# THE USE OF FLY ASH TO MAKE A BUILDING MATERIAL

BY

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A Thesis Submitted to the Graduate Committee
For the Degree of

MASTER OF SCIENCE

in

Chemical Engineering

Approved:

Head of Department

Chairman, Graduate Committee

Virginia Polytechnic Institute
1936

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# I INTRODUCTION

In recent years there has been considerable development along the lines of using coal powdered to a fineness resembling talcum powder for the production of power in practically all the new and larger power plants. This pulverized coal burns similarly to gas when blown into the combustion chamber. Because of the fineness of the individual particles of coal, a very fine ash termed "fly ash" is formed when combustion occurs, a large portion of which is carried on up and out the smoke stack.

The "fly ash," if allowed to go up the stack and out over the surrounding territory, becomes a health hazard as well as a nuisance to the cleanliness of the community. The average fly ash is about fifty per cent silica ($SiO_2$) which should classify it as a silicosis hazard. To avoid this nuisance and hazard, the fly ash may be collected in one of several ways, the most important being by electrostatic precipitation, wet scrubbing, and with cyclone separators. It must then either be used or disposed of.

Several attempts have been made to use fly ash for some useful purpose, but it is not adaptable for filling in, for road construction, or for similar uses made of clinker ash.

In this investigation, an attempt has been made to determine the optimum conditions of both the process and composition which should be used in the manufacture of a building material from fly ash, and special reference will be made to the ash of the Virginia Polytechnic Institute Power Plant, Blacksburg, Virginia.

## ACKNOWLEDGMENT

I wish to thank the Virginia Polytechnic Institute for the opportunity afforded me to carry out this investigation, and the Departments of Chemistry, Applied Mechanics, Mechanical Engineering, Civil Engineering, and particularly Ceramic Engineering for the use of equipment and materials.

The New York Steam Company, New York City, was very kind in furnishing all the fly ash used.

Greatest appreciation is expressed to Dr. Frank C. Vilbrandt for his continued encouragement, suggestions, and helpful criticism throughout the entire investigation.

## II THE REVIEW OF LITERATURE

Health Hazard of Fly Ash  Harrington and Davenport (14) discuss the effects of breathing dusts containing silica. Breathing these substances containing silica results in a disease known as silicosis, for which there is no cure known at present. No treatment has been discovered that prevents the characteristic fibrosis after it has started in the lungs. Two facts stand out rather clearly in the discussions regarding the comparative harmfulness of silica and other dusts. One is that silica is more dangerous because it apparently predisposes to tuberculosis more than other dusts. The second fact is that the mortality rate is highest in the later years. Heavy inhalations of any dust are also liable to cause pulmonary damage. The most deadly of all dusts examined was precipitated silica.

Collection of Fly Ash  Cottrell precipitators (1) are one of the most common methods of arresting fly ash. In June 1934 there were seventeen commercial installations handling 8,000,000 cubic feet of gas per minute at 350° F from sixty-one boilers removing well over ninety per cent of the suspended ash.

General Information  Very little information has been published on the use of fly ash or of any alumino-silicate in the manufacture of a synthetic building material. Most of the work along this line has been done at Purdue University by Peffer, Harrison, and

Shreve (21), and Harrison, Jones, and Shreve (15). They state that because of the relative ease with which silicates may be made to react with lime to form sand-lime brick, it might be thought that aluminosilicates would react similarly to form a calcium aluminosilicate in the place of a calcium silicate. Work done along this line for the past few years has shown that this reaction does take place under appropriate conditions. Three patents (19, 20, 25) have been taken out at Purdue, the fundamental idea of which is that an artificial building material may be made by intimately mixing an alkaline earth base with a material containing finely ground hydrated aluminosilicic acid; heating the mixture in a closed chamber retaining the essential water, forming a material of high compressive and tensile strength. The chief basis of their patents is upon the statement that, although aluminosilicic acids have been in the past considered as having little reactivity, they have found that under the proper conditions of moisture and temperature, they react readily.

Raw Materials The materials used by the Purdue experimenters consisted of shales from the Mississippi, Illinois, and Vermont, and residual fly ash from the combustion of powdered coal (21). Hydrated lime of the usual commercial type was the other chief raw material, with wood rosin added in small amounts.

Process The technical operation used by these investigators consisted essentially of thoroughly incorporating shale of which 90 per cent would pass a 325-mesh sieve with high calcium hydrated lime.

This was done in a revolving wet pan with scraper blades. Sufficient water was added to form a damp mass, which usually amounted to from 18-22 per cent. This moist mix was then compressed in polished steel moulds under a pressure of 2500 pounds per square inch. It was then removed from the mould and allowed to stand in the atmosphere for one or two hours. It was then placed in an autoclave under 75 pounds gauge steam pressure for two hours, the purpose of this operation being to complete the reaction between the aluminosilicate and the alkaline earth base (lime). In this indurating reaction it was necessary to keep the necessary reaction water in the mass and hold it within narrow limits, characteristic for each material. Upon removal from the autoclave the samples had full strength.

Practical Applications Examples of the uses to which "Rostone" may be put were described. Among these were exterior and interior wall medium, flooring slabs, decorative intaglio tile, hollow wall sections, vases and ornaments. Tests were made to show the resistance to exposure to weather which proved successful. Because of the fact that the material is adaptable to the incorporation of inorganic coloring materials, a desirable variety of flooring slabs may be made. Large wall blocks up to 17 x 48 x 2 inches and weighing 130 pounds have been made recently. According to Harrison (15) and his associates, there is the possibility that some day power plants will have the facilities for making such a material directly under the electrostatic separators which collect the fly ash. In a bulletin (2) published by Rostone Inc., a detailed description of the house at A Century of Progress is given including numerous pictures. Other applications are also mentioned.

