
Average	18.33	93.47	57,333	2028

Sample Number	<u>Bloated Shale Concrete</u>			
	Weight	Bloated Shale Concrete Density	Ultimate Load	Unit Strength
	lb	lb/cu ft	lb	lb/sq in
S1	22.39	114.05	117,000	4138
S2	22.59	115.04	121,000	4279
S3	<u>22.35</u>	<u>113.82</u>	<u>123,000</u>	<u>4350</u>
Average	22.44	114.30	120,333	4256

^a Non-weathered Liberty Hall and Martinsburg shales were used to make the test cylinders. Location of shale deposits are shown in Figure 5, page 50, and Figure 6, page 52.

Sample Calculations

The calculations involved in this investigation are presented in the following section:

Bulk Density. The bulk density of a sample of shale was determined by dividing the weight of the sample in air by the volume of the sample in mercury.

The following calculations all relate to sample L-N-1, Table III, page 73.

Bulk Volume. The bulk volume of a sample obtained by dividing the sum of the weight in air and weight in mercury of the sample by the density of mercury.

$$V = \frac{W + \frac{W_s}{a}}{D_m}$$

where:

- V = specimen bulk volume, gm/cc
- W = specimen weight in air, gm
- W_s = specimen weight in mercury, gm
- D_m = density of mercury at 70 °F, gm/cc
- a = constant, supplied by manufacturer of the volumeter⁽⁷²⁾.

$$V = \frac{25.0 + \frac{521}{4.40}}{13.5434}$$

$$V = 10.66 \text{ cc}$$

Bulk Density. The bulk density of a sample is obtained by dividing the weight of the sample in air by the bulk volume.

$$D_b = \frac{W}{V}$$

where:

D_b = bulk density of specimen, gm/cc

$$D_b = \frac{25.0}{10.66}$$

$$D_b = 2.356 \text{ gm/cc.}$$

IV. DISCUSSION

The work performed in this investigation pertaining to the production of a lightweight aggregate concrete from Virginia shale will be reviewed and analyzed under the general headings of discussion of materials, equipment, and conditions, discussion of results obtained, recommendations, and limitations.

Discussion of Materials, Equipment, and Conditions for Producing a Lightweight Aggregate

The following discussion deals with materials, equipment, conditions, and their bearing on the results obtained in this investigation.

Shale Tested. The Liberty Hall (coded L-N and L-W), MacCraday (coded Mc-N and Mc-W), Martinsburg (coded M-N), and Brallier (coded B-N) shales that were used were obtained from outcroppings of extensive deposits which extend from Pennsylvania through West Virginia, Virginia, and into the Carolinas⁽¹⁴⁾. The outcroppings which were used are all located within Montgomery County, Virginia. The exact location of the shale deposits are shown in Figures 5 and 6, pages 50 and 52. Other than the fact that these deposits

were readily available, it was found in the literature⁽¹⁴⁾ that the shales from these deposits would have relatively the same chemical and physical properties as those of the same seam or strata in either Pennsylvania or the Carolinas.

Shale Sampling. Two samples of Liberty Hall shale were obtained. The weathered Liberty Hall shale was taken from the face of a road cut, while the non-weathered shale was taken from the same road cut at a depth of approximately one foot. The weathered Brallier shale was so disintegrated that a satisfactory sample for bloating could not be obtained, therefore, only non-weathered Brallier shale was used. This shale was taken from a road cut by digging to a depth of two feet to remove the overburden weathered shale. Two samples of MacCrady shale, weathered and non-weathered, were used in this investigation. The weathered shale was taken from the face of a road cut, while the non-weathered shale was obtained by digging to a depth of 12 to 18 inches in order to remove all the overburden weathered shale. The non-weathered Martinsburg shale was obtained from a deposit near the surface by first removing the top three to five inches of soil and then removing the sample. It was assumed that this shale suffered no weathering action since it was very hard and dense.

All of the shale samples were obtained from the deposits by the "pick and shovel method." Approximately fifty pounds of each type of shale was obtained.

In view of the manner in which sampling was done, it is recommended for future investigators to obtain the non-weathered shale samples by using core drillings. This method of sampling would probably eliminate the possibility of obtaining any non-weathered shale, and also, a more representative sample of the shale could be obtained.

Muffle Furnace. An electric type muffle furnace was used which had inside dimensions of 10 by 4 by 3 inches. The complete specifications for the furnace used may be found in the section on Apparatus, page 55. This furnace was chosen because its maximum temperature, 2200 °F, was within the temperature range to be employed for bloating the shale.

One of the major difficulties in using a muffle furnace of this size was its small capacity. The furnace used had a maximum capacity of twenty-five pounds of shale per firing cycle. However, when the furnace was filled to capacity with shale it was found that some of the shale failed to

bloat, particularly that on the inside of the pile in the furnace. On the basis of this difficulty and the fact that approximately seventy-five pounds of expanded shale were necessary for making compressive strength cylinders, it is recommended that a rotary kiln capable of handling fifty pounds of shale per hour be designed and constructed to bloat the shale.

The shale samples used for bloating tests were supported on ceramic slabs resting on the floor of the furnace during firing. The ceramic blocks were chosen for their heat and shock resistant qualities.

