

AN INVESTIGATION OF SOME POSSIBLE COMMERCIAL USES OF CHAT, A BY-PRODUCT
OF OKLAHOMA LEAD AND ZINC MINES

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OF OKLAHOMA LEAD AND ZINC MINES

By

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PREFACE

The nature of chat, tailings deposited after the milling of lead and zinc ores, from the standpoint of a high silica content brought forth the possibility that this chat could be utilized in the manufacture of some refractory and non-refractory products in the silica products field. It was the purpose of this investigation to select the products best adapted to the substitution of chat for the raw material furnishing the silica or aggregate content and to determine experimentally whether or not this substitution would yield a suitable product. The products investigated were refractory silica brick, sand-lime building brick, and artificial stone.

Chapter I gives a brief history of the development of the lead and zinc industry in Oklahoma and describes the milling processes that yield the chat and slimes used in the investigation. Chapters II to V discuss in detail the experimental work done. The conclusions and suggestions for further study are contained in Chapter VI.

REVIEW OF THE LITERATURE

A review of the literature revealed only one reference to previous work with chat in industrial manufacturing. Pit and Quarry 22 (9), 15 (1931) contained an article stating that the Komnick Process Brick Company had been formed at Festus, Missouri, for the manufacture of clayless brick. Ground chat obtained from the lead and zinc mines in that region were to be used as the chief raw material, together with silica of sand and lime. A letter to the Chamber of Commerce of Festus brought the reply that the company never actually began production.

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CHAPTER I

INTRODUCTION

HISTORY ¹

In the heart of an agricultural territory which less than ninety years ago was proclaimed to have the ideal soil and climate for the growth of grapes and the establishment of a nationwide wine industry has developed the largest zinc producing and one of the largest lead producing areas in the world. This is the Tri-State Mining District, so-called because it includes the northeast corner of Oklahoma, the southeast corner of Kansas, and the southwestern part of Missouri.

The first ore was discovered near Joplin and, almost simultaneously, at Granby, Missouri, in 1848. Actual mining of the ore began one mile south of the present town of Granby in 1850 and since this date the region has shown almost continuous mining activity and tremendous growth in importance. Prior to 1870, only lead was mined, but by 1875 zinc was playing a very important part in the expanding of the district. In 1876, ore was discovered in Kansas, at Galena, and further impetus was given to the industry. With the discovery and opening up of other camps-- Lehigh, Smelter Hill, Chitwood, Bellville, Zincite, Central City, and Block City in Missouri and Badger, Peacock, and Empire in Kansas--the mining district gradually spread out over an extensive area.

During the nineteenth century, Oklahoma had played a very small part in the development of the mining district. Peoria in Ottawa County, near

¹The American Institute of Mining and Metallurgical Engineers, Convention Booklet, 1931, p. 3 and ff.

the Oklahoma-Missouri boundary, produced both zinc and lead, but its ore-bodies were small. In 1904, ore was struck at Quapaw and Lincolnville. However, it was not until 1906 and 1907, when ore was discovered at Hattenville, now Commerce, that the development of the great Picher camp made Oklahoma the center of production for the entire Tri-State field.

The World War caused the Oklahoma-Kansas field to become the greatest zinc producer in the world, with a peak being reached in 1919. The depression of 1921 struck the industry a blow, but it gradually revived and reached a new peak in 1925. The aggregate production of the Tri-State district from 1848 to January first, 1931, was 15,253,206 tons of zinc concentrates and 2,586,511 tons of lead concentrates. Of this total, the Picher-Miami area yielded from 1909 to 1930, inclusive, 7,630,326 tons of zinc concentrates and 1,365,367 tons of lead concentrates.

MILLING THE ORE

The first process that the lead and zinc ores undergo after they leave the mine is milling. This is the process in which the grains of the ore minerals are mechanically separated from the gangue materials to produce a "concentrate" containing most of the ore minerals and a "tailing" containing the gangue minerals.² The mechanical separation may be accomplished by gravity concentration or flotation. Both methods are employed in the Tri-State district.

Jigs and concentrating tables are the two devices most commonly used in the Tri-State area for gravity concentration. A jig consists essentially of a screen which will support a bed of ore with grain sizes varying from

²Newton, An Introduction to Metallurgy, p. 211.

1.5 inches in diameter to 2 millimeters. By mechanical movements of the screen under water, the light minerals or tailings rise to the top and the heavy minerals or concentrates build up a layer next to the screen surface. Material is continually fed into the screen and the tailings removed from the top of the bed while the layer of concentrate increases. Finally the concentrates are removed either manually or automatically.³

A concentrating table has a flat, sloping surface with a number of parallel riffles. These riffles are deepest near the head end of the table and gradually become shallower, until they disappear altogether before reaching the other end of table. The ore is fed in together with running water, at one corner of the head end of the table. The reciprocating motion of the table causes the feed to flow over the riffles and become separated into a light mineral tailing and a heavier mineral concentrate. The concentrate rides off the table at the ends of the riffles and the tailings flow off near the head end. This is made possible by a relatively slow forward stroke of the table away from the head end, and a more rapid back stroke. The table operates most efficiently on a classified feed.⁴

At the present time, the Tri-State district is one of the few places in the world where jigging is practiced extensively.⁵ The tailings from the jigs and concentrating tables are the so-called "chat" used in this investigation.

³Newton, op. cit., p. 254

⁴Ibid, p. 252

⁵Convention Booklet, op. cit., p. 22

Flotation did not gain a foothold in the Tri-State area until about 1923, but now, with few exceptions, every mill is equipped with flotation machinery. By 1931, between eighteen and twenty-five per cent of the output of the area was flotation concentrate.⁶

For the flotation process the ore is ground to the desired degree of fineness, a "flotation agent" such as oil or a hydrocarbon compound is added (copper sulphate is used in zinc flotation), and the mass is violently agitated to form a great number of air bubbles. Compressed air or a "frothing agent" may be added to aid the bubble formation. The flotation agent tends to adhere to the surface of the minerals, but not to the gangue materials. The oil-coated minerals are surrounded by air bubbles and are carried to the surface of the water in the flotation chamber, where they are skimmed off in the foam. The gangue particles settle to the bottom and are removed as tailings.⁷ These finely ground tailings are called "slimes."

The continual deposition of the tailings near the mill sites has resulted in the building up of huge chat and slime mounds. From 1917 to 1929, inclusive, 117,401,237 tons of ore rock were mined in the Oklahoma-Kansas district. Of this amount 7,726,192 tons became lead and zinc concentrates and 109,675,045 tons were thrown out as waste or tailings.⁸ Fortunately, milling in the Tri-State area has always been a fairly cheap operation, yet, if some valuable commercial use of these chats and slimes could be uncovered, a great saving in money, as well as land space, could be realized. This investigation is one attempt at the solution of the problem.

⁶Ibid., p. 24

⁷Liddell and Doan, Principles of Metallurgy, p. 144.

⁸Convention Booklet, op. cit., p. 36

CHAPTER II

Testing of the Chats and Slimes

The materials selected for this investigation were chats from the Blue Goose and Anna Beaver mills and slimes from the Blue Goose and Central mills. These mills are in the Miami-Picher area.

PROPERTIES OF THE MATERIALS

To determine the chemical and physical properties of the chats and slimes, tests were made using the testing methods of the American Ceramic Society. Chemical analysis was the only chemical property considered. The physical properties observed were particle size distribution, workability, drying and firing behavior, transverse strength, pyrometric cone equivalent or fusion point, and true specific gravity. Firing behavior included calculation of apparent porosity, volume change, and apparent and bulk specific gravities.

Chemical Analysis

The chemical analyses of the chats were made by an analyst of the United States Bureau of Mines in 1929. The slimes were analyzed by quantitative chemistry students of Oklahoma Agricultural and Mechanical College during the spring semester of 1940.

Table Ia

Chemical Analysis of Blue Goose Slime*

	Per Cent
Insolubles.....	93.35
R ₂ O ₃	0.43
CaO.....	2.46
MgO.....	0.59
CO ₂	<u>2.66</u>
Total.....	99.49

*Elizabeth Catlett, Oklahoma Agricultural and Mechanical College, analyst.

Table Ib

Chemical Analysis of Central Mill Slime**

	Per Cent
Insolubles.....	80.89
R ₂ O ₃66
CaO.....	8.45
MgO.....	1.41
CO ₂	<u>8.17</u>
Total.....	99.58

**Analyst, G. H. Sanders, Oklahoma Agricultural and Mechanical College.

Table IIa
 Chemical Analysis of Blue Goose Chat*

	Per Cent
SiO ₂	91.6
Al ₂ O ₃	1.4
Fe ₂ O ₃	0.6
CaO.....	1.6
MgO.....	0.2
S.....	0.9
CO ₂	1.0
Pb.....	0.1
Zn.....	<u>1.8</u>
Total.....	99.2

Table IIb
 Chemical Analysis of Anna Beaver Chat*

	Per Cent
SiO ₂	88.6
Al ₂ O ₃	1.1
Fe ₂ O ₃	1.1
CaO.....	2.9
MgO.....	0.1
S.....	0.8
CO ₂	3.3
Pb.....	0.1
Zn.....	<u>1.3</u>
Total.....	99.3

*Courtesy United States Bureau of Mines.

Particle Size Distribution

The particle size distribution was determined by a screen analysis. Each chat and slime sample was reduced to the desired size for the analysis by the "quartering" method. The master sample was heaped into a conical pile on a smooth concrete floor. This pile was gradually widened into a flat circle of uniform thickness. The circle was marked into quarters and the two opposite quarters were rejected. The remaining two quarters were mixed together by shoveling the material into a conical pile, taking alternate shovels full from the two quarters. This process of piling, flattening, and rejecting two quarters was continued until the sample was reduced to approximately 300 grams for the slimes and 2500 grams for the chats.

One-third of the slime sample and one-fourth of the chat sample were used for each run. This portion of sample was dumped on the first of a nest of six screens and the screens placed in a Ro-Tap Testing Sieve Shaker. Each sample remained in the Ro-Tap for twenty minutes. The screens were then removed and the amount of material on each screen weighed to an accuracy of one-tenth (0.1) of a gram. Three runs for the slimes and four runs for the chats were made.

Table IIIa
Screen Analysis of Blue Goose Slime

	Sieve Opening	Mesh	Per Cent
Retained on	.0232 in.	28	6.2
" "	.0164	35	9.1
" "	.0116	48	13.2
" "	.0082	65	12.5
" "	.0058	100	15.1
" "	.0029	200	21.3
Passing	.0029	200	22.5

Table IIIb

Screen Analysis of Central Mill Slime

	Sieve Opening	Mesh	Per Cent
Retained on	.0232 in.	28	0.9
" "	.0164	35	3.3
" "	.0116	48	12.5
" "	.0082	65	20.9
" "	.0058	100	24.8
" "	.0029	200	24.1
Passing	.0029	200	12.3

Table IVa

Screen Analysis of Blue Goose Chat

	Sieve Opening	Mesh	Per Cent
Retained on	.185 in.	4	24.7
" "	.093	8	28.2
" "	.046	14	24.6
" "	.0328	20	9.9
" "	.0232	28	5.7
" "	.0164	35	3.2
Passing	.0164	35	3.6

Table IVb

Screen Analysis of Anna Beaver Chat

	Sieve Opening	Mesh	Per Cent
Retained on	.185 in.	4	51.5
" "	.093	8	25.1
" "	.046	14	11.5
" "	.0328	20	4.1
" "	.0232	28	2.7
" "	.0164	35	1.8
Passing	.0164	35	3.3

The remainder of the tests were applied to the Blue Goose and Central Mill slimes only.

Workability

When mixed with water neither slime showed any plasticity. However, test pieces made from them had sufficient bond to retain their shape if handled very gently.

Preparation of the Specimens

The test specimens were made in a wooden mold, $1 \frac{1}{8}$ " x $1 \frac{1}{8}$ " x $12 \frac{1}{2}$ ". The slime was mixed with sufficient water to form a mass that would hold together when forced into the mold by hand. The mold was completely filled with the mix, any excess struck off, and the surface smoothed with a spatula. The bar was then removed from the mold and cut into test pieces $1 \frac{7}{8}$ inches long. The edges of each test piece were smoothed over to prevent sharp corners.