Physical Characteristics As described by Harrison (15), this building material has several characteristics which are not common for most building materials. The surfaces are smooth and true with no warpage or shrinkage occurring after shaping in the moulds. According to his experiments, bricks made from fly ash by his process had the following properties:

| | |
|---|---|
| Compressive strength (2-inch cubes) | 6000-8000 lb./sq. in. |
| Modulus of rupture (standard brick test) | 600-1000 lb./sq. in. |
| Absorption (5-hr. immersion) | 3.5-6.0% |

Special reference is made to the hollow load bearing wall block 8 x 8 x 16 inch with 55 per cent core space and weighing 23 pounds per block. Although building codes call for from 700 to 1000 pounds per square inch over gross area, the compressive strength of this hollow brick will run as high as 2226 pounds per square inch. Another very desirable characteristic of brick made from fly ash is the ease with which the absorption value may be varied. In an investigation of the construction of water-tight masonry (15), it was found that the best results occur when there is an optimum relation between the character of the mortar and the porosity of the building material. This porosity, which is measured by the absorption value, may be varied and controlled by varying the amount of resinous material used. Fly ash bricks also gave favorable results to fire tests in which a wall of the blocks was used in the experiment. The hot side obtained a temperature of 1900° F, the air space 825° F, and the cool side reached a temperature of 300° F. A more detailed description of the properties of this type of building material made from shale may be found in the article by Peffer (21) which includes a detailed description of hardness tests, absorption rates, resistance to weathering, abrasion, and toughness.

Application to Fly Ash Fly ash may be obtained in quantities ranging from a few hundred tons a week up to as high as 500 tons a day (15). In order to prevent the scattering of the ash and creation of a nuisance, they are usually collected by the use of electric precipitation, wet scrubbing methods, or cyclone separators. The ash is usually light grey or black in color. The particle size of samples taken from two different localities (in per cent) was as follows (15):

| | Sample A | Sample B |
|---|---|---|
| Retained on 100 mesh | 6.25 | 6.5 |
| Retained on 200 mesh | 9.40 | 0.7 |
| Retained on 325 mesh | 7.75 | 4.6 |
| Through 325 mesh | 76.60 | 88.2 |

When the fly ash is inspected under a microscope, the individual particles have a glass-like appearance and are sometimes in the form of small hollow spheres or bubbles. The chemical analysis will vary with the locality, but the carbon content will depend mostly upon the efficiency of the particular power plant and the character of the original coal used.

TABLE I

A TYPICAL FLY ASH ANALYSIS

| | Sample A | Sample B |
|---|---|---|
| $SiO_2$ | 46.9 | 44.1 |
| $Al_2O_3$ | 29.9 | 20.44 |
| $Fe_2O_3$ | 11.9 | 18.72 |
| CaO | 2.6 | 6.85 |
| MgO | 0.9 | 0.92 |
| TiO | 1.3 | 0.71 |
| C | 6.5 | 8.26 |

Black globules of magnetite and some hematite were shown by petrographic examination.

Control of Efflorescence Bricks made from fly ash have the tendency to exhibit a very bad white efflorescence upon exposure to weather unless they are specially treated. The main constituents of this white efflorescence are sulfates of calcium and sodium. Attempts were made (15) to wash these soluble salts out of the ash before using it. This reduced the efflorescence, but did not eliminate it. It was found that more sodium salts were set free in the steaming process, but that the addition of some organic substance would solve this problem. Pulverized wood rosin was found to be best. The rosin reacts with the soluble salts to form resinates, which results in the elimination of efflorescence. The portion of rosin required varies with the ash used, but it is seldom found necessary to use more than one per cent of the final dry weight.

Coarse Grains Not Practical The coarse ground shale (22) such as that retained on a 48 mesh sieve, undergoes very little reaction with the result that an unsound product is formed.

Other Desirable Properties of the Brick Although normal compressive strengths run from 6,000 to 8,000 lbs. per sq. in. (22), compressive strengths of 22,000 lbs. per sq. in. have been attained with transverse strength of 1500 to 2500 lbs. per sq. in. The typical physical test given here (22) is:

| | |
|---|---|
| Compressive Strength | 10,000 lbs. per sq. in. |
| Flexure | 1,600 lbs. per sq. in. |
| Absorption | 12% |

Results of X-Ray Analysis X-Ray work (22) shows that none of the original alumino-silicic acid and lime is present in the final product, indicating that these two substances have reacted to form a new compound. The X-Ray analysis does show that silica, dolomite and other common ingredients of the original shale remain inert.

Effect of Gypsum on Natural Cements Eckel (9) states that the effect of gypsum in regard to setting time and strength is about the same for natural cements as for Portland Cement. It is pointed out, however, that the degree to which the gypsum affects the product depends entirely upon the chemical composition of the particular cement. Former experimenters seem to have entirely overlooked this fact. The analysis of the particular cement used should be given. In the table (9) showing the effect of plaster on setting time, 2% gave the maximum initial setting time while 3% gave the maximum final setting time. This was true for both brands of natural cement used in the experiments. In experiments run to determine the effect of gypsum on tensile strength it was found that the addition of just 1% had injurious effects on the soundness of the cement, but larger percentages were necessary before the tensile strength was appreciably decreased. The high percentages, 3 to 6%, caused bad cracks.