Sample Preparation. The preparation of the samples for firing was the same for all shales. The shale samples were washed with water and cleaned of all extraneous matter with a wire brush. The samples were broken from large lumps, approximately two inches thick, four inches long, and four inches wide, into small pieces, approximately one-half inch in diameter, of shale in an iron mortar with a pestle. The weight of each of the samples that were used in each test was 25.0 ± 1.0 grams.

Calibration of Thermocouple and Pyrometer. The alumel-chromel thermocouple, which was used to measure the temperature of the muffle furnace, was calibrated against the

melting points of tin, lead, and antimony. The curve that was obtained from plotting the temperature versus the electromotive force for the melting points of the metals agreed completely with a standard millivolt-temperature curve which was drawn from published data⁽⁸¹⁾.

The optical pyrometer, which was used to check the temperatures of the muffle furnace, was calibrated against the melting points of copper and zinc wires. The pointer of the meter attached to the pyrometer was then corrected to the melting points of the two wires.

When both the thermocouple and optical pyrometer were used together it was found that from 500 to 1500 °F both the pyrometer and thermocouple were in complete agreement, as shown in Figure 7, page 72. Then, as the temperature increased from 1400 to 2200 °F there was a gradual variation between the two instruments. When the temperature of the optical pyrometer was 2125 °F, the thermocouple temperature was recorded as 2200 °F. These points were plotted and the calibration curves were drawn and used throughout the investigation.

Temperature Distribution. The temperature distribution in the muffle furnace was determined by placing a chromel-alumel

thermocouple at the back end of the furnace and moving it forward in one-inch increments along an axis approximately through the center of the furnace from back to front. The distance between the thermocouple head and the back end of the furnace, together with the temperature, was recorded for each one-inch increment.

The results of this test indicated that there was no temperature gradient existing between the back wall and front end of the furnace.

The fact that there was no temperature gradient in the furnace was further substantiated by the use of pyrometric cones. Plaques, each containing three cones, 03 (1920 °F), 2 (2050 °F), and 4 (2100 °F), were placed in the four corners of the muffle furnace. When the temperature of the furnace was 2050 °F, as indicated by the alumel-chromel thermocouple, it was noted that all of the cones were in approximately the same position; that is, cone 2 down and cone 4 at 2 o'clock. Since the cones on all four plaques were in the same position it was assumed that the temperatures in all four corners of the furnace were the same.

Firing Schedule. The firing schedule that was used throughout this investigation was determined by subjecting the four types of shale used to various rates of heating

until a firing schedule was obtained that would allow the shale to be bloated uniformly.

In the determination of the firing schedule many difficulties were encountered. It was found that the temperature of the furnace had to be increased slowly from 70 to 240 °F in order for the mechanically combined water to be driven off. If the temperature was raised too rapidly through this range the shale samples would shatter. Another difficulty which was encountered was that in the temperature range from 900 to 1500 °F the shale would shatter if the rate of heating was too rapid. Searle⁽⁶⁸⁾ stated that between 932 and 1472 °F a quick evolution of waters occurs. It was, therefore, necessary to maintain an arrest in the firing curve between 900 and 1500 °F to avoid shattering of the shale sample. Between 1500 and 2200 °F it was necessary to obtain a rate of heating that would enable the shale to bloat uniformly. Too rapid a rate of heating in this temperature range was found to produce an expanded shale product with very large pores, approximately three-quarters inch in diameter, which were all interconnected. A bloated shale of this type would not be of any use as a lightweight aggregate due to its very low strength.

After a considerable number of preliminary tests a firing schedule was obtained that would allow the shale to expand properly. This firing schedule was then duplicated for all tests.

Bloating Conditions. All of the tests were performed at atmospheric pressure in an oxidizing atmosphere. Since an electric furnace was used instead of a gas-fired furnace, there were no unburned gases available to produce a reducing atmosphere. It is recommended that in future investigations, a reducing atmosphere be used so that its effect, if any, on the bloating of shale may be determined.

Discussion of Results

The following discussion is presented in an attempt to interpret and clarify the experimental data and results of this investigation.

Non-weathered Liberty Hall Shale. The non-weathered Liberty Hall shale deposit, the location of which is shown in Figure 6, page 52, yielded a shale of excellent bloating quality at 2050 °F. Samples L-N-4, 3, and 1, ranked first, second, and third among the samples of shale tested with ratios of unburned to burned bulk densities of 4.32, 3.41, and 2.79, respectively. The average ratio of unburned to burned bulk density of all the samples tested was 3.20. This average value compares favorably with that of the commercial lightweight aggregate Haydite⁽²⁹⁾ which has an average ratio of unburned to burned bulk density of 2.75. Hayde⁽²⁹⁾ stated that the two most important factors involved when choosing a shale for the commercial production of a lightweight aggregate are, first, the shale must have an average ratio of unburned to burned bulk density of 2.00, or more, and secondly, the temperature of bloating must be below 2400 °F. Since the results of this test show that

non-weathered Liberty Hall shale readily fulfills both of the requirements as stated by Hayde, it is believed that a concrete of very light weight could be produced commercially from this expanded shale.