Twenty-four of the 1 7/8 inch specimens were made from each slime. A series of ten 6 inch specimens were also made to be used for the transverse strength tests.

Drying

The test pieces were dried at room temperature for at least twenty-four hours and then dried in an electric oven at 110° Centigrade to constant weight.

Dry Volume

The dried 1 7/8 inch specimens were submerged in kerosene and allowed to soak for at least twelve hours. The volume of each piece was then determined using a Schurecht type of overflow volumeter, as described in the Journal of the American Ceramic Society, Volume III, No. 9, pp. 730-734. The volumes were read to the nearest one-tenth (0.1) of a cubic centimeter.

After the dry volumes were determined, the test pieces were allowed to air dry at room temperature for twenty-four hours and then at 110° Centigrade until all the kerosene was expelled.

Firing Procedure

The 1 7/8 inch specimens were next placed in a refractory muffle in a down-draft gas-fired laboratory kiln, the manner of setting being such as to allow draw trails at progressive temperatures. Pyrometric cones were used on each draw trail plaque and further temperature control was obtained with a platinum-platinum-rhodium thermocouple. Heating was approximately at the rate of 45° Centigrade per hour until a temperature was reached

corresponding to the softening point of the third consecutive cone below that at which the first trial was to be drawn, and at the rate of 20° Centigrade per hour from that point until the end of the firing.

Draw trials were made as rapidly as possible to avoid cooling the kiln unnecessarily. Upon removal from the kiln, the test pieces were immediately covered with sand. When cool enough to handle, they were placed in a desiccator, containing CaCl₂, until they had cooled to room temperature. The last set of test pieces were allowed to cool down in the kiln.

Apparent Porosity

The fired test pieces were weighed on a balance to an accuracy of one-tenth (0.1) of a gram. Then they were placed in distilled water in a suitable vessel, boiled for two hours, and allowed to cool to room temperature while still immersed in the water. Each test piece was dried lightly with a damp towel to remove excess water and weighed in air to an accuracy of one-tenth (0.1) of a gram. The specimens were again dried to constant weight and the fired volumes determined in the same manner as were the dry volumes. The apparent porosity was calculated by means of the formula:

$$P = \frac{Sf - Wf}{Vf} \times 100$$

in which

P = per cent apparent porosity
 Sf = weight of saturated test piece in grams
 Wf = weight of the fired test piece in grams
 Vf = volume of the fired test piece in cubic centimeters

Volume Change

From the dry and fired volumes of the specimens, determined above, the per cent volume change was found using the formula:

$$b_1 = \frac{V_d - V_f}{V_d} \times 100$$

in which

b_1 = per cent volume change
 V_d = volume of dry test piece in cubic centimeters.
 V_f = volume of fired test piece in cubic centimeters.

Apparent Specific Gravity

The apparent specific gravity was calculated by the use of the following formula:

$$G = \frac{W_f}{V_f - (S_f - W_f)}$$

in which

W_f = fired weight of the piece
 V_f = fired volume of the piece
 S_f = saturated fired weight
 G = the apparent specific gravity

Bulk Specific Gravity

The following formula was used to calculate the bulk specific gravity:

$$G_b = \frac{W_f}{V_f}$$

in which

G_b = the bulk specific gravity

Table Va

The Fired Properties of Blue Goose Slime*

Cone	Apparent Porosity	Volume Changes**	Apparent Specific Gravity	Bulk Specific Gravity
4(1190°C.)	40.4%	1.5%	2.48	1.48
6(1230°C.)	40.1	-2.6	2.39	1.35
8(1260°C.)	41.0	-3.3	2.38	1.41
10(1305°C.)	38.7	-5.1	2.33	1.43
12(1335°C.)	37.1	-6.7	2.29	1.40
14(1400°C.)	37.0	-10.9	2.21	1.35
14(1410°C.)	35.8	-11.2	2.20	1.34
15(1420°C.)	43.6	-9.9	2.25	1.35

*Average of 3 tests pieces.

**Negative sign indicates an expansion.

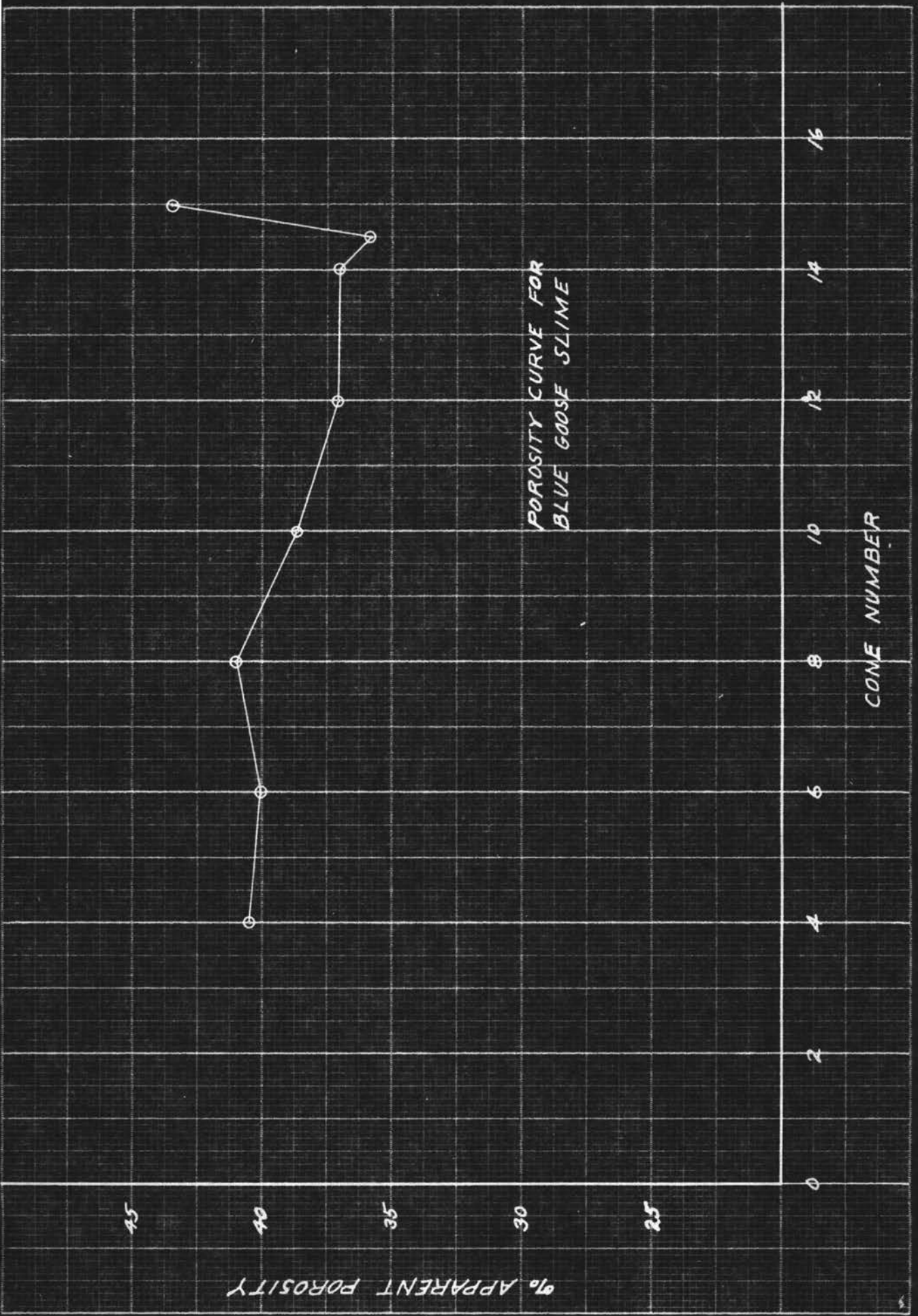


Table Vb
The Fired Properties of Central Mill Slime

Cone	Apparent Porosity*	Volume Change	Apparent Specific Gravity	Bulk Specific Gravity
4(1190°C.)	----	0.9%	---	---
6(1230°C.)	----	1.4	---	---
8(1260°C.)	----	4.7	---	---
10(1305°C.)	46.3	-3.2	---	---
12(1400°C.)	51.0	-5.5	2.34	1.28
14(1410°C.)	52.2	-7.3	2.60	1.28
14(1410°C.)	51.0	-2.6	2.74	1.31
15(1420°C.)		-1.9	2.75	1.35

*Test pieces fired to cones 4, 6, 8, and 10 crumbled during the boiling for the porosity test.

Transverse Strength

The test pieces for the transverse strength tests were made as explained under the Preparation of the Specimens, except that four per cent flour was added as a bond to aid in the handling of the green specimens. A Fairbanks cement testing machine, adapted to the breaking of test bars by the addition of a special stirrup to replace the tensile strength clamps was used to test the pieces.

Steel rollers served as knife edges. The distance between them could be adjusted for either a three or five inch span. The load was applied to a third knife edge placed at the mid-point of the test specimen. The transverse strength, or modulus of rupture, was calculated in pounds per square

inch by use of the formula:

$$M = \frac{3Pl}{2bd^2}$$

in which

M= modulus of rupture
 P= breaking load in pounds
 l= distance between knife edges in inches
 b= breadth of test piece in inches
 d= depth of test piece in inches

Two series of test pieces were used for each slime. One series was fired to cone 16(1465°C.) and the other to cone 18(1490°C.). The final results are an average of five specimens.

Table VI

Results of Transverse Strength Test on the Slimes

Cone	Material	Modulus of Rupture, lbs/in ²
16	Blue Goose slime	150
16	Central Mill slime	144
18	Blue Goose slime	364
18	Central Mill slime	464

Pyrometric Cone Equivalent

The sample of slime was ground in an agate mortar to pass a 65 mesh screen. The test pieces were formed in a steel mold in the shape of tetrahedra, seven millimeters along the edge of the base and thirty millimeters high. Casein glue was used as a bonding agent.

The test pieces were mounted on a refractory plaque with their troweled faces set at an angle of 82° with the plaque and the bases imbedded in the

plaque not more than two millimeters. They were arranged in an alternating order with Seger pyrometric cones of successive numbers. The plaque was then placed in a furnace and the heating carried out as follows:

<u>Time Interval</u>	<u>Minutes</u>
To reach cone 20	45
" " " 23	20
" " " 26	6
" " " 27	4
" " " 28	4
" " " 29	10
" " " 30	4
" " " 31	8
" " " 32	4
" " " 33	8
" " " 34	18
" " " 35	6

The pyrometric cone equivalent is indicated when the tip of the test piece bends over and touches the plaque. The softening point is expressed in terms of the number of the pyrometric cone which corresponds in time of softening with the test piece. The values could not be determined for the slimes because the furnace failed to reach high enough temperatures.

True Specific Gravity

The sample of slime was ground in an agate mortar to pass a 65 mesh screen. About ten grams were prepared and dried to constant weight at 110°

Centigrade. A pycnometer with a stopper containing a capillary tube was dried to constant weight at 110° Centigrade and weighed. It was then filled with distilled water and reweighed. All weighings were made on a chemical balance to the third decimal place in grams.

About two grams of the sample were placed in the pycnometer. The pycnometer was filled to approximately one-third its capacity with distilled water, the stopper put in place, and boiled for 15 minutes with care. The pycnometer was then filled to the base of the neck and allowed to cool to room temperature. When it was cool and the sample settled, the filling was completed, the pycnometer wiped dry with a soft cloth and weighed to the same accuracy as above.

The true specific gravity was calculated from the formula:

$$Gt = \frac{w-p}{(w-p)-(w_2-w_1)}$$

in which

Gt- the true specific gravity
 w- weight of the stoppered pycnometer and sample
 p- weight of the stoppered pycnometer
 w₁- weight of the stoppered pycnometer filled with water
 w₂- weight of the stoppered pycnometer, sample and water

Two determinations gave the following average results:

Blue Goose slime.....2.73

Central Mill slime.....2.95

SUMMARY

Blue Goose and Central Mill slimes are sand-like materials consisting of particles that range in size from slightly larger than 28 mesh to less than 200 mesh on the Tyler Standard Screen Scale. The particles that went through a 200 mesh screen constituted 22.5 per cent of the

Blue Goose slime and 12.3 per cent of the Central Mill slime. The amount that passed a 100 mesh screen, but not a 200 mesh, was 21.3 per cent and 24.1 per cent of Blue Goose and Central Mill slimes, respectively.