Soundness of Lime for Structural Use Bessey and Eldridge (6) did some work on the soundness of lime, with special reference to its use in the manufacture of sand-lime brick. Although it is desired that sand-lime brick as well as other building materials have good properties as regards strength, hardness, resistance to weather, color and surface texture, the only one very appreciably affected by the quality of lime used is the strength. Consequently, tests as to the adaptability of lime for sand-lime brick manufacture are based on the strength of the resulting bricks which are manufactured. Thorough and extensive tests were made using specimens 1.5 inches in diameter and 2.1 inches high. These data proved conclusively that soundness was the paramount property and the chemical analysis of the lime of secondary importance. Therefore, most any lime may be used if it is sound, regardless of the chemical composition. As regards chemical composition, however, it may be generally stated that magnesium limes should be avoided due to the fact that they require very careful treatment because of the ease with which magnesia is overburned. High calcium and moderately hydraulic limes are normally quite satisfactory. Incorrect hydrating or overburning is the usual cause of unsuitable limes.

Soundness Test In the soundness test conducted here (16), the expansion of specimens was used as a basis for the determination. All specimens with low expansion gave a brick with high compressive strength, and all those with high expansion gave a weak brick with very low compressive strength. The actual test was carried out by placing a split brass cylinder mould on a piece of glass and filling with a mixture of hydrated lime and silica sand. The distance between the indicator arms on either side of the split was measured. The mould was then placed in an autoclave for at least 4 hours with the temperature not less than 170° C. The distance between the indicator arms was again measured and the increase was a record of the expansion of the mixture. 'Soundness' was found to be the essential point.

"Expansions ranging from zero up to 68.5 mm. were obtained, and all those limes giving strengths of less than 3,000 lbs./sq. in. gave expansions of over 5 mm. while those with higher strengths gave expansions below that figure.

"This relation may be considered remarkably consistent when it is remembered that the limes tested varied so widely in chemical composition and also in certain of their physical properties, such as fineness and workability.

"The mould, placed upon a small piece of glass, is filled with the mix prepared as described below, and the material tamped in by hand, care being taken to keep the jaws closed. The mix is tamped level with the top of the mould and covered with another glass plate upon which a small weight is placed.

"The mix used consists of 30 gr. of the hydrated lime, 50 gr. of fine silica sand passing a B. S. sieve No. 150, and 120 gr. of silica sand passing a B. S. sieve No. 18 and retained on a B. S. sieve No. 25, with 13.0 cc. of water. The whole is thoroughly mixed before filling the mould.

"The distance separating the indicator points is measured, and the filled mould transferred immediately to an autoclave and exposed for at least 4 hours to a temperature not less than 170° C, corresponding to 115 lbs. per sq. in. steam pressure. One hour should be allowed for the autoclave to attain its pressure and one hour for cooling or blowing off steam, the total time in the autoclave being therefore not less than 6 hours. The distance separating the indicator points is then measured; the difference between the two measurements represents the expansion of the sample."

The A. S. T. M. (3) in its standard specifications for hydrated lime gives a soundness test. The test is carried out by mixing 20 gr. of the lime sample with 100 gr. of standard Ottawa sand to which enough water is added to give the mixture the consistency of a rather dry and fairly plastic mortar. A pat of definite thickness is spread on a glass plate and allowed to stand under definite conditions for 24 hours. If any cracks appear at this time the sample is discarded because of improper consistency. The good pat is then suspended in a vessel partially filled with cold water, so the water can boil without touching the pat. The water is gradually brought to boiling and kept boiling gently for 5 hours, the pat being surrounded by steam during the time. The pat is removed after the setup has cooled for at least 12 hours. The sample is considered as being 'sound' if the steam has no visible effect on the pat. The sample is considered as being 'unsound' if the pat disintegrates. If the pat cracks, pops, or shows other minor defects it is considered as being either sound or unsound, but its behavior should be noted.

In the A. S. T. M. (4) tentative methods, a slightly different method of determining 'Soundness' is described. A pat is used here also, but an autoclave is used for the steaming operation which takes place for 2 hours at a pressure of 120 lbs. per sq. in. Unsoundness is manifested by popping of the specimen.

Emley (11) states that lime when used in sand-lime brick must be completely hydrated before the bricks are pressed if you wish to avoid expansion during the steam treatment which would cause internal strains and disruption of the brick.

Sand-Lime Brick Manufacture In a Technologic Paper of the Bureau of Standards (11), a detailed description of the steps in the process of making sand-lime brick is given. The essential points in the process are:

(a) add the required amount of thoroughly hydrated lime to the sand and mix the two intimately,

(b) add enough water to give the material such a consistency that it will hold together when pressed in a mould;

(c) press the damp mixture to the desired shape,

(d) cure the brick by means of steam under pressure.

It was believed at one time that monocalcium silicate (18) composed most of the bonding material, but recent researches by the Bureau of Standards (10) have failed to disclose satisfactory evidence of the presence of any silicate other than the mono calcium silicate. Monocalcium silicate (11) has never been obtained in the crystalline form. It has the formula $CaO.SiO_2$ so that it contains 48.25 per cent lime and 51.75 per cent sand.

Effect of Particle Size  Emley (11) points out the fact that under ordinary conditions silica sand is very inactive and even under the autoclave conditions it is only the surface of the grains which is attacked by the lime. Since the area of a sphere varies as the square of the diameter and the volume as the cube, the smaller the particles, the greater the surface area for the same weight of sand. If the sand is so reduced in size as to obtain the amorphous condition (16) it can take up water and will become quite chemically active (8) Peppel has shown that larger grains tend to produce a brick with high crushing strength and low transverse strength, while the opposite holds if the majority of the grains are small.