Samples L-N-2 and 5 showed ratios of unburned to burned bulk density of 2.29 and 2.08, respectively. These values are considerably lower than the average ratio of 3.20 for all of the non-weathered Liberty Hall shale and is probably due to variance in the chemical and physical composition of the shale and the method of sampling.

The average burned bulk density of the non-weathered Liberty Hall shale, Table V, page 75, was found to be 0.84 gram per cubic centimeter. This compares favorably with Kanamite⁽⁶³⁾ which fires to an average burned bulk density of 0.96 gram per cubic centimeter. The importance of the fired bulk density was reported by Klinefelter⁽³³⁾ who stated that a burned bulk density of approximately one gram per cubic centimeter was desirable for a lightweight aggregate since this lessens the tendency for the aggregate to sink to the bottom or rise to the top of a wet concrete mass.

The appearance of the expanded non-weathered Liberty Hall shale was the same for all five samples tested. Their color was a glossy grey and the average pore size was relatively large, ranging visually from one-thirty second to one-quarter inch in diameter. The strength of these five fired samples of shale was not as good as Martinsburg shale as it was possible to break and crush the samples by hand. However, very few, if any, of the pores were interconnected and it is believed that a non-load bearing concrete of very light weight could be made from this shale.

Weathered Liberty Hall Shale. The weathered Liberty Hall shale that was obtained from the deposit located as shown in Figure 5, page 50, did not bloat very well at 2050 °F. The average ratio of unburned to burned bulk density of this shale was 1.66, as shown in Table V, page 75. This average ratio is approximately one-half of that obtained for the non-weathered Liberty Hall shale. The difference is probably due to the fact that weathering of the Liberty Hall shale caused a decrease in the amount of carbonates or other water-soluble constituents within the shale by leaching, thus reducing the amount of gas producing substances required for bloating. The average bulk density

of the bloated shale, Table V, page 75, was found to be 1.71 grams per cubic centimeter. This value is high in comparison to that of the commercial cinders which have a bulk density of 1.41 grams per cubic centimeter⁽¹⁸⁾. The high burned bulk density and the low ratio of unburned to burned bulk density are ample evidence that this shale, by itself, could not be used as a source of lightweight aggregate; however, it is believed that this shale would not be detrimental to the extent of warranting its removal from the non-weathered Liberty Hall shale deposit.

The appearance of the burned weathered Liberty Hall shale was the same for all five samples tested. The color was a dark grey and the average pore size was relatively small, ranging visually from one-thirty second to one-eighth inch in diameter. The apparent strength of this shale was poor, it being possible to crush the expanded shale by hand.

Martinsburg Shale. From the results shown in Table V, page 75, it is to be noted that of the seventeen samples that were bloated, no samples of non-weathered Martinsburg shale ranked below nine in ability to bloat. The best bloating Martinsburg shale was M-N-1 which showed a ratio of unburned

to burned bulk density of 2.66. The poorest bloating sample of Martinsburg shale was M-N-3 which had a ratio of densities of 2.13. The average ratio was found to be 2.50 at a bloating temperature of 2050 °F. This shale also fulfills the requirements set forth by Hayde⁽²⁹⁾ in that its average ratio of unburned to burned bulk density is greater than 2.00 and its temperature of bloating is less than 2400 °F.

It is interesting to note that the Martinsburg shale may be made to bloat to a bulk density varying from 0.88 to 1.11 grams per cubic centimeter with the average being 0.99 gram per cubic centimeter. This value compares quite favorably with the value proposed by Klinefelter⁽³³⁾ of approximately one gram per cubic centimeter as previously mentioned in the discussion on non-weathered Liberty Hall shale. Also, the range of the bulk densities of the burned Martinsburg shale is less than that of the commercial cinders used as lightweight aggregate, thus indicating a more uniform aggregate may be obtained. Part of the so-called cinders consists of very light coke-like material which has a bulk density of about 1.5 gram per cubic centimeter. On the other hand, glass-like slag is often found in cinders which has a bulk density of more than 2.0 grams per cubic

centimeter. Of course, other material may be found in the cinders having bulk densities of anywhere from 0.5 to 2.0 grams per cubic centimeter producing an average bulk density of 1.41 grams per cubic centimeter⁽¹⁸⁾.

The appearance of the burned Martinsburg shale was a very dark brown color. The apparent strength of the burned shale was good, it being impossible to crush or break the shale by hand. The average pore size was relatively large, ranging visually from one-eighth to one-quarter inch in diameter. Very few, if any, of the pores were interconnected and it is believed that a load-bearing concrete of very light weight could be made from this shale.

Brallier Shale. The shale samples from the Brallier deposit, location of which is shown in Figure 5, page 50, did not show good bloating characteristics. This was probably due to the high limestone and other carbonate content of the shale. Riley⁽⁶⁶⁾ determined that for any material to bloat it must satisfy two conditions. First, the shale must have a chemical composition with the proper balance of fluxing agents such as calcium and magnesium carbonates, sodium and potassium oxides, and ferrous and ferric oxides, and secondly, the material should contain

some substance which evolves a gas at or above the fusion point. He also stated that an excess of gas-producing substances within the shale such as calcium and magnesium carbonates cause an increase in the bloating temperature and also a higher fluidity of the viscous mass due to the fluxing action of the carbonates and their respective oxides. Although a quantitative test for limestone and other carbonates was not made on the Brallier shale, the fact that effervescence occurred when the samples were treated with acid, together with the fact that the burning of the samples according to the firing schedule shown in Figure 7, page 72, caused the samples to become powdery and white, was considered ample evidence that the samples were too high in carbonate content to be used as a lightweight aggregate.