When the slimes were mixed with water, they showed no plasticity. The finer grains afforded some bond to the specimens, but this bond was not sufficient to allow much handling of the dry specimens.

The results of the volume changes revealed a shrinkage in the Blue Goose specimens at cone 4, but an expansion from cone 6 through cone 14. The Central Mill specimens had a shrinkage at cone 4 and through cone 8. The test pieces drawn from the kiln at cones 10, 12 and 14 had expanded. Those specimens which were allowed to cool in the kiln expanded less than the cone 14 draw trials.

Porosity changes were somewhat irregular. Progressive changes in the porosity of the Central Mill slime below cone 12 could not be determined, for the test pieces crumbled badly in contact with moisture. At the higher cone temperatures, the apparent porosity of the Central Mill specimens was greater than that of the Blue Goose specimens. The highest porosity value for the Blue Goose test pieces was 43.6 per cent at slightly above cone 14. The value for the Central Mill pieces at the equivalent temperature was 52.5 per cent.

All the test specimens fired to a light gray color except the Central Mill pieces that were allowed to cool in the kiln. These specimens turned a light brown.

The Central Mill specimens had greater strength than the Blue Goose specimens when tested for modulus of rupture. No outstanding defects were noticed in either set of test pieces. The greater strength of the central Mill specimens was caused by a more complete fusion of the material and better cementing of the grains, due to the higher amount of impurities (calcium oxide and magnesium oxide) in the Central Mill slime.

CONCLUSIONS

As a result of the testing of the slimes, the following conclusions were reached:

(1) The better bonding of the Blue Goose slime at low temperatures (below glass formation) is due to the higher percentage of fine grain sizes distributed throughout the slime.

(2) Specimens made from Blue Goose slime expand more than pieces made from the Central Mill slime. This is caused by the higher percentage of silica (93% as compared with 81%) in the Blue Goose slime.

(3) The large amounts of calcium oxide and magnesium oxide in the Central Mill slime lowers the fusion point below that desirable for a suitable refractory material.

(4) Neither of the slimes can be used to form bricks, or similar products, without the use of a bonding agent to furnish strength in the unfired state.

CHAPTER III

The Use of Slimes for Silica Brick

Silica brick are refractories for use in the construction of open-hearth and electric steel furnaces, by-product coke ovens, gas retorts, glass tanks, copper reverberatory and refining furnaces and vertical lime kilns.¹ Great mechanical strength and resistance to abrasive action, rigidity at high temperatures, high refractoriness, high thermal conductivity, a definite thermal expansion, and high resistance to corrosion by acid slags are the properties responsible for the service rendered by a high quality silica brick.

In commercial practice, the raw material used in the manufacture of these brick is a crystalline form of silica called quartzite. The quartzite, crushed to pass through a one-half inch ring, is tempered in a wet pan with sufficient water to keep down the dust. Lime, in the form of milk of lime, is added as a bonding material. The brick are formed by either the soft-mud process, in which the mixture is poured into molds and bumped into shape, or by dry pressing. After they have been formed, the brick are dried completely and then carefully fired to a temperature of approximately 1485° Centigrade. The finished brick are a yellow-white color.

The quartzites used in the high quality silica brick contain from 97 to 99 per cent silica (SiO_2). Blue Goose slime contains 93 per cent silica. In spite of the lower silica content, it was decided that by the selection and proper distribution of grain sizes and the use of the proper amount and type of bonding agent, this slime might be employed to manufacture a suitable re-

¹Harbison-Walker, Modern Refractory Practice, p. 42

fractory that would find enough uses to make it practical.

The following portion of this investigation is a discussion of the work done in the attempt to produce a refractory brick from Blue Goose slime. Both the soft-mud process and the dry press process were employed to form the test specimens.

STUDY OF GRAIN SIZING

One of the fundamental considerations in the manufacture of refractories is the size of the grains of the raw material. The distribution of grain sizes in the mixes has an important effect on many of the physical properties of the finished product. Spalling resistance and the ability to carry loads at high temperatures are improved by the proper distribution of grain sizes. The unfired and fired strengths are also influenced.²

A study was made of the influence of the various grain sizes on the unfired and fired strength of silica brick prepared from mixes using the Blue Goose slime. This was done by bonding various grain sizes with a constant percentage of bonding agent and then determining the modulus of rupture of the specimens.

To obtain the desired grain sizes, the slime was pulverized in a Braun laboratory pulverizer and put through a series of Tyler screens. Any particles of magnetic material present after pulverizing were removed with a magnet.

Bonding Agent

A commercial grade of quicklime was selected as the bonding medium. For the dry pressed pieces, the proportion was 3 parts lime to 97 parts slime.

²Greaves-Walker and Stone, The Production of Unfired and Fired Forsterite Refractories from North Carolina Dunites, p. 33.

For the specimens formed by the soft-mud process, the proportion was 2 parts lime to 100 parts slime.

Mixes Employed

The amount of the bonding agent was kept constant and the size and the proportion of grains were varied. The proportion of grain sizes is shown in the following table:

Table VI
The Composition of the Mixes Investigated

Series	Composition
AA	97 parts slime as-received 3 parts lime
AB	97 parts slime through 28 on 65 mesh* 3 parts lime
AC	97 parts slime through 65 on 100 mesh 3 parts lime
AD	97 parts slime through 65 mesh 3 parts lime
AE	97 parts slime through 100 mesh 3 parts lime
AF	48.5 parts slime through 28 on 65 mesh 48.5 parts slime through 100 mesh 3 parts lime
AG	48.5 parts slime through 28 on 65 48.5 parts slime through 65 mesh 3 parts lime
AH	97 parts slime through 28 mesh 3 parts lime
AU	100 parts slime as-received 2 parts lime
AV	100 parts slime through 28 on 65 mesh 2 parts lime
AW	100 parts slime through 65 mesh 2 parts lime

*The Tyler Standard Screen Scale.

Preparation of the Specimens

The specimens in series AA to AH were formed by dry pressing in a Fred S. Carver hydraulic laboratory press under a pressure of 10,000 pounds per square inch. In all mixes, the batch consisted of 528 grams of slime, 18 grams of lime, and 60 cubic centimeters of distilled water. The slime and lime were mixed thoroughly in the dry condition and then added to the water. Mixing was continued until a uniformly moist mix was obtained. The dry pressed specimens were $2 \frac{3}{16}$ inches wide by $4 \frac{3}{16}$ inches long by approximately $\frac{7}{16}$ inches thick. Six specimens were made from each mix.

Series AU to AW were made by the soft-mud process. They were molded by hand in a wooden mold to a size approximately $1 \frac{1}{8}$ inches square by $5 \frac{1}{2}$ inches long. In each batch, which contained 2500 grams of slime and 50 grams of lime, the slime and lime were mixed thoroughly and sufficient water added to form a mix that molded easily, but did not slump when removed from the mold. Twelve specimens of each mix were made.

Drying

The dry pressed specimens were dried at room temperature for 36 hours and in a dryer at 170° Fahrenheit for 5 hours.

The soft-mud specimens were dried as follows:

AV--8 days at room temperature. 12 hours at 100-110° Centigrade.

AU--48 hours at room temperature. 4 hours at 100-110° Centigrade in an electric oven

AW--4 days at room temperature. 4 hours at 100-110° Centigrade.

Two of the dry pressed specimens were selected from each series for the unfired tests and two for the fired tests. Four of the soft-mud pieces were selected for the unfired test, four for a cone 16 firing and four for a cone 18 firing.

Firing

The specimens to be fired were placed in a refractory muffle in a down-draft gas-fired laboratory kiln. A one-fourth inch space was left between each piece to allow for any expansion of the pieces during the firing. The dry pressed specimens were fired to 1440° Centigrade. One set of soft-mud pieces was fired to cone 16(1465°C.) and the other to cone 18(1490°C.).

Testing

(1) Modulus of rupture

To determine the force necessary to break each dry pressed test piece, the piece was suspended flatwise on two parallel knife edges. A third knife edge with attachments for applying weights was brought to bear on the suspended piece, midway between the two bottom knife edges and on the opposite side of the specimen. Weights were added until the test piece broke. The soft-mud pieces were broken in a Fairbanks testing machine.

The modulus of rupture was calculated in pounds per square inch using the following formula:

$$\text{Modulus of rupture} = \frac{3Pl}{2bd^2}$$

in which

P= the breaking force in pounds
 l= distance between the knife edges in inches
 b= breadth of specimen in inches
 d= depth of specimen in inches

(2) Linear expansion

The linear expansion was calculated for the dry pressed pieces only. The length of the piece was measured before the piece was fired and again after the piece was fired. The per cent linear expansion, based on the dry length, was determined from:

$$\% \text{Linear Expansion} = \frac{L_f - L_d}{L_d} \times 100$$

in which

L_f = length fired in centimeters
 L_d = length dry in centimeters

The results of these tests are given in tables VII and VIII. Working qualities at the press included the degree of ease with which the specimens could be removed from the mold and the apparent firmness of the specimens immediately after pressing. Hardness of the edges was their resistance to chipping or rubbing away when scratched with the fingernail. Color in the fired pieces was based on the number and size of any red spots or splotches on the piece. Those rated excellent were free of these blemishes.

Table VIIa

Unfired Properties of the Dry Pressed Grain Size Mixes

Series	Working Qualities at Press	Hardness of Edges	Defects	Modulus of Rupture, lbs/in. ² *
AA	Good	Fair	None	19
AB	Fair	Poor	None	7
AC	Fair	Poor	Slight Striations	9
AD	Excellent	Fair	None	29
AE	Excellent	Fair	None	32
AF	Excellent	Fair	None	26
AG	Excellent	Fair	None	20
AH	Good	Fair	None	18

*Average of two specimens.

Table VIIb

Unfired Properties of the Soft-Mud Process Grain Size Mixes

Series	Work-ability	Hardness of Edges	Defects	Modulus of Rupture, lbs/in. *
AU	Good	Fair	None	29
AV	Good	Fair	None	31
AW	Good	Fair	None	54

Table VIIIa

Fired Properties of the Dry Pressed Grain Size Mixes. (1440°C.)

Series	Hardness of Edges	Color	Per Cent Linear Expansion	Modulus of Rupture, lbs/in. *
AA	Fair	Fair	5.2	396
AB	Poor	Fair	5.8	176
AC	Poor	Good	4.7	252
AD	Good	Excellent	4.1	482
AE	Good	Excellent	3.5	942
AF	Fair	Fair	4.7	462
AG	Good	Good	4.7	492
AH	Fair	Fair	5.1	355

*Average of two specimens.

Table VIIIb
Fired Properties of Soft-Mud Process Grain Size Mixes
(1465 and 1490°C.)

Series	Color	Hardness of Edges	Modulus of Rupture, lbs/in.*	
			Cone 16	Cone 18
AU	Fair	Good	479	495
AV	Poor	Poor	275	151
AW	Good	Good	980	734

*Average of four specimens.

Summary

The dry pressed specimens pressed easily and slid off the die plates without difficulty. They broke readily if handled in the wet condition, but developed strength during the drying period.

Grain size distribution had a definite effect on the green and fired strength of the test pieces. The maximum size of the coarse material was through 28 on 65 mesh. Unfired specimens made from this material were unsatisfactory, for they had very little strength. The fired specimens in the same series (series AB) were also low in strength.

The size of the fine particles was through 100 mesh. Specimens formed from this material were the greatest in strength in both the green and fired state (series AE). Other mixes yielded specimens with green and fired strengths that varied between the limits set by the coarse and fine grain size series mentioned.

The color of all specimens was the typical yellow-white of silica brick. Those containing coarse particles were impregnated with red spots

or splotches due to metal impurities. The fine particles formed test pieces that were free from these spots.

Conclusions

The dry pressed silica brick specimen with the best grain size distribution had the following composition:

97 parts slime through 100 mesh.
3 parts lime.

The best soft-mud mix was:

100 parts slime through 65 mesh.
2 parts lime.

These were selected because of (1) strength in the green condition (2) strength in the fired state (3) hardness of edges and (4) color.