Mixing of Sand-Lime Brick  The mixture (11) consists of about 10% lime, and 90% sand. In order for chemical reaction to take place between the calcium oxide and silica oxide, this mixing must be thorough and complete. Fine grinding should not result from the mixing operation; about 15% should pass a 100 mesh screen. Sufficient water is added so that the material will just stick together when pressed into a ball with the hand.

Pressing and Hardening of Sand-Lime Brick   The most important function of pressing (11) is to bring the sand and lime into very intimate contact and not just to give the brick its desired shape. The proportion of voids is also decreased by increase in pressure. Peppel (24) found that the maximum strength resulted from a pressure of 15,000 lbs. per sq. in. The rotary type press (27) has been found most suitable for the manufacture of sand-lime brick though the vertical may be used.

Before the bricks are hardened (11) they must be handled with a great deal of care as they are quite fragile. The curing time varies from 4 to 11 hours and the steam pressure from 100 to 150 lbs. per sq. in.

Tests on Brick   Directions for making tests may be found described in detail in the report of Committee C-3 of the American Society for Testing Materials (5). Emley (11) discusses tests of sand-lime brick for crushing strength, transverse strength, absorption, freezing, and fire resistance.

The Three Component System   The Ternary System of $CaO-Al_2O_3-SiO_2$ (29) is shown graphically indicating the various binary compounds and the two ternary compounds, Anorthite $CaO-Al_2O_3-2SiO_2$, and Gehlenite $2CaO-Al_2O_3-SiO_2$. The percentage compositions of the various compounds are given with the formation temperatures.

<u>Analysis of Fly Ash</u> Fish (12) gives a very good detailed analysis for coal ash. Other similar methods of analysis are given by Griffin (13) and Scott (26). Boroditzkaya (7) gives a rapid method for the analysis of aluminosilicates including the determination of $SiO_2$, $TiO_2$, $Al_2O_3$, $Fe_2O_3$, and CaO in clays, kaolins, feldspars and other aluminosilicates. Less complete analyses for ash are given by White (28) and Parr (17).

## III THE INVESTIGATION

Object The object of this investigation was to determine the most desirable ratio of components, of which fly ash was chief, and the best method of procedure by which a material, suitable for structural purposes, could be made. The possibility of using ash from the power plant of the Virginia Polytechnic Institute, Blacksburg, Virginia, was also considered.

Plan of Attack The method used for arriving at these optimum conditions was to make a brick by a standard run and then vary, one at a time, each of the factors involved in the process and determine what value of each factor varied would indicate the best brick. The yield point under compression was the main criterion for determining the best brick.

As the Virginia Polytechnic Institute Power Plant ash was entirely unsuitable for such a use as it now exists, the fly ash from another plant (The New York Steam Company) was used in the investigation of optimum conditions. Analyses, both chemical and physical, were made of the two ashes so they could be compared.

Special consideration was given to a determination of the "soundness" of the lime used.

## APPARATUS

1-Autoclave The autoclave used in this investigation is shown in Plate I. It is a one gallon autoclave made by the Alberger Chemical Machinery Company, New York. Although it was designed for work with organic liquids, it was adapted to this procedure by inserting the pipe connections as shown in the picture. The valve in the fore part of the picture was used to regulate the condensed water outlet as the pipe to which this valve was attached ran to within an inch of the bottom of the autoclave. The other valve was used to let off steam from the top of the autoclave. A needle valve was employed to control the inlet steam coming in through the pipe running up highest of the three. A 300 pound gage indicated the steam pressure within the apparatus, and eight one-inch bolts were placed in the slots between the flanges to hold on the top. This autoclave was located in the basement of the Mechanical Engineering Laboratory.

2-Small Wet Pan Plate II gives a view of the interior of the Clearfield Laboratory Wet and Dry Pan made by Clearfield Machine Company, Clearfield, Pennsylvania.

3-Large Wet Pan A picture of the large three-foot Grinding Pan made by The Stevenson Company, Wellsville, Ohio, is shown on Plate III.

4-Compression Machine Plate IV shows the Tinius Olsen Universal Testing Machine made by the Tinius Olsen Testing Machine Company, Philadelphia, Pennsylvania. It may be used for tension, compression,