The general appearance of these samples after burning was a cellular structure in which this powdery white substance was deposited. The burned shale had a black color and its apparent strength was very poor in that the samples fractured when heated to 2050 °F.

In view of the results on the tests on Brallier shale of the non-weathered type, it is not deemed advisable to use the shale obtained from the deposit located as shown in Figure 5, page 50, for the production of a lightweight aggregate.

Weathered and Non-weathered MacCraday Shale. The MacCraday shale deposit located as shown in Figure 6, page 52, yielded a hard red shale that burned without bloating to a brick-like product. Accordingly, bulk density tests were not made on this burned shale. The brick-like product formed is probably due to the high alumina and silica content of the shale. From the very nature and appearance of the fired product the shale of this deposit may prove feasible for building brick manufacture and it is recommended that the necessary tests required on raw materials for brick manufacture be performed and the shale evaluated for such use.

Determination of the Temperature of Bloating. In this test it was desired to determine the temperature at which the poorer bloating shales expanded since any piece of apparatus used to bloat the shales used in this investigation would have to be operated at the temperature required to bloat the poorest shale. Therefore, non-weathered Liberty Hall, Martinsburg, and Brallier shales, together with weathered Liberty Hall shale, were selected for these tests. Figure 8, page 77, shows graphically the results of a test in which ten samples of non-weathered Liberty Hall shale were burned according to the firing schedule as shown in Figure 7, page 72.

One sample of the non-weathered Liberty Hall shale was withdrawn from the furnace as the temperature reached each of the following points: 1500, 1750, 1850, 1900, 1950, 2000, 2050, 2100, and 2200 °F. The bulk density of each of the samples was determined and plotted against the temperature at which it was withdrawn from the muffle furnace, as shown in Figure 8, page 77. The resulting curve shows that from 70 to 2000 °F, the bulk density decreased only from 2.36 to 2.04 grams per cubic centimeter while from a temperature of 2000 to 2200 °F the bulk density decreased from 2.04 to 0.84 gram per cubic centimeter. Thus, it is seen that very little bloating occurred before the temperature reached 2000 °F and practically all of the bloating occurred in the temperature range from 2000 to 2200 °F.

The test made on the Liberty Hall weathered shale resulted in the curve shown in Figure 9, page 78. In this case, the bulk density decreased from 2.47 grams per cubic centimeter at 70 °F to 2.24 grams per cubic centimeter at 2000 °F, while it decreased from 2.24 to 1.19 grams per cubic centimeter in the temperature range from 2000 to 2200 °F.

The test made on non-weathered Martinsburg shale shown in Figure 10, page 79, was similar to the test made on the non-weathered Liberty Hall shale. In this case, the bulk density decreased from 2.32 grams per cubic centimeter at 70 °F to 2.24 grams per cubic centimeter at 2000 °F, while it decreased from 2.24 to 0.89 gram per cubic centimeter in the temperature range from 2000 to 2200 °F.

The test made on the Brallier shale, which was the poorest bloating shale that was considered during this investigation, resulted in the curve shown in Figure 11, page 80. The bulk density decreased from 2.39 grams per cubic centimeter at 70 °F to 2.17 grams per cubic centimeter at 2000 °F, while from 2000 to 2200 °F it only decreased from 2.17 to 1.80 grams per cubic centimeter.

Thus, it is seen that none of these samples bloated appreciably until a temperature above 1950 °F was reached. Each curve, as shown in Figures 8, 9, 10, and 11, pages 77, 78, 79, and 80, respectively, broke sharply downward when this temperature was reached, indicating that the minimum temperature for bloating of the shales tested was 1950 °F when heated according to the firing schedule shown in Figure 7, page 72. However, the extent of complete

bloating necessary to produce a lightweight aggregate of desired bulk density was not obtained until a temperature of 2050 °F was reached.

Determination of the Effect of Time of Exposure to 2050 °F on the Bloating of Non-weathered Liberty Hall Shale.

As shown in Figure 8, page 77, bloating began at 2000 °F for the non-weathered Liberty Hall shale. It was desired to determine if prolonged exposure to this temperature would increase the bloating of the shale. Accordingly, eleven samples of non-weathered Liberty Hall shale were placed in the furnace and the temperature raised according to the firing schedule as shown in Figure 7, page 72, to 2050 °F. The temperature was maintained at this point for the duration of the test. Samples were removed from the furnace after being exposed to 2050 °F for 5, 10, 15, 20, 25, 30, 35, 40, 45, and 50 minutes. The bulk density of each sample was determined to indicate the extent of bloating.