STUDY OF BONDING AGENTS

The Bonding Agent

Before the slime could be used to form silica brick, some bonding agent had to be employed to give the brick strength. This bonding agent had to meet the following requirements:

(1) Develop strength in the green (unfired) brick sufficient to withstand handling and stacking in the kiln.

(2) Lower to no great extent the ability of the brick to withstand load at high temperatures.

(3) Lose none of its bonding properties until the fired brick has fused sufficiently to retain its shape under load.

Types of Bonding Agents

Numerous bonding agents were capable of bonding the non-plastic slime grains into brick. These were portland cement; Lummite cement; sodium

silicates; organic binders, such as gums, flour, etc; clays; chemicals bonds, such as lime, oxychlorides, etc.; and colloids. From this group the following were selected for use in the investigation: (1) Lime, (2) gypsum, (3) sodium silicate, (4) wheat flour, and (5) Lumnite cement.

Four brands of sodium silicate were used. Their compositions were as follows:*

<u>Sales Name</u>	<u>Ratio Na₂O:SiO₂</u>	<u>Na₂O</u>	<u>SiO₂</u>	<u>°Baume</u>
U	1:2.40	13.80	33.1	52.0
O	1:3.22	9.16	29.5	42.2
G	1:3.22	19.40	62.5	17.5% H ₂ O
G-C	1:2.00	27.50	55.0	17.5% H ₂ O

The composition of the Lumnite cement was the following:**

Insoluble residuo.....	0.6
TiO ₂	1.8
SiO ₂	7.5
Al ₂ O ₃	38.0
FeO.....	7.9
Fe ₂ O ₃	5.9
CaO.....	36.3
MgO.....	1.2
SO ₃	0.1
S.....	0.3

The lime was a commercial grade of quicklime. The gypsum was plaster of paris.

Mixes Investigated

The composition of the mixes investigated are given in detail in table IX and table X.

*Courtesy of Philadelphia Quartz Company.

**Courtesy of the Atlas Lumnite Cement Company.

Table IX

Per Cent Bonding Agent in Mixes Investigated Dry Pressed Specimens

Series	Composition
AI-1	98 parts slime 2 parts lime
AI-2	97 parts slime 3 parts lime
AI-3	96 parts slime 4 parts lime
AJ-1	98 parts slime 2 parts "O" Brand sodium silicate
AJ-2	97 parts slime 3 parts "O" Brand sodium silicate
AJ-3	95 parts slime 5 parts "O" Brand sodium silicate
AK-1	97 parts slime 3 parts gypsum
AK-2	95 parts slime 5 parts gypsum
AK-3	93 parts slime 7 parts gypsum
AL-1	97 parts slime 3 parts Lumite cement
AL-2	95 parts slime 5 parts Lumite cement
AL-3	93 parts slime 7 parts Lumite cement

Table X

Per Cent Bonding Agent in Mixes Investigated Soft-Mud Process Specimens

Series	Composition
AM	100 parts slime 2 parts lime
AN	100 parts slime 4 parts wheat flour
AO	100 parts slime 3 parts "O" Brand sodium silicate
AP	100 parts slime 3 parts "G" Brand sodium silicate
AQ	100 parts slime 3 parts "G-C" Brand sodium silicate
AR	100 parts slime 3 parts "U" Brand sodium silicate
AS	100 parts slime 3 parts Lumnite cement

Preparation of the Test Specimens

The dry pressed specimens were formed under a pressure of 10,000 lbs/in.² using a Fred S. Carver hydraulic laboratory press. Each total batch weight was 400 grams. Forty cubic centimeters of water were used in the mixing. The specimens (four of each mix) were 2 3/16 inches wide by 4 3/16 inches long by approximately 7/16 inches thick.

The soft-mud process specimens were molded in a wooden mold. 2500 grams of slime were used in each batch composition. Twelve specimens 1 1/16 inches wide by 1 1/8 inches thick by 5 1/2 inches long, were formed. In each instance

the slime and bonding agent were mixed thoroughly and sufficient water added to form a mix that could be molded easily without slumping when removed from the mold.

Drying

The dry press specimens were dried at room temperature for 36 hours and in the dryer at 170° Fahrenheit for 5 hours. Two test pieces of each series were selected for unfired tests and two for fired tests.

The drying schedule for the soft-mud specimens was as follows:

AM--48 hours at room temperature. 4 hours in an electric oven at 100-110° Centigrade.

AN--24 hours at room temperature. 4 hours at 100-110° Centigrade.

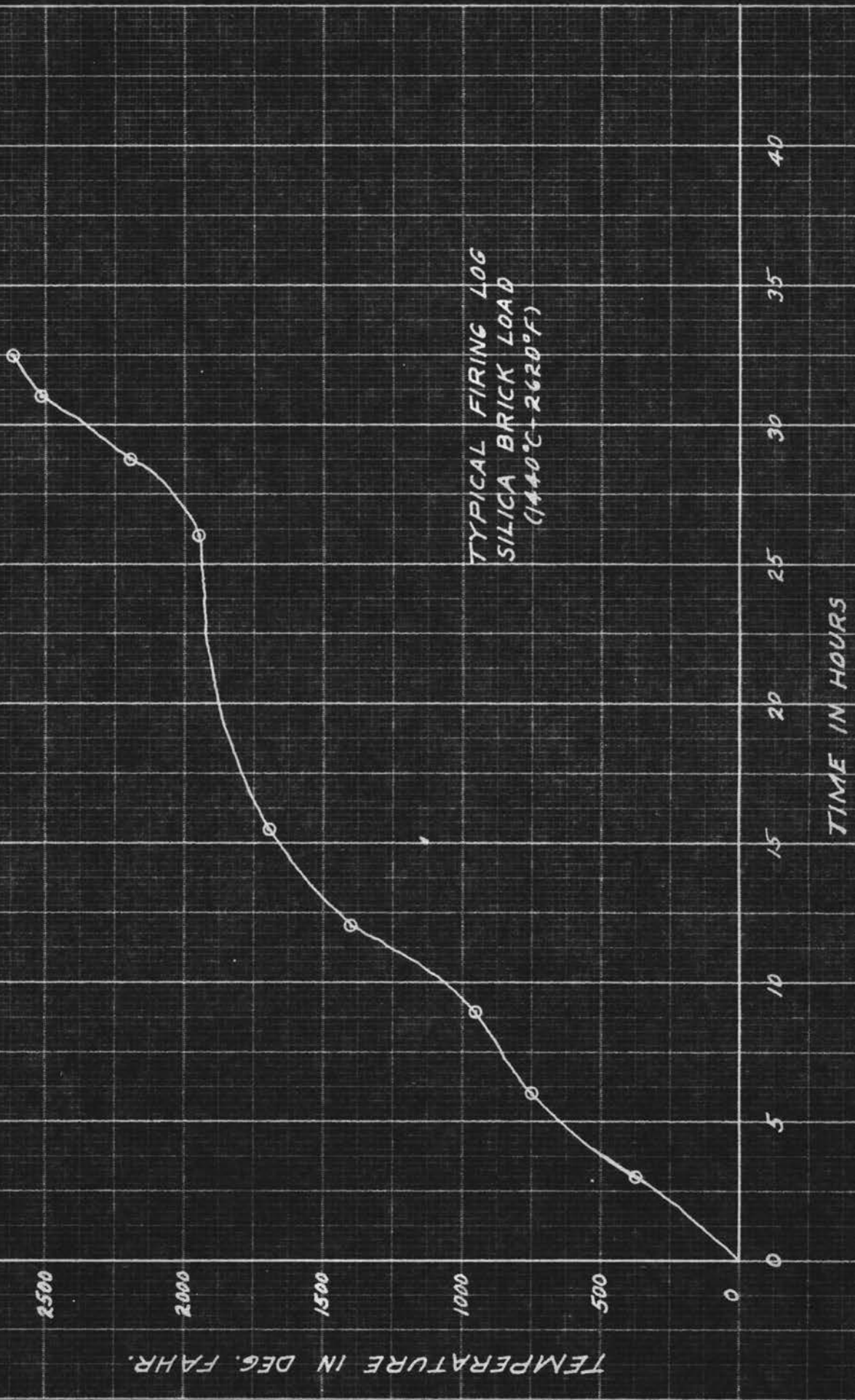
AO--12 hours at room temperature. 24 hours at 70° Centigrade. 3 hours at 100-110° Centigrade.

AP--Covered with a damp cloth immediately after forming and left at room temperature for 12 hours. 3 hours at room temperature with the damp cloth removed. 24 hours at 30-40° Centigrade. 3 hours at 100-110° Centigrade.

AQ--Covered with a damp cloth immediately after forming and left at room temperature for 12 hours. 3 hours at 100-110° Centigrade.

AR--Dried in a manner similar to that used for AP.

AS--Covered with a damp cloth immediately after forming and left at room temperature for 24 hours. 48 hours at room temperature with cloth removed. Specimens 1 to 4 dried at room temperature for 24 additional hours while specimens 5 to 12 were recovered with a damp cloth. All specimens then dried for 4 hours at 100-110° Centigrade.



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Firing

The dry pressed pieces were fired to 1440° Centigrade. One set of the soft-mud specimens was fired to cone 16(1465°C.) and the other set to cone 18(1490°C.). The specimens were placed in a refractory muffle in a down-draft gas-fired laboratory kiln. A one-fourth inch space separated each piece to allow for expansion. The rate of heating is shown in the firing curves.

Testing

The modulus of rupture for the dry pressed and the soft-mud process specimens was obtained in the manner explained under the testing of grain size mixes, page 24.

In addition to the modulus of rupture test, the absorption test was made on the soft-mud specimens. One-half of each of the transverse strength test pieces was dried at 100-110° Centigrade to constant weight and weighed to an accuracy of one-tenth (0.1) of a gram. The pieces were then immersed in distilled water and boiled for two hours. They were allowed to cool to room temperature in the water. Excess moisture was removed from the surface of the pieces with a damp cloth and they were weighed to the same accuracy as before. The per cent absorption was calculated from the following formula:

$$\text{Per cent absorption} = \frac{W_s - W_d}{W_d} \times 100$$

in which

W_s = weight in grams of saturated specimen

W_d = weight in grams of dry specimen

The results of these tests are given in tables XI and XII. If the color of the specimen was the characteristic yellow-white, it was rated according to the degree of freedom from spots due to impurities as poor, fair, good, or excellent. If the specimen was not yellow-white, the actual color was given. Other column headings have the same significance as those in tables VII and VIII.

Table XIa

Unfired properties of Mixes Containing Bonding Agents Dry Pressed Specimens

Series	Working Qualities at Press	Hardness of Edges	Defects	Modulus of Rupture, lbs/in. ² *
AI-1	Fair	Poor	None	8.6
AI-2	Good	Fair	None	18.
AI-3	Good	Fair	None	20.2
AJ-1	Fair	Fair	None	11.9
AJ-2	Fair	Good	None	28.2
AJ-3	Fair	Excellent	None	85.2
AK-1	Fair	Poor	None	8.
AK-2	Good	Fair	None	41.9
AK-3	Good	Fair	None	58.3
AL-1	Fair	Poor	None	14.2
AL-2	Good	Fair	None	20.2
AL-3	Good	Fair	None	20.5

*Average of two specimens.

Table XIb

Unfired Properties of Mixes Containing Bonding Agents Soft-Mud Specimens

Series	Workability	Hardness of Edges	Defects	Modulus of Rupture, lbs/in. ² *
AM	Good	Fair	None	29
AN	Good	Good	None	56
AO	Good	Good	None	53
AP	Good	Excellent	Cracked slightly	100
AQ	Good	Good	Bottom faces cracked	
AR	Good	Good	Bottom faces cracked	
AS	Good	Fair	None	31

*Average of four specimens.

Fired Properties of Mixes Containing Bonding Agents Dry Pressed Specimens
(1440°C.)

Series	Hardness of Edges	Color	Linear Expansion	Modulus of Rupture lbs/in. ² *
AI-1	Fair	Good	5.1	159
AI-2	Fair	Fair	5.1	355
AI-3	Good	Poor	5.2	387
AJ-1	Fair	Gray	5.0	184
AJ-2	Good	Gray	5.2	218
AJ-3	Good	Gray	5.0	254
AK-1	Fair	Good	5.1	227
AK-2	Fair	Fair	5.2	270
AK-3	Good	Poor	5.1	359
AL-1	Fair	Tan	5.1	163
AL-2	Fair	Tan	5.1	125
AL-3	Fair	Tan	5.2	138

*Average of two specimens.