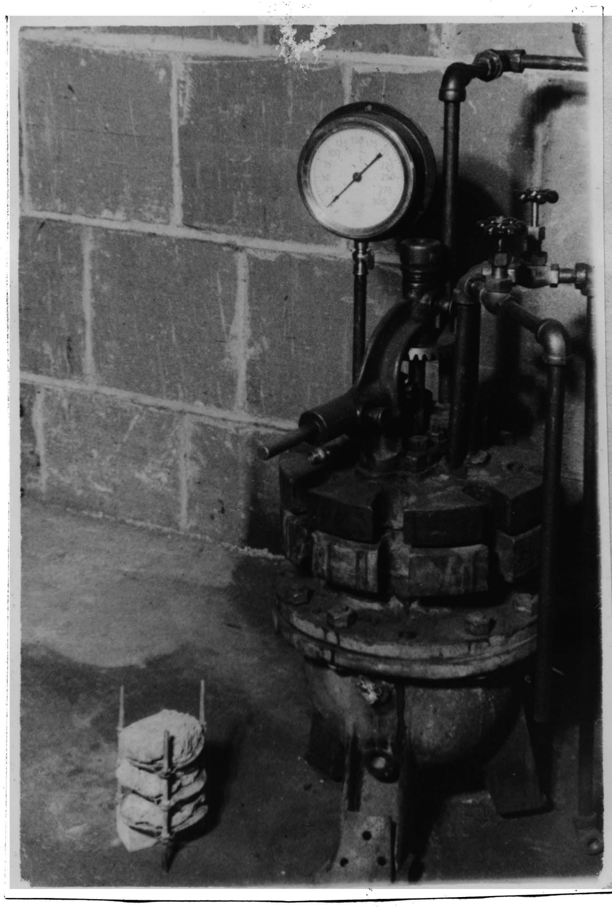


Plate I

AUTOCLAVE

Plate II

SMALL WET PAN

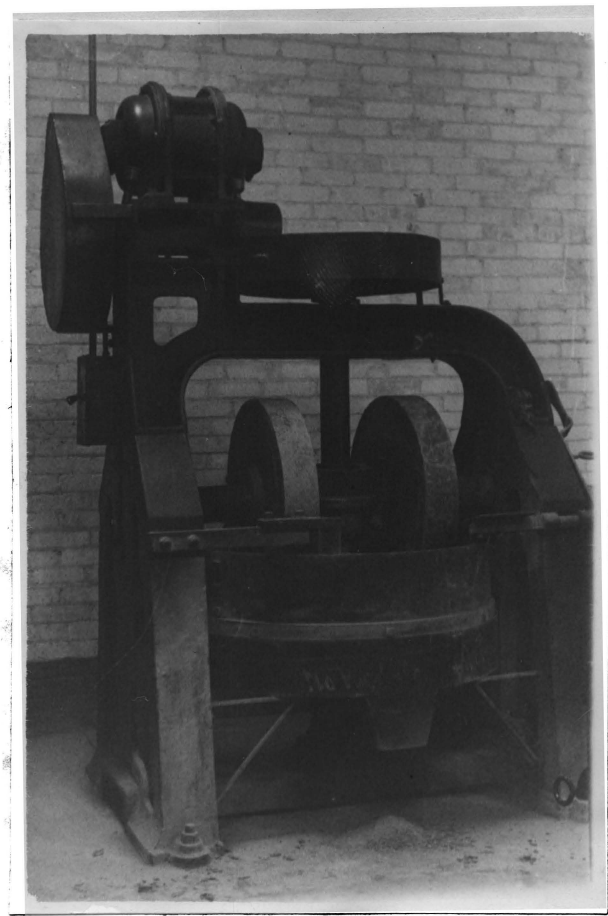


Plate III

LARGE WET PAN

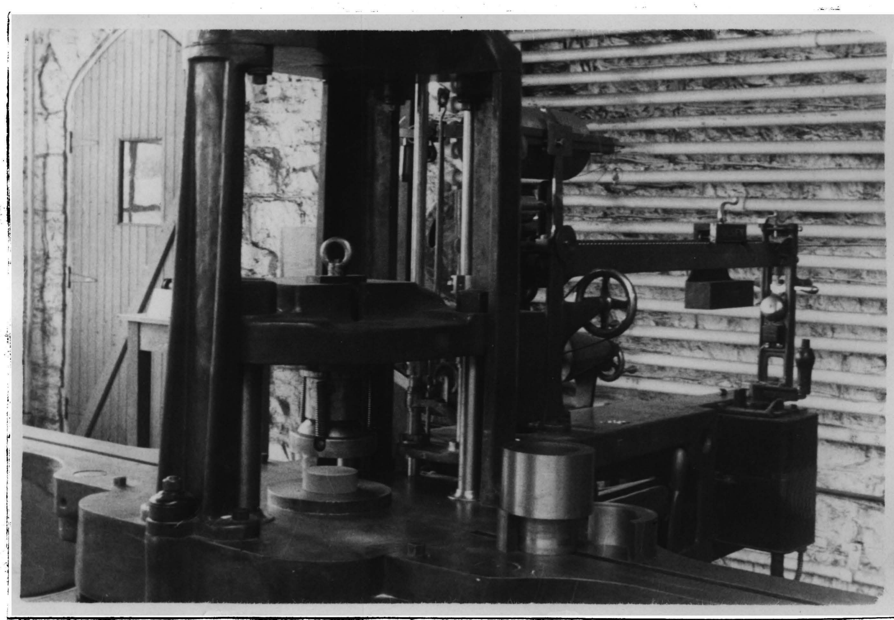


Plate IV

TINIUS OLSEN UNIVERSAL TESTING MACHINE

AND MOULD

and bending tests, and has a capacity of 100,000 pounds total force.

5-Tensile Testing Machine   A Riehle Testing Machine made by Riehle Bros. Testing Machine Company, Philadelphia, Pennsylvania, is shown on Plate V.

6-LeChatelier Soundness Tester   Plate VI shows the soundness tester assembled as used. The essential part of the outfit is the split cylinder made of brass with indicator arms on either side of the split. The cylinder itself is made of 0.5 m.m. thick brass. It is 30 m.m. high with an internal diameter of 30 m. m. The distance from the center of the cylinder to the ends of the pointed indicator arms is 165 m.m. A millimeter ruler is shown perpendicular to and at the end of the indicator arms which rest on a copper support. Two pieces of glass are held over the ends of the cylinder by a clamp and small spring. The board on which the instrument rests acts as the top for the box which is arranged to hold the various parts.

7-Mould   The mould used in this investigation was made by the Machine Shops of the Virginia Polytechnic Institute. It is shown on Plate IV resting on the beam support of the testing machine. It consists of:

a. A top nine inches in diameter and fifteen-sixteenths inch thick. This is shown with a brick resting on it.

b. A hollow cylinder with a four-inch inside diameter and five and fifteen-sixteenths inch outside diameter. It stands four inches in height.