This test was made with the expectation that the longer a particular sample was exposed to this temperature of 2050 °F, the greater the degree of bloating that could be expected. However, it was also conceived that after exposure for a certain period of time, bloating would

begin to cease and the bulk density of the sample would increase due to the fusing of the sample. Contrary to expectations, however, no definite relation could be found between the degree of bloating of the non-weathered Liberty Hall shale and the time of exposure to 2050 °F. This is brought out by the results of the test, shown in Table VII, page 81, which show that the bulk density of the shale when exposed to 2050 °F for 5, 10, 15, 20, 25, 30, 35, 40, 45, and 50 were 0.601, 0.851, 0.744, 1.215, 0.991, 0.932, 1.484, 1.115, 0.485, and 1.515 grams per cubic centimeter, respectively.

From these results, it can be seen that no regular increase or decrease in bulk density can be expected by reason of heating non-weathered Liberty Hall shale at 2050 °F for periods up to 45 minutes. This is probably due to variance in chemical and physical composition of the non-weathered Liberty Hall shale that was tested.

Determination of the Effect of Rate of Heating on the Bloating of Non-weathered Liberty Hall Shale. This test was made to determine the effect of heating the shale rapidly to 2050 °F on its bloating. The temperature of three samples of non-weathered Liberty Hall shale was raised from 70 to 2050 °F in a period of 20 minutes, after which time the

samples were removed from the muffle furnace and cooled in air to ambient temperature. The temperature of three other samples of non-weathered Liberty Hall shale was raised from 70 to 2050 °F over a period of one hour and 45 minutes according to the firing schedule shown in Figure 7, page 72.

Table VIII, page 82, gives the results of these tests and it is seen that the shale bloated to a bulk density of 0.469 gram per cubic centimeter when the temperature was raised from 70 to 2050 °F in 20 minutes while it bloated to a bulk density of 0.581 gram per cubic centimeter when the temperature was raised from 70 to 2050 °F over a period of one hour and 45 minutes. Therefore, the rapid heating does not increase the amount of bloating but, in this case, the rapidly heated samples were lower in bulk density than the slowly heated samples. It is possible that this was due to a variation in the chemical and physical composition of the samples.

It is believed that the slowly heated samples bloated to a maximum point and then, due to continued heating, began to fuse and run together and in this way experienced this slight increase in density. On the other hand, the rapidly

heated shales were not exposed to 2050 °F for a long enough period to undergo this fusion and resulted in increased bulk density.

Effect of Chemical Composition on the Bloating Qualities of Shale. Chemical analyses were made of two types of shale. Non-weathered MacCraday shale was selected as a typical non-bloating shale and non-weathered Liberty Hall shale was selected as typical of good bloating shale. By comparing the analyses of these two shales, it was thought that the component or components responsible for the superior bloating qualities of non-weathered Liberty Hall shale could be detected. Table IX, page 83, shows the results of the chemical analyses. The analyses of the burned and unburned Liberty Hall shales indicates a difference in chemical composition of the two samples analyzed. This difference is shown in the analysis for calcium oxide calculated as calcium carbonate in the unburned sample and calcium oxide in the burned sample. The unburned Liberty Hall shale contained 5.75 per cent calcium carbonate and the burned sample of Liberty Hall shale analyzed contained 4.28 per cent calcium oxide. The theoretical amount of calcium oxide that could be obtained from 5.75 per cent calcium carbonate is 3.22 per cent and when corrected for the

loss on high temperature ignition this percentage of calcium oxide is 3.32. The increase of 0.96 per cent is probably due to a difference in chemical composition between the two non-weathered Liberty Hall shale samples analyzed.

Riley⁽⁶⁶⁾, in 1951, developed a composition diagram of major oxides showing the area in which clays fired below 2400 °F form a mass viscous enough to insure good bloating. This diagram, Figure 12, page 112, shows the position of the two shales, non-weathered MacCrady and Liberty Hall, together with Haydite⁽¹⁸⁾, a commercial lightweight aggregate. The points representing the composition of these three shales on the diagram were obtained from the chemical analyses of the shales. An average composition value was used for the non-weathered Liberty Hall shale due to a variance in the chemical composition of the Liberty Hall shale analyzed. It can be seen from Figure 12, page 112, that the non-weathered Liberty Hall shale lies within the area of bloating as does the commercial aggregate, Haydite. According to Riley, any point falling within this area of bloating satisfies one of the two conditions necessary for bloating; that is, it must have a chemical composition with a proper balance of fluxing agents, silica, and alumina to allow it to produce a melt of viscosity