Table XIIb

Fired Properties of Mixes Containing Bonding Agents Soft-Mud Process
(Cone 16-1465°C.)

Series	Hardness of Edges	Color	Per Cent Absorption*	Modulus of Rupture, lbs/in. ² *
AM	Good	Poor	29.2	479
AN	Fair	Fair	34.7	150
AO	Good	Gray	30.8	218
AP		Cracked badly when fired		
AQ		Cracked badly when fired		
AR		Cracked badly when fired		
AS	Good	Tan	29.7	479

*Average of four specimens.

Table XIIc

Fired Properties of Mixes Containing Bonding Agents Soft-Mud Process
(Cone 18-1490°C.)

Series	Hardness of Edges	Color	Per Cent Absorption*	Modulus of Rupture, lbs/in. ² *
AM	Good	Fair	27.8	495
AN	Good	Good	30.7	364
AO	Good	Gray	----	146
AP		Cracked badly when fired		
AQ		Cracked badly when fired		
AR		Cracked badly when fired		
AS	Good	tan	29.5	510

*Average of four specimens.

Summary

The following is a brief summary of the properties, both unfired and fired, of the bonding agent mixes. The slime was as-received material for all soft-mud mixes and through 28 mesh for all dry pressed mixes.

1. Series AI

Bonding Agent: Lime.

Workability: Satisfactory in all proportions.

Drying: No difficulties.

Transverse Strength: Both the unfired and fired strengths increased with the increase in per cent of lime. The maximum green strength was 20 lbs/in.²; the maximum fired strength, 387 lbs/in.².

Fired Color: Yellow-white with a few red spots and splotches. These red splotches were most numerous in the specimens containing the highest percentage of lime.

2. Series AJ

Bonding Agent: "O" Brand sodium silicate.

Workability: All specimens tended to stick slightly to the bottom mold plate. Oil was used to prevent the sticking.

Drying: No difficulties.

Transverse Strength: Both the unfired and fired strengths increased with the increase in per cent of sodium silicate. The maximum green strength was 85 lbs/in.²; the maximum fired strength, 254 lbs/in.².

Fired Color: Gray with a few yellow spots. With an increase in per cent of sodium silicate, the gray color became lighter and the spots fewer.

3. Series AK

Bonding Agent: Gypsum.

Workability: Satisfactory in all proportions.

Drying: No difficulties.

Transverse Strength: Both the unfired and fired strengths increased with the increase in per cent of gypsum. The maximum green strength was 58 lbs/in.²; the maximum fired strength, 359 lbs/in.².

Fired Color: Yellow-white with a few red spots and splotches. The splotches became more numerous with an increase in per cent of gypsum.

4. Series AL

Bonding Agent: Lummite cement.

Workability: Satisfactory in all proportions.

Drying: Precautions had to be taken to prevent evaporation of the water before crystallization was complete.

Transverse Strength: The unfired transverse strength increased with an increase in the per cent of Lummite cement. The maximum value was 21 lbs/in.². The fired strength was irregular with the specimens with 5 per cent Lummite cement having the greatest strength. This value was 163 lbs/in.².

5. Series AM

Bonding Agent: Lime.

Workability: Satisfactory.

Drying: No difficulties.

Transverse Strength: Unfired, 29 lbs/in.²; fired to cone 16(1465°C.), 479, lbs/in.²; fired to cone 18(1490°C.), 495 lbs/in.².

Absorption: Cone 16, 29.2 per cent; cone 18, 27.8 per cent.

Fired Color: Reddish yellow with red spots.

6. Series AN

Bonding Agent: Wheat flour.

Workability: Too much water made the mixes rubbery.

Drying: No difficulties.

Transverse Strength: Unfired, 58 lbs/in.²; cone 16, 150, lbs/in.²; cone 18, 364 lbs/in.².

Absorption: Cone 16, 34.7 per cent; cone 18, 30.7 per cent.

Fired Color: Reddish white color with red spots.

7. Series AO

Bonding Agent: "O" Brand sodium silicate.

Workability: Satisfactory.

Drying: Bottom face on which the specimen rested tended to crack slightly if the piece was not dried carefully.

Transverse Strength: Unfired, 53 lbs/in.²; cone 16, 218 lbs/in.²; cone 18, 146 lbs/in.². The cone 18 specimens cracked slightly when fired.

Absorption: Cone 16, 30.8 per cent; cone 18, not determined because of cracks in the specimens.

Fired Color: Gray with a few dark spots near the bottom edges.

8. Series AP

Bonding Agent: "G" Brand sodium silicate.

Workability: Satisfactory.

Drying: Specimens cracked. Scumming occurred if the specimens were air dried.

Transverse Strength: Not determined because of cracked test pieces.

Absorption: Not determined.

Fired Color: Light gray.

9. Series AQ

Bonding Agent: "G-C" Brand sodium silicate.

Workability: Satisfactory.

Drying: Specimens cracked. Scumming occurred if the specimens were air dried.

Transverse Strength: Not determined because of cracked test pieces.

Absorption: Not determined.

Fired Color: Light gray.

10. Series AR

Bonding Agent: "U" Brand sodium silicate.

Workability: Satisfactory.

Drying: Specimens cracked. Scumming occurred if the pieces were air dried.

Transverse Strength: Not determined because of cracked test pieces.

Absorption: Not determined.

Fired Color: Light gray.

11. Series AS

Bonding Agent: Lumnite cement.

Workability: Satisfactory.

Drying: Precautions had to be taken to prevent evaporation of the water before crystallization was complete.

Transverse Strength: Unfired, 31 lbs/in.²; cone 16, 479 lbs/in.²; cone 18, 510 lbs/in.².

Absorption: Cone 16, 29.7 per cent; cone 18, 29.5 per cent.

Fired Color: Tan with a few red spots.

CONCLUSIONS

The greatest unfired strength is obtained with sodium silicate. The "O" Brand is the most satisfactory. The other brands cause cracking and scumming in drying.

In dry pressed specimens, the greatest fired strength is obtained with lime and gypsum. Lime and Lumnite cement offer the greatest strength in soft-mud process specimens. Lumnite cement mixes require careful drying to develop maximum strength.

Specimens made by the soft-mud process have greater unfired and fired strength than specimens made by the dry press process with an equal amount of bonding agent. One exception is the fired strength given by "O" Brand sodium silicate. This is greater for the dry pressed specimens.

SLIME-BAUXITE MIXTURES

In connection with the work with silica brick, a series of slime-bauxite mixes were prepared to determine the effect of bauxite as a bond for silica brick. A slime-bauxite mix that might yield a mullite brick was also considered, but lack of equipment made it impossible to discover whether, or not, mullite had been formed. Mullite brick is a super-refractory with the formula $3Al_2O_3 \cdot 2SiO_2$.

The bauxite was supplied by the Republic Mining and Manufacturing Company of Bauxite, Arkansas. A pulverized form of the Alum Grade bauxite was used. Its probable analysis was as follows:

Fe ₂ O ₃	2.5-2.75%
SiO ₂	6-8
Al ₂ O ₃	58-59
Moisture.....	1.5-2

Mixes Investigation

The compositions of the mixes investigated are given in the following table:

Table XIV
Per Cent Bauxite In The Mixes Investigated

Series	Composition
SA-1	100 parts slime 2 parts bauxite
SA-2	100 parts slime 4 parts bauxite
SA-3	100 parts slime 6 parts bauxite
SA-4	100 parts slime 8 parts bauxite
SA-5	100 parts slime 10 parts bauxite
SA-6	100 parts slime 15 parts bauxite
SA-7	100 parts slime 20 parts bauxite
SA-8	100 parts slime 25 parts bauxite
SA-M	100 parts bauxite 35.5 parts slime

Preparation of the Test Specimens

Each batch consisted of 300 grams of as-received Blue Goose slime, the proper amount of bauxite, and 10 per cent by weight of water. The slime and bauxite were mixed together thoroughly in the dry state and then added to the water. Mixing was continued until a uniformly moist mixture was obtained. The specimens were molded in a hydraulic laboratory press under a pressure of 10,000 pounds per square inch. Two specimens of each mix were made. They were $2 \frac{3}{16}$ inches wide by $4 \frac{3}{16}$ inches long by approximately $\frac{9}{16}$ inches thick.

Drying

The specimens were dried at room temperature for 12 hours and then in a dryer at 170° Fahrenheit for 12 hours.

Firing

The firing was done in a gas-fired laboratory kiln. Cone 16 ($1465^{\circ}\text{C}.$) was obtained.

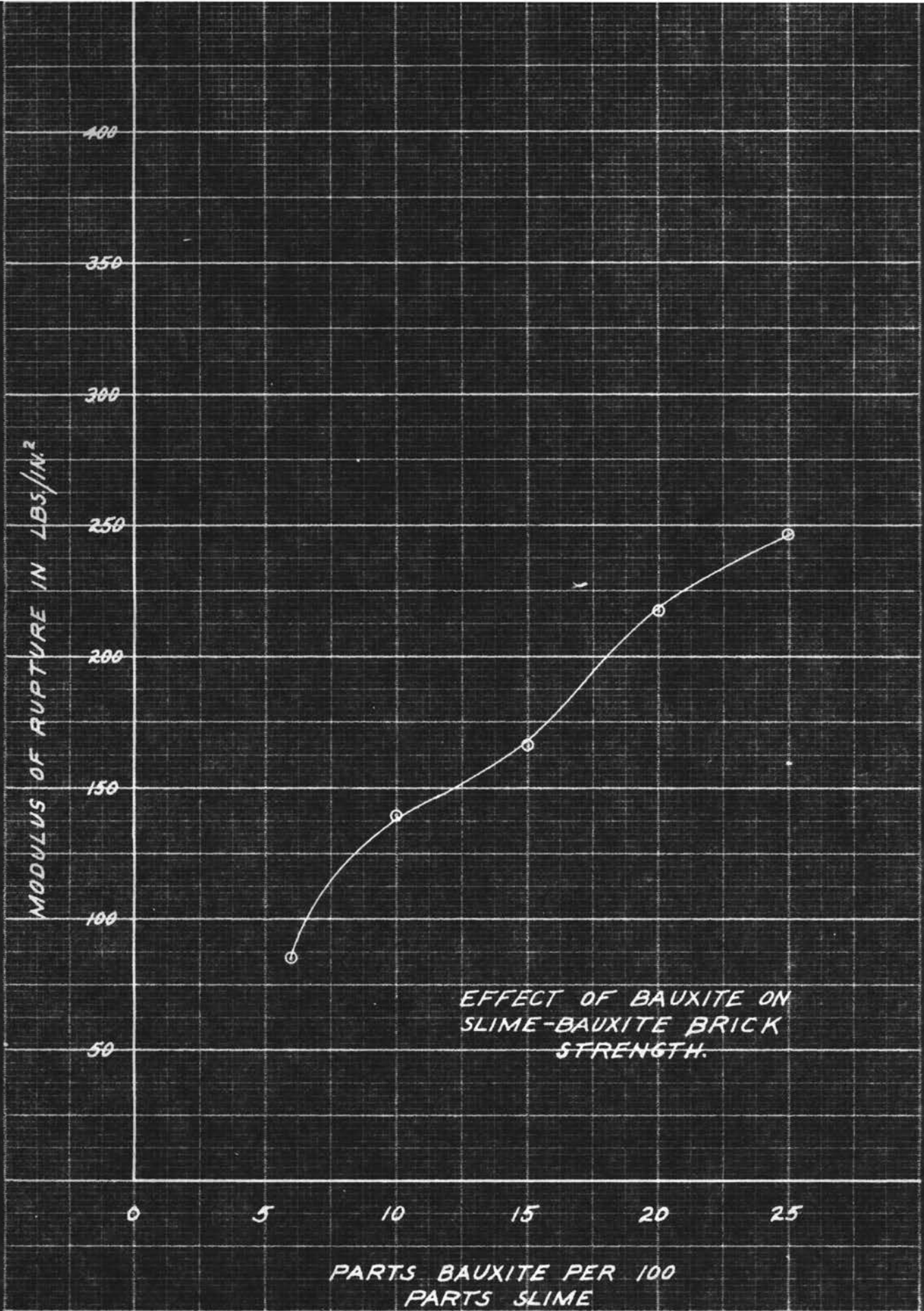
Testing

The only test made on the slime-bauxite specimens was modulus of rupture. The results are given in Table XV.