Plate V

RIEHLE TESTING MACHINE

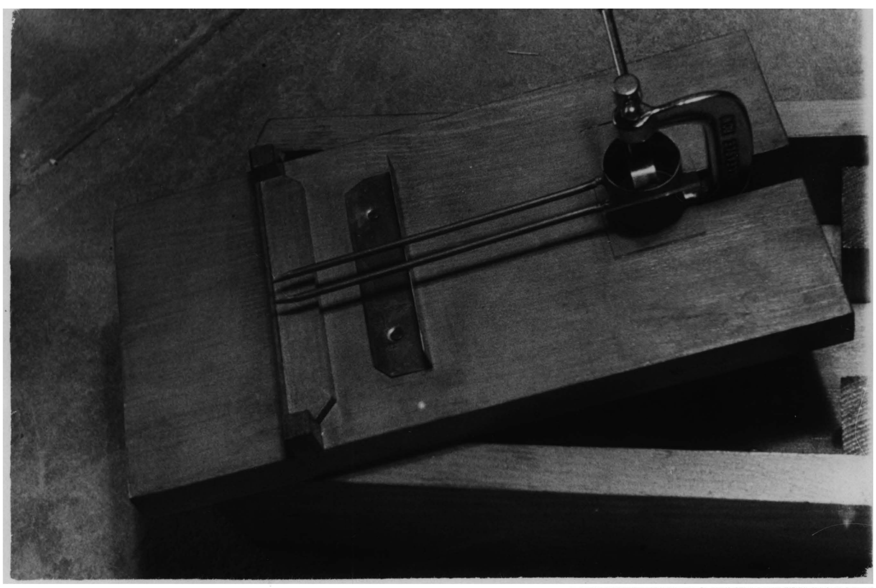


Plate VI

LECHATELIER SOUNDNESS TESTER

c. A solid plug five inches high and four inches in diameter.

d. Two supports two-and-one-half inches high with a one-eighth inch rim around the outer edge of the top.

This makes the mould one and one-half inches deep.

8-Rack for Autoclaving Bricks Plate I shows the rack used to hold the bricks while in the autoclave. It was made from three brass rods threaded the entire length and copper screen wire as the supports for the bricks. This screen wire was wrapped around a heavy piece of copper wire which was held in place by six brass nuts, three above and three below each support.

9-Sieves A set of three U. S. Standard Sieves (100 mesh, 200 mesh, and 325 mesh) including a top and bottom was used. These were made by the Precision Scientific Company, Chicago, Illinois.

10-Sieve Shaker A Ro-Tap Testing Sieve Shaker No. S. B. 603 made by The W. S. Tyler Company, Cleveland, Ohio.

11-Analytical Laboratory A complete analytical laboratory including reagents.

12-Ball Mill The mill and pebbles are sold by Paul O. Abbe Inc., Little Falls, New Jersey.

13-Hydraulic Piston Extrusion Machine This machine was located in the front laboratory of the Ceramics Department.

14-Mortar and Pestle A large size mortar and pestle was used to pulverize the rosin.

15-Wet Box A large close fitting box in which there was always about four inches of water in the bottom. The humidity was always nearly a hundred per cent.

## MATERIALS

1-**Fly Ash** Fly ash obtained from the New York Steam Company was employed in making all the bricks in this investigation. Tables IX and X give the chemical and sieve analysis of this ash and also that from the Virginia Polytechnic Institute Power Plant.

2-**Lime** Mason's Hydrate Lime, made by the Ripplemead Lime Company, Inc., Ripplemead, Virginia, was used exclusively. The table showed the per cent calcium oxide to be eighty, and that of magnesium oxide three.

3-**Rosin** Lump rosin (CCC) distributed by Phipps and Bird Inc., Richmond, Virginia.

4-**Calcium Sulfate** $CaSO_4:2H_2O$, reagent, Code 1537, manufactured by the General Chemical Company, New York.

5-**Lycopodium Powder** A yellow pollen powder which may be purchased from a pharmacist. It is not wetted by water.

6-**Water** Tap and distilled.

7-**Steam** From 175 pound line of the Virginia Polytechnic Institute Power Plant.

8-**Sand** Pure Berkshire and Standard Ottawa.

## METHOD OF PROCEDURE

### MIXING

Use of Ball Mill  A mixture of 1,000 grams of fly ash from the New York Steam company and 100 grams of mason's hydrated lime was prepared to which was added 5 grams of rosin. The three constituents were partially mixed with a spatula and 400 grams of water added. This was again mixed byhand and spatula, resulting in a moist mass. This mixture and 50 one-inch flint pebbles were placed in the porcelain jar of the ball mill and ground for fifteen minutes at forty R.P.M. The ball mill was stopped in the middle of the run to clean out the corners. The mixture packed in the corners so badly that practically no mixing was accomplished, with the result that specks of lime could be seen all through the mass.

Use of Wet Pan  The second mixture was made of 1,000 grams of ash, 100 grams of lime, 5 grams of rosin, and 200 grams of water. This was partially mixed with a spatula and then in the small wet pan described under "Apparatus." The wet pan operation which lasted fifteen minutes, gave much better mixing than was accomplished above. A trowel was used to direct the material under the mullers and to break up the cake forming under them. The completeness of the mixing was indicated by the absence of lime particles large enough to be detected by eye when a handful of the mix was pressed between the fingers.

## MOULDING

First Attempt to Make Bricks in Mould   A portion of the material taken from the ball mill was placed in the mould and compressed at 2500 pounds per square inch. The material placed in the mould was packed with moderate pressure of the hands before putting on the top of the mould and using the Tinius Olsen Machine. In the attempt to remove this brick from the mould it broke, due mostly to the sticking of the brick to the plug of the mould.