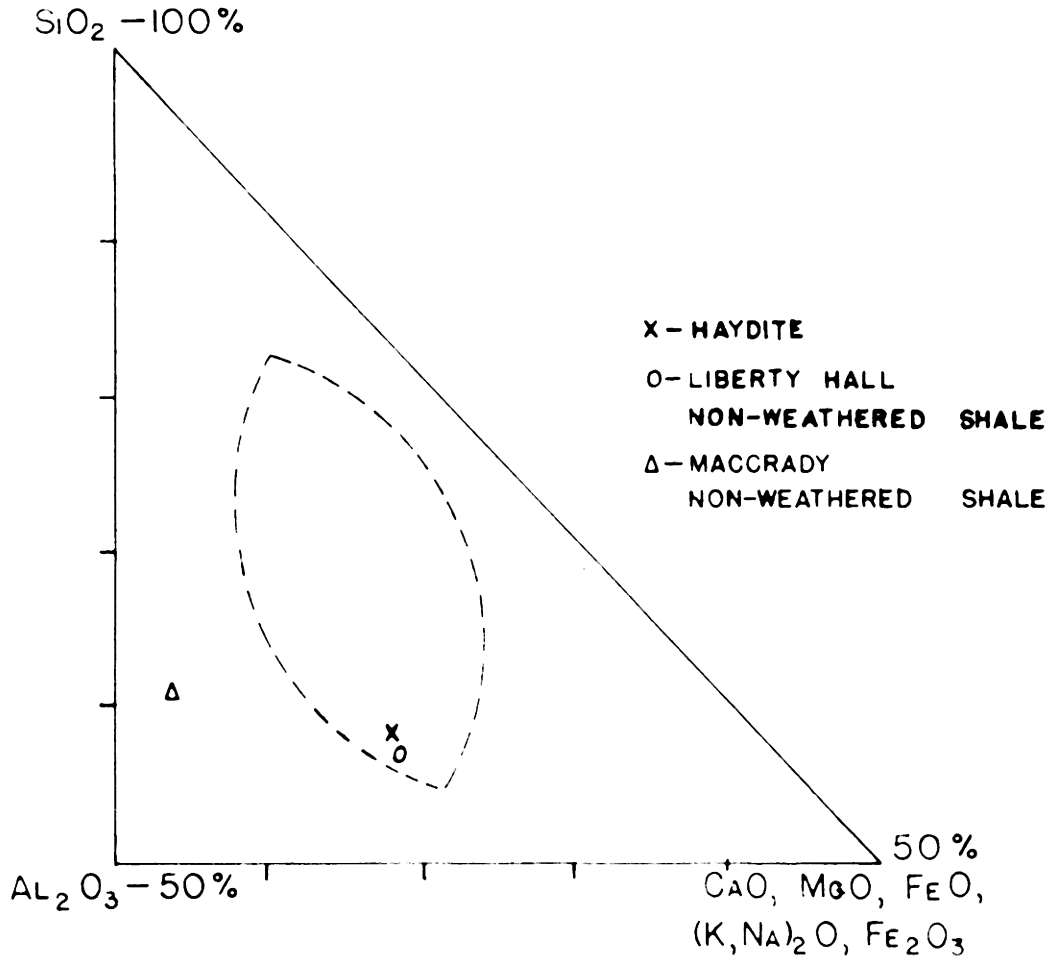


FIGURE 12. COMPOSITION DIAGRAM OF MAJOR OXIDES SHOWING AREA IN WHICH CLAYS FIRE TO A MASS VISCOUS ENOUGH TO INSURE GOOD BLOATING

RILEY, C. M. : RELATION OF CHEMICAL PROPERTIES OF THE BLOATING OF CLAYS, JOUR. AM. CER. SOC., 34, 121-128 (1951).

high enough to trap a gas. It is evident that the non-weathered Liberty Hall shale had the proper balance of fluxing agents which in the case of this shale were Fe_2O_3 , CaO , MgO , and $(\text{Na}, \text{K})_2\text{O}$, the latter being assumed present in the undetermined component of the analyses shown in Table IX, page 83. The non-weathered Liberty Hall shale also undoubtedly satisfies the second condition necessary for bloating as reported by Riley; that is, a gas be liberated at or above the fusion temperature which in the case of the Liberty Hall shale was 2050 °F. From the chemical analyses it can be seen that the gas liberated was probably due to the calcium carbonate and the magnesium carbonate decomposing into their respective oxides and carbon dioxide gas. This carbon dioxide was entrapped in the viscous mass causing the Liberty Hall shale to bloat.

From the chemical analysis of the non-weathered MacCraday shale, together with the composition diagram shown in Figure 12, page 112, two definite reasons can be advanced for the non-bloating properties of this shale at 2050 °F. The first reason is that the shale contained only traces of gas producing substances such as calcium and magnesium carbonates. This is evident from the chemical analysis of the MacCraday shale which shows that 95.52 per cent of the shale was composed of

alumina and silica, 1.26 per cent volatile matter, traces of calcium and magnesium carbonates, 1.66 per cent ferric oxide, and 1.61 per cent undetermined. The two latter components were assumed to be the fluxing agents in the shale. The second and probably the most important reason that the non-weathered MacCrady shale would not bloat at 2050 °F is due to the improper balance of the fluxing agents within the shale. In Figure 12, page 112, non-weathered MacCrady shale lies outside the area of bloating proposed by Riley⁽⁶⁶⁾. This is probably due to the low quantity of fluxing agents which is assumed to be 3.27 per cent and also the high, 95.52 per cent of alumina and silica.

This shale could probably be made to bloat by the use of additives such as calcium and magnesium carbonates which would increase the amount of fluxing agents so that the chemical composition of the shale would fall within the area of bloating. The use of such additives as carbonates would probably produce the necessary gas that would enable the MacCrady shale to bloat at its fusion point.