Table XV

Fired Properties of Slime-Bauxite Mixes Investigated (Cone 16- $1465^{\circ}\text{C}.$)

Series	Parts Bauxite per 100 parts Slime	Hardness of Edges	Modulus of Rupture lbs/in. ²
SA-1	2	No bond--Broke before firing	
SA-2	4	No bond--Broke before firing	



EFFECT OF BAUXITE ON
SLIME-BAUXITE BRICK
STRENGTH.

PARTS BAUXITE PER 100
PARTS SLIME

Table XV Continued

Fired Properties of Slime-Bauxite Mixes Investigated (Cone 16-1465°C.)

Series	Parts Bauxite per 100 parts Slime	Hardness of Edges	Modulus of Rupture lbs/in.
SA-3	6	Fair	84*
SA-4	8	Fair	151**
SA-5	10	Good	140*
SA-6	15	Good	167***
SA-7	20	Very Good	217**
SA-8	25	Excellent	246***

*Average of two specimens.

**Value for one specimen only. Second specimen defective.

***Average of three specimens.

Summary

The addition of increasing amounts of bauxite increased the strength of the slime-bauxite specimens (see curve). The specimens fired to a brown color. This color became darker with higher percentages of bauxite.

The mullite mix (series SA-M) was a very dark brown when fired. The edges were good and the modulus of rupture was 245 lbs/in.².

The slime-bauxite mixes had satisfactory workability. In the mullite mix, the addition of slime reduced the rubbery consistency that makes bauxite difficult to mold when a slight excess of water is added.

CHAPTER IV

Sand-Lime Brick

Sand-lime brick is a type of brick widely used as a building material for masonry walls. These brick have a pleasing light sand-stone-like finish and, if they are of high grade, have a quality similar to face brick. As the name "sand-lime" implies, the main raw materials used in the manufacture of the brick are sand and lime. The mixture is shaped into brick by dry pressing and the brick are hardened by treatment with steam. There is evidence of a reaction between the lime and the sand during the heat treatment. Hydrated bi-calcium silicates are formed.

There are three commercial grades of sand-lime brick. The specifications of the American Society for Testing Materials designated these as follows:¹

1. Grade SW---"Brick intended for use where exposed to temperature below freezing in the presence of moisture." Example; Brick used for foundation courses and parapets in the northeast quarter of the United States.

2. Grade MW---"Brick intended for use where exposed to temperature below freezing but unlikely to be saturated with water." Example; Brick exposed in the face of a wall other than foundations or parapets, or brick intended for use in structures located in regions of the United States characterized by less severe frost action or by drier climate than is found in the northeast quarter of the United States.

¹A. S. T. M. Designation; C73-39.

3. Grade NW---"Brick intended for use as back-up or interior masonry or if exposed, for use where no frost action occurs; or if frost action occurs, where the average annual precipitation is less than 15 inches."

The values for the modulus of rupture of these grades are:

<u>Grade</u>	<u>Average of Five Brick</u>	<u>Individual Minimum</u>
SW	600 lbs/in. ²	400 lbs/in. ²
MW	450 "	300 "
NW	300 "	200 "

The Blue Goose and Central Mill slimes resemble in many ways the sands commercially used in the manufacture of sand-lime brick. Therefore, it was possible to attempt the making of sand-lime brick substituting the slime for sand. Numerous mixes were prepared and tested. The variable factors considered were (1) percentage of lime used as the bond, (2) the amount of forming pressure to dry press the specimens, (3) the temperature and length of steam treatment, and (4) grain sizes.

GENERAL CONSIDERATIONS

Preparation of Test Specimens

All test specimens used in the investigation of sand-lime brick mixes were formed with a Fred S. Carver hydraulic laboratory press. With the exception of the series of specimens used to determine the effect of variable forming pressures, the specimens were pressed at 10,000 pounds per square inch.

The sand and lime were mixed together thoroughly in the dry condition and then added to the water. Sufficient water was used to slake the lime and leave the mix with ten per cent moisture. Mixing was continued until a uniformly moist mixture was obtained. The specimens were 2 3/16 inches

wide and $4 \frac{3}{16}$ inches long. The thicknesses of the many specimens varied from $\frac{3}{8}$ to $\frac{15}{16}$.

Drying

The specimens were allowed to dry at room temperature 12 to 15 hours.

Steam Treatment

The steam treatment was accomplished in an autoclave made from a sixteen inch length of standard ten inch pipe. One end of the pipe was sealed with a welded steel plate. A ten hole standard flange was attached to the other end. Three inches from the bottom, a three inch pipe coupling was welded into a hole in the side of the pipe section. A hole for attaching a steam trap was drilled and tapped in the bottom plate and a smaller hole for the installation of a pressure gauge drilled and tapped near the top of the pipe. A circular steel plate, with holes drilled to match those in the flange, constituted the cover. The cover was separated from the flange by a gasket and held in place with bolts. This apparatus was installed in the laboratory with connections arranged for the use of both low and high pressure steam. A view of the autoclave is given in the accompanying out.

The test pieces were placed on end in the autoclave so that as much of the surface of the piece as possible would be exposed to the steam and not be in contact with other specimens or the metal autoclave. After the test pieces were sealed in the autoclave, they were heated slowly for one hour under pressure ranging from zero to five pounds per square inch, gauge pressure. Then the pressure was rapidly increased to the prescribed point



and maintained for the desired length of time. After this time, the steam was shut off and the pieces allowed to cool to room temperature before removal from the autoclave.

Testing

All specimens were tested for modulus of rupture.

VARIATION IN THE LIME CONTENT

Mixes Investigated

Table XVI gives the composition of the mixes investigated.

Table XVI
Per Cent Lime in Mixes Investigated

Series	Composition
A-1	100 parts Blue Goose slime 5 parts lime 7 parts water
A-2	100 parts Blue Goose slime 7 parts lime 9 parts water
A-3	100 parts Blue Goose Slime 10 parts lime 13 parts water
A-4	100 parts Blue Goose slime 15 parts lime 17 parts water
A-5	100 parts Blue Goose slime 20 parts lime 17 parts water

Table XVI Continued

Per Cent Lime In Mixes Investigated

Series	Composition
A-6	100 parts Blue Goose slime 25 parts lime 17 parts water
B-1	100 parts Central Mill slime 5 parts lime 7 parts water
B-2	100 parts Central Mill slime 7 parts lime 9 parts water
B-3	100 parts Central Mill slime 10 parts lime 13 parts water
B-4	100 parts Central Mill slime 15 parts lime 17 parts water
B-5	100 parts Central Mill slime 20 parts lime 17 parts water
B-6	100 parts Central Mill slime 25 parts lime 17 parts water

The specimens from these mixes were formed at 10,000 pounds per square inch. The steam treatment under pressure was for four hours at 100 pounds per square inch. Two specimens were made from each mix.

Results

The results are summarized in the following table:

Table XVII

Effect of Variation of Per Cent Lime in Sand-Lime Brick Mixes

Series	Parts Lime per 100 Parts Slime	Hardness of Edges	Modulus of Rupture, lbs/in.
A-1	5	Fair	329
A-2	7	Good	511
A-3	10	Very Good	707
A-4	15	Excellent	683
A-5	20	Excellent	785
A-6	25	Excellent	790
B-1	5	Fair	278
B-2	7	Good	383
B-3	10	Very Good	515
B-4	15	Excellent	491
B-5	20	Excellent	648
B-6	25	Excellent	698

VARIATION OF MOLDING PRESSURE

Mixes Investigated

The composition of the mixes was the same for all the specimens in each series and was as follows:

Series AM-1 to AM-6

100 parts Blue Goose slime
10 parts lime
13 parts water

Series BM-1 to BM-6

100 parts Central Mill slime
10 parts lime
13 parts water

The molding pressure was varied for each series as follows:

AM-1	6,000	lbs/in. ²
AM-2	8,000	"
AM-3	10,000	"
AM-4	12,000	"
AM-5	14,000	"
AM-6	16,000	"
BM-1	6,000	"
BM-2	8,000	"
BM-3	10,000	"
BM-4	12,000	"
BM-5	14,000	"
BM-6	16,000	"

The steam treatment under pressure was for 4 hours at 100 pounds per square inch. Three specimens were formed from each mix.

Results

The results of testing the mixes formed under variable pressures are given in the following table:

Table XVII
Properties of the Variable Forming Pressure Mixes

Series	Forming Pressure	Hardness of Edges	Modulus of Rupture, lbs/in.
AM-1	6,000	Good	577
AM-2	8,000	Very Good	622
AM-3	10,000	Very Good	704
AM-4	12,000	Very Good	645
AM-5	14,000	Very Good	656
AM-6	16,000	Very Good	669
BM-1	6,000	Good	414
BM-2	8,000	Good	432
BM-3	10,000	Good	513

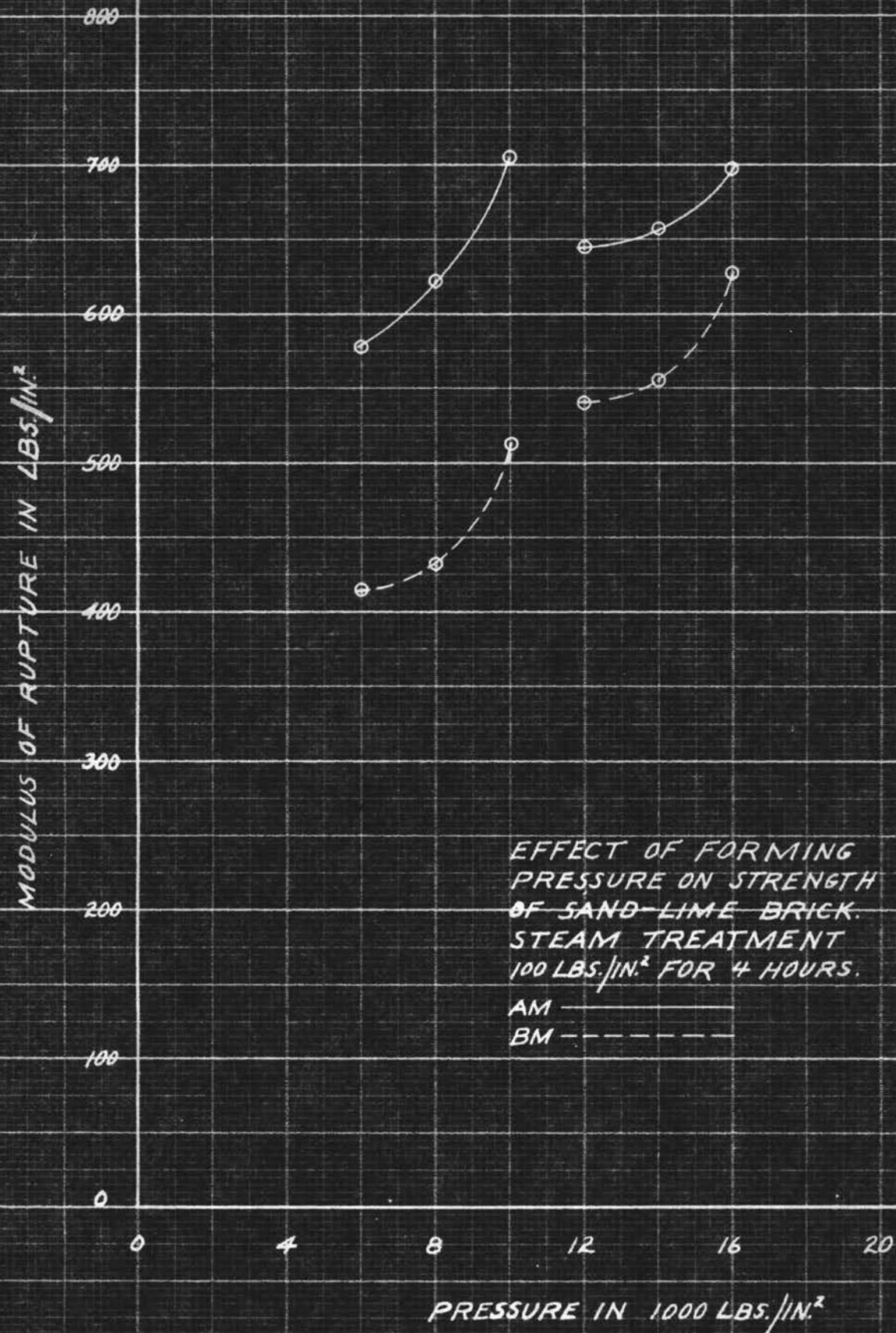


Table XVII Continued

Properties of the Variable Forming Pressure Mixes

Series	Forming Pressure	Hardness of Edges	Modulus of Rupture, lbs/in. ²
BM-4	12,000	Good	540
BM-5	14,000	Good	555
BM-6	16,000	Good	627