A piece of bond paper was then placed in the mould between the plug and the brick and a pressure of 1500 pounds per square inch applied. This produced a satisfactory brick, but due to the fact that the constituents were not properly mixed, as evidenced by specks of lime, and due to lack of time on that day, the brick was not autoclaved. In the pressing of both bricks, excess water ran out of the mould, indicating that too much water had been put in the mix.

Second Attempt to Form Bricks in Mould   For the first brick this time the mould was made an extra inch and a half deep by placing the supports for the cylinder on a piece of wood one and a half inches thick, making the total depth of the mould three inches. To prevent sticking, the top of the mould was oiled with lubricating oil, and a sheet of oiled wax paper placed on the plug. No packing with the hands was used this time, and a pressure of 2500 pounds per square inch was employed. The material used was that taken from the wet pan mixing. When the brick was pushed out of the mould, it cracked badly across the top. This appeared to be the result of the expansion of air compressed within the brick during formation. A large reduction of volume within the mould took place as the material was so loosely arranged at the start. The brick stuck to the plug, but not to the top.

A second brick was pressed using 2500 pounds per square inch with the mould at its regular depth, one and a half inches. Oil and oil paper were used as before. Results were bad this time as both top and bottom of the brick broke, due to sticking to the mould.

Third Attempt to Form Bricks in Mould This time the mix, consisting of 1,000 grams of ash, 200 grams of lime, 5 grams of rosin, and 250 grams of water, was placed in the small wet pan for fifteen minutes and pressed at 2500 pounds per square inch. Lycopodium powder was dusted on the plug of the mould this time with the result that the bricks did not stick and were easily removed. Three good bricks were made and a fourth only slightly injured where it stuck to a place on the plug which was not thoroughly covered by lycopodium powder. To prevent the top of the mould from sticking to the brick, it was always given a twisting motion before removing, with the result that the interface between the mould and the top of the brick was broken. It was discovered in the next batch moulded that the bottom also would not stick if the interface was broken. This was accomplished by holding the plug stationary and twisting the cylinder of the mould. The cohesion between the brick and the side of the mould was greater than that between the brick and the plug with the result that the interface between the latter two was broken, and the brick could be pushed out in good condition. Only a slight film of oil and no Lycopodiun was required using this procedure.

## AUTOCLAVING

First Attempt to Autoclave Bricks   The three good bricks mentioned above were placed in the brass rack and put into the autoclave. The top of the autoclave was fastened tightly and the steam allowed to enter. At the end of five minutes the pressure had reached 70 pounds per square inch which pressure was maintained for twenty-five minutes. At the end of this half hour, a quantity of material looking like fly ash was seen coming out of the autoclave along with the condensed water. The steam pressure was released by cutting off the inlet steam valve and opening the exhaust valve. The top of the autoclave was removed and the contents inspected. The bricks were about half washed away by this operation, the greatest injury being on the side nearest the inlet steam pipe. The bricks, of course, had no strength at all.

Second Attempt to Autoclave Bricks   A large batch consisting of 2,000 grams of ash, 200 grams of lime, 10 grams of rosin, and 500 grams of water was prepared and placed in the small wet pan, but it was found that this was too big a batch as the material was thrown out the sides of the pan. Then two small batches, each consisting of exactly half the above amount of constituents, were prepared and the two mixed together by hand after being mixed separately for fifteen minutes in the small wet pan. Seven bricks were moulded using a pressure of 2500 pounds per square inch, and then placed in the wet box. It was in the moulding of the seventh brick of this batch that it was found possible to eliminate the use of the lycopodium powder and substitute for it oil and the twisting of the plug relative to the cylinder.

After the bricks had been in the wet box two days, two of them were placed on the brass rack in the autoclave. Then, instead of bringing the steam into the autoclave by a pipe line running to about two inches from the bottom of the apparatus, this pipe inside the autoclave was removed and replaced by a short nipple and elbow so that the incoming steam was directed horizontally and tangent to the inside of the autoclave near the top. This was done in an effort to prevent the incoming steam from splashing condensed water up on the bricks and to divert the force of the steam away from the surface of the bricks.

This time the steam pressure was built up to 70 pounds per square inch over a period of twenty minutes instead of only five. The steam pressure was held between 65 and 75 pounds per square inch for a period of two hours. The supply of steam was then cut off and one hour allowed for the pressure to gradually go down to atmospheric. Upon inspection of the contents of the autoclave, the bricks were found to be badly cracked all over and especially on the bottom. The bottom brick was in worse shape than the top one and its bottom was very weak and crumbly. The tops of the bricks were warped concavely, and the entire brick in each case was covered with a hard crust about one-sixteenth inch thick.

Third Attempt to Autoclave Bricks Before this attempt was made, the valve controlling the incoming steam was changed from a three-eighths inch globe valve to a one-quarter inch needle valve. This was done in an effort to more accurately control the steam supply. A new exhaust water pipe was also arranged so that less water would stand in the bottom of the autoclave. This was accomplished by bending the exhaust pipe within the autoclave so as to fit the curvature near the bottom, with the result that the water depth could be kept down to about one-half inch. Three bricks were then moulded under the same conditions as in the "Second Attempt" and placed in the wet box for two hours. After this they were placed in the autoclave and the needle valve given only one-quarter of a turn for the first half hour. The exhaust valve was so regulated during this time that the steam pressure remained around 5 pounds. The needle valve was then given another quarter turn, and during the next half hour the steam pressure was gradually built up to 75 pounds per square inch where it was held (within 5 pounds) for two hours. An hour was then allowed for the pressure to be gradually released. The next morning, the autoclave was opened and the contents inspected. The autoclave was full of water and the bricks badly washed away. The particles which had washed off the bricks had stopped up the exhaust valve, which worked very poorly during the entire operation. The exhaust line was dissembled and thoroughly cleaned out. The ash had packed tightly, especially in the valve.