Comparative Compressive Strengths of Concrete Made with Cinder as an Aggregate and of Concrete Made with Bloated Shale as an Aggregate. Table XI, page 85, gives the results of

compressive strengths made on cinder-concrete cylinders and bloated shale-concrete cylinders. A two to one weight ratio of non-weathered Liberty Hall shale to non-weathered Martinsburg shale was used as the aggregate in these tests. The weight ratio of cement to aggregate was one to three. Approximately seventy-five pounds of expanded shale were necessary for making the test cylinders and since the capacity of the furnace was only twenty-five pounds per firing cycle, it was thought that by using approximately twenty-five pounds of previously bloated non-weathered Martinsburg shale considerably time could be saved. As shown in Table XI, page 85, the weight of the individual cylinders made with cinder did not vary more than 0.50 pound while cylinders made with bloated shale varied less than 0.25 pound. Thus, it is believed that the method of making and tamping the concrete cylinders was quite adequate for this test.

The density of the cinder concrete was 93.47 pounds per cubic foot, while the density of the bloated shale concrete was 114.3 pounds per cubic foot. The bloated shale concrete was, therefore, 20.83 pounds per cubic foot heavier than the cinder concrete. Ordinary sand and gravel concrete weighs approximately 145.0 pounds per cubic foot, which is more than

thirty pounds per cubic foot heavier than the bloated shale concrete. The reason for the bloated shale concrete being heavier than the cinder concrete was due to the fact that two furnaces, each containing twenty-five pounds of non-weathered Liberty Hall shale, were used to bloat enough shale for the cylinders. The result was that some of the shale failed to bloat, particularly that on the inside of the pile in the furnace. From a comparison of the fired bulk densities of the cinders and non-weathered Liberty Hall and Martinsburg shales it is believed that if all of the shale had been properly fired, the density of the shale-concrete would have ranged from 86 to 88 pounds per cubic foot.

The average compressive strength of the three cinder-concrete cylinders was 2020 pounds per square inch, while the average of the three bloated shale-concrete cylinders tested was 4256 pounds per square inch. The average compressive strength for a one to three sand to cement weight ratio is 5280 pounds per square inch⁽⁶³⁾. The unit strengths of both the cinder and expanded shale concrete cylinders did not vary more than 212 pounds per square inch. The bloated shale-concrete was more than twice as strong as the cinder-

concrete but some of this strength is due to the fact that all of the aggregate in the bloated shale concrete did not bloat thus making the concrete more dense and stronger than if all of the non-weathered Liberty Hall shale had expanded as in previous control tests. In spite of this, the compressive strengths obtained are ample evidence that a superior concrete can be made from a mixture of non-weathered Liberty Hall and Martinsburg shales to that which can be made from commercial cinders now available for making concrete.

Recommendations

A number of improvements in both experimental apparatus and technique suggested themselves during the course of this investigation. The following paragraphs contain recommendations to be considered in future investigations.

Rotary Kiln. A rotary kiln capable of handling fifty pounds of shale per hour should be designed and constructed to bloat the shale. Also, the lining of the kiln should be of a material capable of withstanding temperatures to 2500 °F.

Higher Bloating Temperatures. The shale samples should be subjected to temperatures above 2200 °F to determine the temperature at which bloating ceases and the bulk density begins to increase again.

Chemical and Petrographic Analyses. Thorough chemical and petrographic analyses should be made of a good bloating shale and a poor bloating shale, which includes the determination of the alkali metals, to determine the effect of their fluxing action on the bloating of the shale.

Compressive Strength Test. A minimum of fifty pounds shale of each type should be bloated in order to make concrete cylinders so that a comparison can be made of the strengths of concrete produced from aggregate of different bulk densities.

Types of Shale. Only four types of shale were used during this investigation. It is therefore recommended that several other types of shale formations, namely, Rome, Cambrian, Athens, Millboro, and Warsaw, be investigated so that a comparison can be made of all the different types of shale formations throughout the state of Virginia.

Condition of Shale. A thorough investigation should be made of the effect of weathering and non-weathering on the bloating characteristics of Virginia shale.

Type of Atmosphere. Tests should be made to determine if the type of atmosphere, oxidizing or reducing, have any effect on the bloating of shale.

Limitations

The following paragraphs contain the limitations under which this investigation was performed.

Shale Samples. The shale samples used in this investigation were of four different types of shale formation, Liberty Hall, MacCraday, Martinsburg, and Brallier shales. The location of these deposits as shown in Figures 5 and 6, pages 50 and 52, are a definite limitation in that shale varies in its chemical composition for each deposit.

Muffle Furnace. A maximum of twenty 25-gram samples of shale could be bloated at one time in the muffle furnace available. It was found, when the muffle furnace was loaded with approximately twenty pounds of crushed shale, that much of the shale on the interior of the pile did not bloat. It is for these reasons that future investigators in this field are advised to employ a rotary kiln capable of handling between thirty and fifty pounds per hour.

Temperature Range. Figures 8, 9, 10, and 11, pages 77, 78, 79, and 80, show that bloating continues with the shales tested until the maximum temperature of 2200 °F used in this investigation was reached. It would be desirable to know the

temperature at which this decrease in bulk density ceases and the temperature at which the bulk density begins to increase again due to fusion of the shale. The lining of the muffle furnace used would not withstand temperatures in excess of 2200 °F. One attempt to raise the temperature to 2300 °F resulted in severe damage to the lining of the muffle furnace.