VARIATION IN THE PRESSURE AND TIME OF STEAM TREATMENT

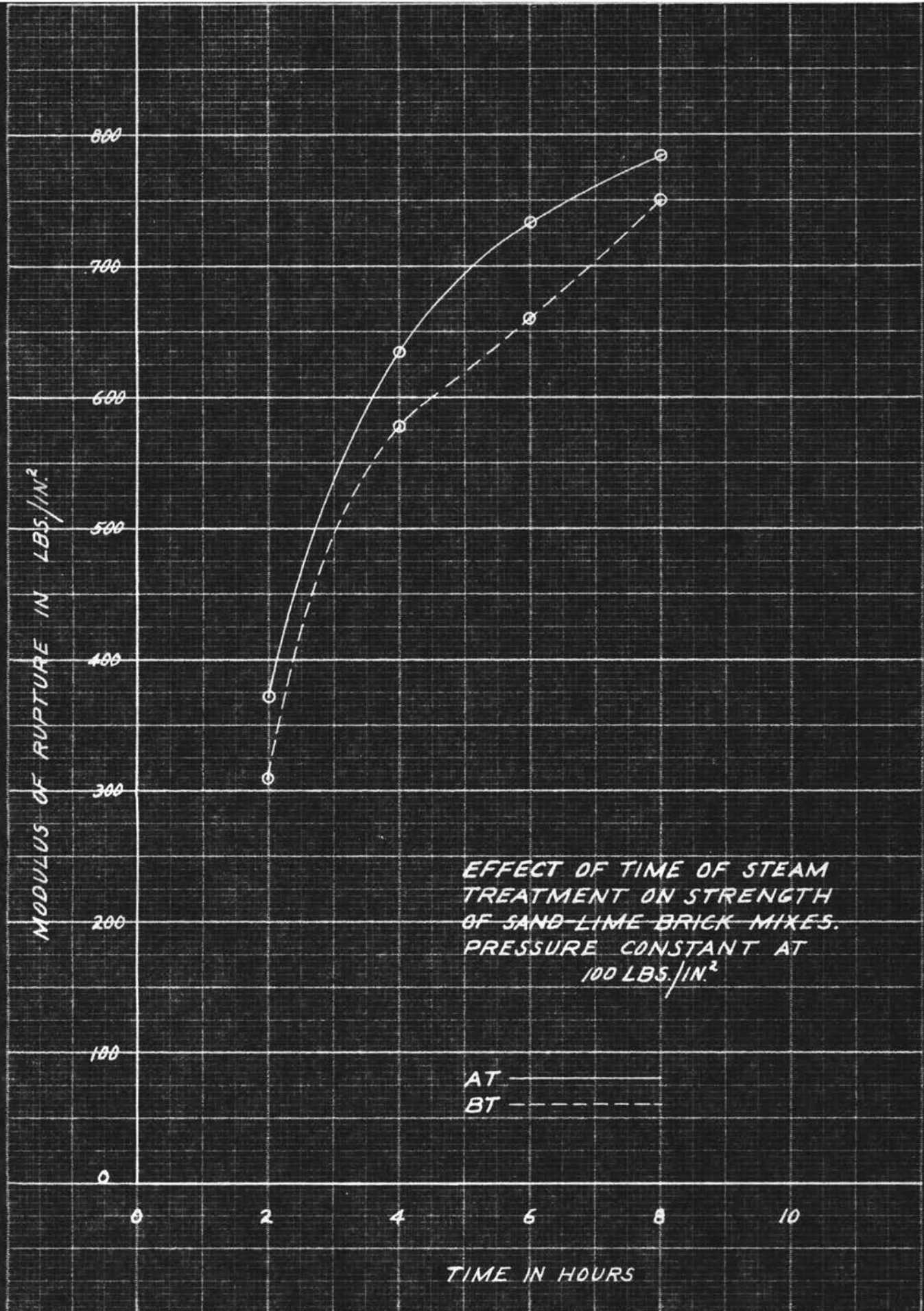
Mixes Investigated

The compositions of the mixes used for the specimens in each set were:

AT	100 parts Blue Goose slime 10 parts lime 11.5 parts water
BT	100 parts Central Mill slime 10 parts lime 11.5 parts water

All specimens were formed under a pressure of 10,000 pounds per square inch. The steam treatment was as follows:

<u>Series</u>	<u>Pressure and Time</u>
AT-1 and BT-1	2 hours at 100 lbs/in. ²
AT-2 and BT-2	4 hours at 100 lbs/in. ²
AT-3 and BT-3	6 hours at 100 lbs/in. ²
AT-4 and BT-4	8 hours at 100 lbs/in. ²
AT-5 and BT-5	4 hours at 50 lbs/in. ²
AT-6 and BT-6	4 hours at 75 lbs/in. ²
AT-7 and BT-7	4 hours at 125 lbs/in. ²

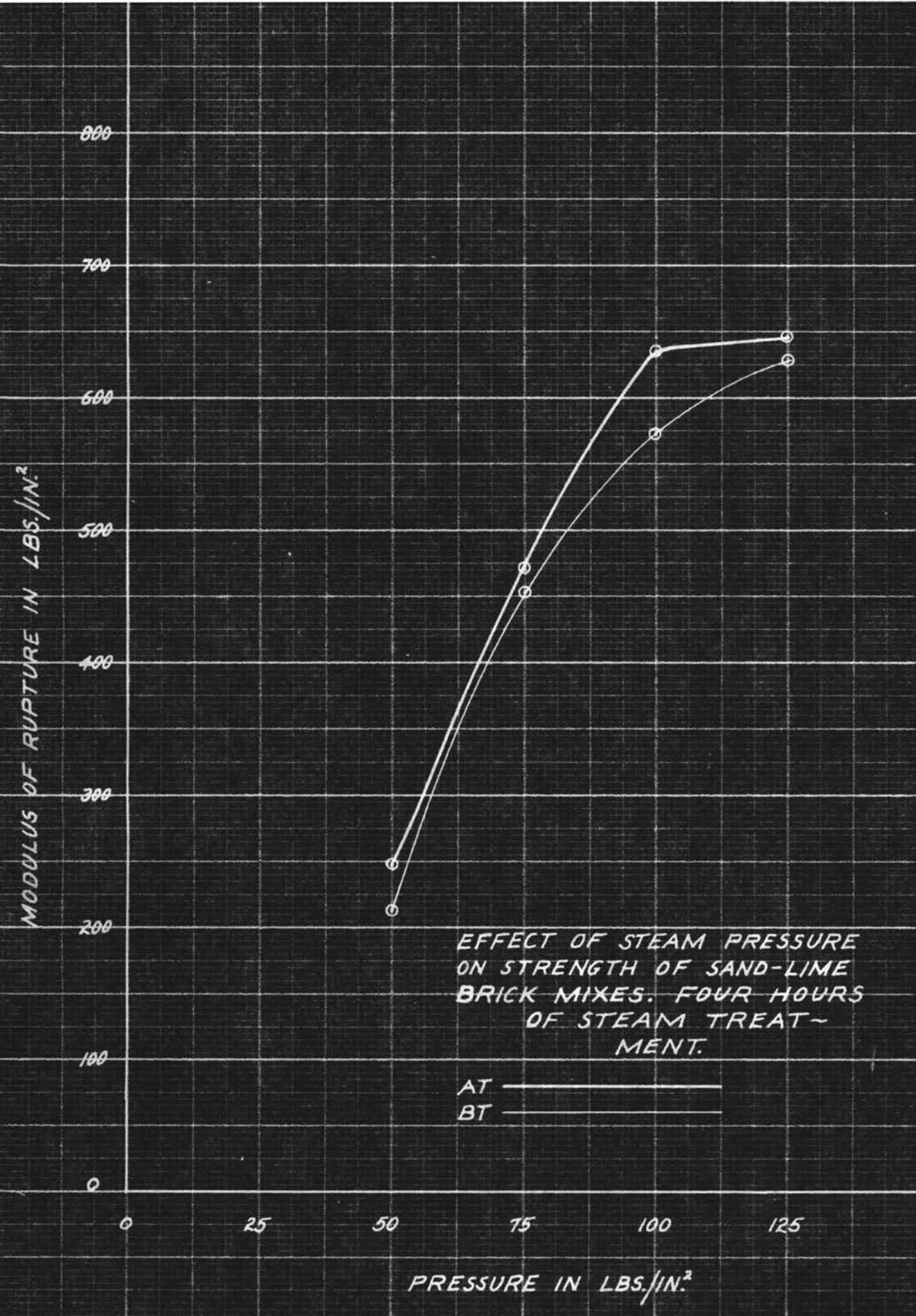


EFFECT OF TIME OF STEAM TREATMENT ON STRENGTH OF SAND-LIME BRICK MIXES. PRESSURE CONSTANT AT 100 LBS./IN.²

AT ———
BT - - - -

MODULUS OF RUPTURE IN LBS./IN.²

TIME IN HOURS



EFFECT OF STEAM PRESSURE ON STRENGTH OF SAND-LIME BRICK MIXES. FOUR HOURS OF STEAM TREATMENT.

AT —————
BT —————

PRESSURE IN LBS./IN.²

Results

A summary of the results of varying the pressure and time of steam treatment is given in table XIX.

Table XIX

Properties of the Steam Pressure--Time Mixes Investigated

Series	Pressure and Time	Hardness of Edges	Modulus of Rupture, lbs/in. ² *
AT-1	2 hrs. at 100 lbs/in. ²	Fair	172
AT-2	4 hrs. at 100 "	Good	635
AT-3	6 hrs. at 100 "	Good	744
AT-4	8 hrs. at 100 "	Very Good	783
AT-5	4 hrs. at 50 "	Fair	247
AT-6	4 hrs. at 75 "	Good	471
AT-7	4 hrs. at 125 "	Good	646
BT-1	2 hrs. at 100 "	Fair	310
BT-2	4 hrs. at 100 "	Good	577
BT-3	6 hrs. at 100 "	Good	660
BT-4	8 hrs. at 100 "	Very Good	750
BT-5	4 hrs. at 50 "	Fair	214
BT-6	4 hrs. at 75 "	Fair	454
BT-7	4 hrs. at 125 "	Good	638

*Average of three or four specimens

VARIATION OF GRAIN SIZE

Mixes Investigated

The grain sizes used in the mixes are given in the following table;

Table XX

Mixes Employed in Studying Effect of Various Grain Sizes

Series	Composition
AP-1	100 parts Blue Goose slime as-received 10 parts lime 11.5 parts water
AP-2	100 parts Blue Goose slime through 65 mesh* 10 parts lime 11.5 parts water
AP-3	100 parts Blue Goose slime through 28 on 65 mesh* 10 parts lime 11.5 parts water

*Tyler Standard Screen Scale.

The specimens were molded at 10,000 pounds per square inch and steam treated for 4 hours at 100 pounds per square inch pressure.

Results

The results of the testing of the specimens are summarized in table XXI.

Properties of the Variable Grain Size Sand-Lime Mixes

Series	Size of Grains	Hardness of Edges	Modulus of Rupture, lbs/in. ² *
AP-1	As-received	Good	649
AP-2	Through 65 mesh	Fair	614
AP-3	Through 28 on 65 mesh	Good	501

*Average of three specimens.

SUMMARY

The sand-lime specimens made from Blue Goose and Central Hill limes molded easily. Sixteen thousand pounds per square inch was the maximum pressure applied, but even at this pressure the wet pieces could not be handled without danger of breaking. However, the dry strength of the specimens was very satisfactory.

Difficulty was encountered in the steam treatment when specimens were stacked on top of other specimens. Those that had to bear the weight became deformed and showed marks where the test pieces had rested on them. Placing the test specimens one course high eliminated this difficulty and resulted in uniform treatment.

The effects of the variable factors applied during the investigation of the sand-lime specimens are given below and in the accompanying curves:

- (1) Increases in the per cent lime used increased the strength of the test pieces and made their color a lighter shade of gray. The small difference in modulus of rupture between mixes containing 20 and 25 parts lime per 100 parts lime made impractical the addition of more than 20 parts lime.
- (2) The strength of the specimens increased with an increase in the length of time of steam treatment and also with an increase of pressure and, consequently, temperature of the steam. The extension of the steam pressure beyond 100 pounds per square inch did not prove feasible.
- (3) The effect of variable molding pressures was irregular. The results are best shown by the curves for the AM and BM series.
- (4) Specimens made from as-received material gave better strength than those formed with 65 mesh material or the coarse 28 or 65 mesh grain size. Although water was not considered a variable, there was evidence that the amount of water used in forming the mix affected the strength.

CONCLUSIONS

Blue Goose slime mixtures with at least 10 per cent lime content and molded at pressures of 10,000 pounds per square inch, or greater, have a modulus of rupture equivalent to that of the highest grade sand-lime brick (Grade SW).* The Central Mill mixtures have a strength that is comparable to SW grade brick if 20 per cent lime is used with a molding pressure of 10,000 pounds per square inch. Reduced pressures for molding or reduced lime content cause the quality of the brick to fall to the lower quality grades (MW and NW). The results indicate, therefore, that with the proper amount of lime for bond and with suitable molding pressure both slimes may be used to produce high quality sand-lime brick.

*For explanation of the grades of sand-lime brick see page 44.

CHAPTER V

ARTIFICIAL STONE

Artificial stone or Rostone, as it is more commonly called, is a structural material possessing chemical and physical properties that make it adaptable for use in decorative and ornamental arts and also for structural purposes. The material results from the reaction between the proper proportions of aluminosilic acid, alkaline earth, and water. Heat must be supplied for the reaction, and so supplied as to prevent loss of the water added to the original mix until the reaction is complete. A coarse aggregate, such as rock, stone, gravel, or similar substances, can be mixed with the above raw materials and after the reaction it becomes an intimate part of the mass.

In the experimental work with artificial stone performed as a part of the chat investigation, Miami-district clays were used to furnish the aluminosilic acid; CaO and MgO, the alkaline earth; and Anna Beaver and Blue Goose chats, the aggregate. The heat treatment was carried out in an autoclave using low pressure steam. The clays were those designated by Mr. L. F. Sheerar in his report on Miami clays as Nos. 26, 27, and 28. The MgO was light MgO, U.S.P. grade, and the CaO was a commercial grade of quicklime. Steam pressure varied from atmospheric to thirty pounds per square inch. Test pieces were molded both by hand and under pressure with a Fred S. Carver laboratory hydraulic press, the pressures ranging from zero to sixteen thousand pounds per square inch gauge pressure.

EXPERIMENTAL DETAILS AND DATA

Preparation of the Test Pieces

The test pieces were molded in a cylindrical steel mold 1 1/8 inches inside diameter and 3 inches in height. In each series the dry materials were thoroughly mixed together and the mixture added to the water, gradually and with continued mixing. The clays were ball milled to pass a No. 200 sieve. Reprints of U. S. patents 1,852,672, and 1,877,959 were used as references.

(1) Clay-Lime-Water mixes to determine the effect of variable amounts of water.

<u>Series</u>	<u>Composition</u>
SW-1	11 parts lime 50 parts Miami No. 28 clay 13 parts water (20%)
SW-2	11 parts lime 50 parts Miami No. 28 clay 16.5 parts water (25%)
SW-3	11 parts lime 50 parts Miami No. 28 clay 19.8 parts water (30%)
SW-4	11 parts lime 50 parts Miami No. 28 clay 23 parts water (35%)

These test pieces were molded at a pressure between 400 and 500 lbs/in.². After they were molded, the pieces were placed, in the autoclave and a steam pressure of 5 lbs/in.² was applied for 1 1/2 hours.

(2) Clay-Lime-Water mixes to determine the effect of variable amounts of lime.

<u>Series</u>	<u>Composition</u>
SL-1	20 parts lime 50 parts Miami No. 28 clay 17.5 parts water (25%)
SL-2	15 parts lime 50 parts Miami No. 28 clay 16 parts water (25%)

<u>Series</u>	<u>Composition</u>
SL-3	10 parts lime 50 parts Miami No. 28 clay 15 parts water (25%)
SL-4	5 parts lime 50 parts Miami No. 28 clay 14 parts water (25%)
SL-5	Same composition as SL-1, except that Miami No. 27 clay was substituted for Miami No. 28 clay
SL-6	Same composition as SL-2, except that Miami No. 27 clay was substituted for Miami No. 28 clay
SL-7	Same composition as SL-3, except that Miami No. 27 clay was substituted for Miami No. 28 clay
SL-8	Same composition as SL-4, except that Miami No. 27 clay was substituted for Miami No. 28 clay

These test pieces were molded at a pressure of 400 to 500 lbs/in.². After they were molded, they were placed in the autoclave and steam treated at 25 lbs/in.² for two and one-half hours.

(3) Clay-Magnesia-Water mixes to determine the effect of variable amounts of water. A mixture of equal parts of Anna Beaver chat through No's. 16, 20, and 30 sieves was added as an aggregate.

<u>Series</u>	<u>Composition</u>
SM-1	11 parts magnesia 50 parts Miami No. 28 clay 75 parts chat 15 parts water (20%) of the weight of the magnesia and clay
SM-2	11 parts magnesia 50 parts Miami No. 28 clay 75 parts chat 16.5 parts water (25%)

<u>Series</u>	<u>Composition</u>
SM-3	11 parts magnesia 50 parts Miami No. 28 clay 75 parts chat 20 parts water (30%)
SM-4	11 parts magnesia 50 parts Miami No. 28 clay 75 parts chat 23 parts water (35%)

The pieces were molded at a pressure between 400 and 500 lbs/in.². Immediately after molding, the pieces were sealed in the autoclave and steam treated at 20 lbs/in.² gauge pressure for 4 hours.

(4) Clay-Alkaline Earth-Calcium Sulphate-Water mixes to determine the effect of variable amounts of calcium sulphate.

<u>Series</u>	<u>Composition</u>
SC-1	7.5 parts lime 25 parts Miami No. 27 clay 5 parts CaSO ₄ (Tech. grade) 9.4 parts water
SC-2	7.5 parts lime 25 parts Miami No. 27 clay 7.5 parts CaSO ₄ 10 parts water
SC-3	7.5 parts lime 25 parts Miami No. 27 clay 10 parts CaSO ₄ 10.6 parts water
SC-3	7.5 parts lime 25 parts Miami No. 27 clay 10 parts CaSO ₄ 10.6 parts water
SC-4	5.4 parts magnesia 25 parts Miami No. 27 clay 5 parts CaSO ₄ 9 parts water
SC-5	5.4 parts magnesia 25 parts Miami No. 27 clay 2.5 parts CaSO ₄ 8 parts water

<u>Series</u>	<u>Composition</u>
SC-6	5.4 parts magnesia 25 parts No. 27. clay 7.5 parts CaSO ₄ 9.5 parts water

The pieces were molded by hand pressure only. Immediately after being molded, they were placed in the autoclave and steam treated for 3 hours at 20 lbs/in.² pressure.

(5) Clay-Magnesia-Chat-Water mixes to determine the effect of magnesia when chat is used as an aggregate.

<u>Series</u>	<u>Composition</u>
SM-5	9 parts magnesia 25 parts Miami No. 28 clay 37.5 parts chat 8 parts water
SM-6	7 parts magnesia 25 parts Miami No. 28 clay 37.5 parts chat 8 parts water
SM-7	5 parts magnesia 25 parts Miami No. 28 clay 37.5 parts chat 7.5 parts water
SM-8	3 parts magnesia 25 parts Miami No. 28 clay 37.5 parts chat 7 parts water

The chat used was Anna Beaver chat screened through a No. 14 sieve and retained on an No. 100 sieve. The pieces were molded at about 500 lbs/in.² gauge pressure and placed in the autoclave. The steam was run into the autoclave for 1/2 hour at 3 lbs/in.² pressure and then for 4 hours at 20 lbs/in.².

(6) Clay-Magnesia-Chat-Water mixes to determine the effect of variable molding pressures.

The test pieces were steam treated at atmospheric pressure for 1/2 hour and at 20 lbs/in.² for 3 hours.

The ends of each test piece were imbedded in plaster of paris and an even layer allowed to adhere to these ends to insure a flat surface for the application of the compressive pressure. The pressure was obtained with a Fred S. Carver hydraulic laboratory press.

<u>Series</u>	<u>No. of Piece</u>	<u>Compression, lbs/in.²</u>
SP-1	1	---
	2	---
SP-2	1	800
	2	600
SP-3	1	1200
	2	1300
SP-4	1	1400
	2	1500
SP-5	1	1300
	2	1700

SUMMARY AND CONCLUSIONS

Since with the exception of the SP series, using magnesia as a bond and varying the molding pressure, there were no actual tests performed because of lack of sensitive compression testing equipment, the following results are based on observation and feel:

- (1) The SW and SM series in which the water content was the variable appear to be strongest when 25% of water by weight was used.
- (2) Within the range of precents used in the work to date, an increase in the alkaline earth content increased the strength of the test piece.
- (3) CaSO₄ of the technical grade when added to a mix using lime as the alkaline earth increased the strength.
- (4) Magnesia gave much stronger pieces than did equivalent amounts of lime.

(5) Substitution of Miami No. 27 clay for Miami No. 28 clay appeared to effect the color of the stone only.

(6) Increased molding pressure on mixes using magnesia increased the strength as shown by the following compression data:

SP-1	Not strong enough to give a recording
SP-2	About 700 lbs/in. ² gauge pressure
SP-3	1250 lbs/in. ² gauge pressure
SP-4	1450 lbs/in. ² gauge pressure
SP-5	1500 lbs/in. ² gauge pressure

The results indicate that with the proper ration of alkaline earth, clay, and water a bond for the chat aggregate may be obtained which will yield a favorable artificial stone. If the Miami clays do not give the desired results, other kinds of clay may be used to greater advantage.

CHAPTER VI

CONCLUSIONS

This investigation has considered three possible commercial uses of chats and slimes. Many others could have been selected.

If this work were to be continued, the following products could be suggested:

(1) Plastic refractories

A plastic mix would be made with chats or slimes as one of the refractory ingredients. The mix would be prepared with the proper water content, packed in air tight containers, and sent to the consumer to be employed as a ramming mixture to line furnaces.

(2) Fused refractory block

This would be an attempt to use silica in a new manner, following the principle of the Corhart process for electrocast corundum blocks. The chats would be melted completely and cast into the desired shape. Utilization of a cheap source of electrical power would be involved.

(3) Rock or mineral wool

This would be a chat-limestone mixture to form a fusible slag that could be blown into rock wool.

(4) Acid proof cement

Slimes would be used with sodium silicate to make a suitable cementing material.

(5) Foundry parting sand

(6) Corrosion resistant cement-chat mixes for the manufacture of conduits

(7) Roofing granules

The purity of the chats and slimes may be improved by a careful flotation process that would remove the impurities and increase the silica (SiO_2) to a higher percentage.

The results of the investigation have shown that silica brick can be formed using the slimes as the source of silica. However, before the quality of these brick as a refractory can be estimated, the following properties have to be considered: (1) ability of the refractory to withstand load at high temperatures, (2) resistance of the brick to abrasive action, (3) resistance to spalling and (4) pyrometric cone equivalent to softening temperature.

The slimes have proven to be a satisfactory substitute for sand in sand-lime brick. The manufacture of these brick commercially is dependent on a suitable local market.

Artificial stone should be experimented with further to make the Miami-Picher area clays more suitable for bonding the chat aggregate.

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