Analyses. Only chemical analyses were performed in this investigation; consequently, no knowledge of the mineral composition or structure was obtained for the shale. It is for this reason that a petrographic analysis as well as a chemical analysis should be performed.

V. CONCLUSIONS

From the results of the investigation of the bloating qualities of weathered and non-weathered Liberty Hall shale, weathered and non-weathered MacCrady shale, Martinsburg shale, and Brallier shale taken from deposits located in the state of Virginia, as shown in Figures 5 and 6, pages 50 and 52, the following conclusions can be drawn:

1. For the best bloating shale tested, non-weathered Liberty Hall, the bulk density was reduced from 2.349 to 0.544 gram per cubic centimeter and for the poorest bloating shale, Brallier, the bulk density was reduced from 2.448 to 2.389 grams per cubic centimeter when heated in a muffle furnace to 2200 °F over a period of one hour and thirty-six minutes. The average reduction in bulk density for the shale that was bloated was from 2.351 to 1.210 grams per cubic centimeter.

2. The average reduction in bulk density of Liberty Hall, weathered and non-weathered, Martinsburg, and Brallier shales when brought to temperature of 2200 °F in a period of one hour and thirty-six minutes is from 2.770 to 1.200 grams per cubic centimeter. Of this reduction in bulk density, only 12.5 per cent occurred when the samples were brought from

70 to 1900 °F, while 87.5 per cent of the reduction in bulk density occurred while the temperatures of the samples were being raised from 1900 to 2200 °F.

3. Non-weathered Liberty Hall shale does not decrease in bulk density by reason of prolonged heating up to fifty minutes at a temperature of 2050 °F. The bulk density of a sample of the non-weathered Liberty Hall shale when heated only five minutes at 2050 °F was 0.601 gram per cubic centimeter, while another sample of the same type shale heated for thirty-five minutes was 1.484 grams per cubic centimeter.

4. The bulk density of non-weathered Liberty Hall shale when heated to a temperature of 2050 °F in a period of one hour and forty-five minutes is greater, 0.623 gram per cubic centimeter, than when heated to 2050 °F in a period of twenty minutes, 0.524 gram per cubic centimeter.

5. Weathered and non-weathered MacCraday shale does not bloat within the temperature range of 70 to 2200 °F. The average bulk density of MacCraday shale, weathered and non-weathered, was 2.327 grams per cubic centimeter.

6. Cinder concrete made from commercial power plant cinders obtained from Concrete Products Co., Inc., Christiansburg, Virginia, weighed 93.4 pounds per cubic foot and had a

compressive strength of 2028 pounds per square inch, while concrete made from a mixture of Liberty Hall non-weathered shale and Martinsburg shale weighed 114.3 pounds per cubic foot and had a compressive strength of 4256 pounds per square inch. Both concrete mixes were made in the proportions of one part water to 1.54 parts cement to 4.38 parts aggregate by weight. Both the cinder aggregate and the bloated shale aggregate were separated into eight size ranges in identical amounts. Both types of concrete were made the same day, cured in the same manner, and tested in compression on the same day.

VI. SUMMARY

This investigation was undertaken to determine the feasibility of producing a cellular, lightweight product by subjecting shales to temperatures of 2100 ± 100 °F in a muffle furnace. Such a product can be utilized as an aggregate for lightweight concrete to supplement cinders which have long been used. The growing scarcity of cinders and the inferior quality of the cinders now available account for the demand for another material to replace them.

Samples of weathered and non-weathered, Liberty Hall shale; non-weathered, Martinsburg shale; weathered and non-weathered, MacCrady shale; and non-weathered, Brallier shale were obtained from deposits located in the state of Virginia. The degree of bloating or expansion of the shales was measured by determining the bulk density of the shale before and after exposure to 2100 ± 100 °F.

The best bloating shale investigated, non-weathered Liberty Hall, bloated from a bulk density of 2.349 to 0.544 gram per cubic centimeter; while the poorest bloating shale, Brallier, bloated from 2.448 to 2.389 grams per cubic centimeter. In a firing test on four types of shale, weathered and non-weathered Liberty Hall and non-weathered Martinsburg

and Brallier, it was determined that only 12.5 per cent of the total bloating occurred in the temperature range between 70 and 1900 °F, while 87.5 per cent occurred in the temperature range from 1900 to 2200 °F.

In firing tests using non-weathered Liberty Hall shale it was found that no additional bloating occurred after prolonged heating at 2050 °F for forty-five minutes. This was indicated by no change in the bulk densities of the fired and unfired test specimens.

Additional firing tests using specimens of non-weathered Liberty Hall shale indicated that a rapid firing schedule was conducive to good bloating. The specimens showed bulk densities of 0.524 and 0.623 gram per cubic centimeter when heated to 2050 °F in twenty minutes and one hour and forty-five minutes, respectively.

Weathered and non-weathered MacCraday shale showed no bloating tendencies at the temperatures used in this investigation.

The densities of cinder concrete and bloating shale concrete were 93.47 and 114.3 pounds per cubic foot, respectively. The densities of these materials compare favorably with ordinary sand and gravel concrete which weighs approximately 145 pounds per cubic foot.

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