Fourth Attempt to Autoclave Bricks Three bricks were made as before except the composition was slightly changed, the proportion of water being reduced twenty per cent. The mixture consisted of 1,250 grams of fly ash, 125 grams of lime, 6.25 grams of rosin, and 250 grams of water. This was mixed in the small wet pan fifteen minutes as before and moulded under a pressure of 2500 pounds per square inch. A few white specks of lime could be noticed after moulding, indicating not quite sufficient mixing.

Before placing the bricks in the autoclave this time, steam was run through the empty autoclave for thirty minutes with the inlet steam valve wide open. This was done to heat up the apparatus and thereby prevent the condensation of so much steam at the start of the operation. The autoclave top was then removed, the rack of three bricks placed inside and the top fastened down. The needle valve was given one-eighth of a turn at the start. The exhaust valve was opened every few minutes to make sure that the level of the condensed water was not above the bottom of the exhaust pipe. A period of thirty minutes was employed in gradually building the steam pressure up to 15 pounds per square inch. By this time the needle valve had been opened a total of one-half turn. About this time a grey color was noticed in the exhaust water, indicating that some of the fly ash was being washed away. During the next fifteen minutes, the steam pressure was built up to 75 pounds per square inch at which pressure it was maintained until two hours from the starting time. The steam supply was then cut off, and the pressure allowed to gradually go down over a period of one hour. The autoclave top was removed and the contents inspected. Very little water was found in the bottom of the autoclave, but the bricks were in very bad condition. Plate I shows the appearance of the bricks after the steam treatment. They were soft and crumbly with the sides toward the steam inlet and outlet badly washed away. No part of the bricks possessed any strength at all.

## EFFECT OF MOULDING PRESSURE AND AGING ON COMPRESSIVE STRENGTH

Basis for Procedure One of the lot of seven bricks made as described under "Second Attempt to Autoclave Bricks" was removed from the wet box after being in seventeen days. Then, after remaining in the atmosphere for three days, it was tested in the Tinius Olsen Machine as shown on Plate IV. The brick was laid on the top of the mould as shown in the picture to insure a smooth surface, for the base plate of the testing machine had a small hole in the center. When the pressure was applied, cracks appeared in the specimen after a load of 31,000 pounds (2,470 pounds per square inch), with the yield point appearing at a total load of 44,850 pounds (3,570 pounds per square inch). The edge of the brick finally cracked off all around for about three-quarters of an inch toward the center. Small particles of lime could be seen where the pieces broke off. The manner in which these bricks failed under compression may be seen on Plate VII which shows a brick before and after failure.

Large Wet Pan Used  In order to make a large batch, it was necessary to use a larger wet pan than previously employed, so the one shown in Plate III was used. A batch of material consisting of 10,000 grams of ash, 1,000 grams of lime, 50 grams of rosin, and 2500 grams of water was made up and mixed in this large wet pan for twenty minutes. This resulted in a very thorough and complete mixing as the mullers were quite heavy and the pan rotated rapidly.

Moulding the Bricks  Twenty-four bricks were made as follows:

8 pressed at 200 pounds per square inch.

8 pressed at 1,000 pounds per square inch.

8 pressed at 2,500 pounds per square inch.

Eighteen bricks, six of each pressure, were placed in the wet box, and two of each pressure left in the atmosphere (in Room 404 Davidson Hall). The six bricks of the same pressure were placed in the bottom part of the wet box one on top of the other, but separated from each other by oil paper. Two of each of these were tested for yield point in compression at the end of one week, and two at the end of two weeks. At the end of four weeks, the remaining six bricks were removed from the wet box and one of each pressure tested for compressive strength. The other three, after remaining in the atmosphere for three days, were tested for tensile strength, using the machine shown on Plate V.

Plate VII

BRICKS BEFORE AND AFTER FAILURE

DUE TO COMPRESSION TESTS

The six bricks left in the atmosphere showed, two days after moulding, a considerable amount of white formation around the outer portion of the upper exposed surface of the brick. This fine white substance had the appearance of lime and was found in the largest quantities on those bricks made at the lower moulding pressure. These bricks seemed to be quite hard (by feeling with the hand), after this two day period. After two weeks were up, they were also tested for yield point in compression. The results of all these tests are shown in the following tables and graphs:

## TABLE II

## EFFECT OF MOULDING PRESSURE AND WET BOX AGING ON COMPRESSIVE STRENGTH

Diameter of brick = 4 inches

Weight of brick = 424 to 479 grams

Height of brick = 1 1/6 to 1 1/3 inches

| Weeks of Aging in Wet Box | Moulding Pressure Lbs./sq. in. | Av. Yield Point Under Compression Lbs./sq. in. |
|---|---|---|
| 1 | 200 | 1520 |
| 1 | 1000 | 3380 |
| 1 | 2500 | 4720 |
| 2 | 200 | 2565 |
| 2 | 1000 | 4400 |
| 2 | 2500 | 6110 |
| 4 | 200 | 3020° |
| 4 | 1000 | 5160° |
| 4 | 2500 | 6850° |

°Only one brick tested.

*These data are used for Graph I following.*

## TABLE III

## EFFECT OF MOULDING PRESSURE AND ATMOSPHERIC AGING ON COMPRESSIVE STRENGTH

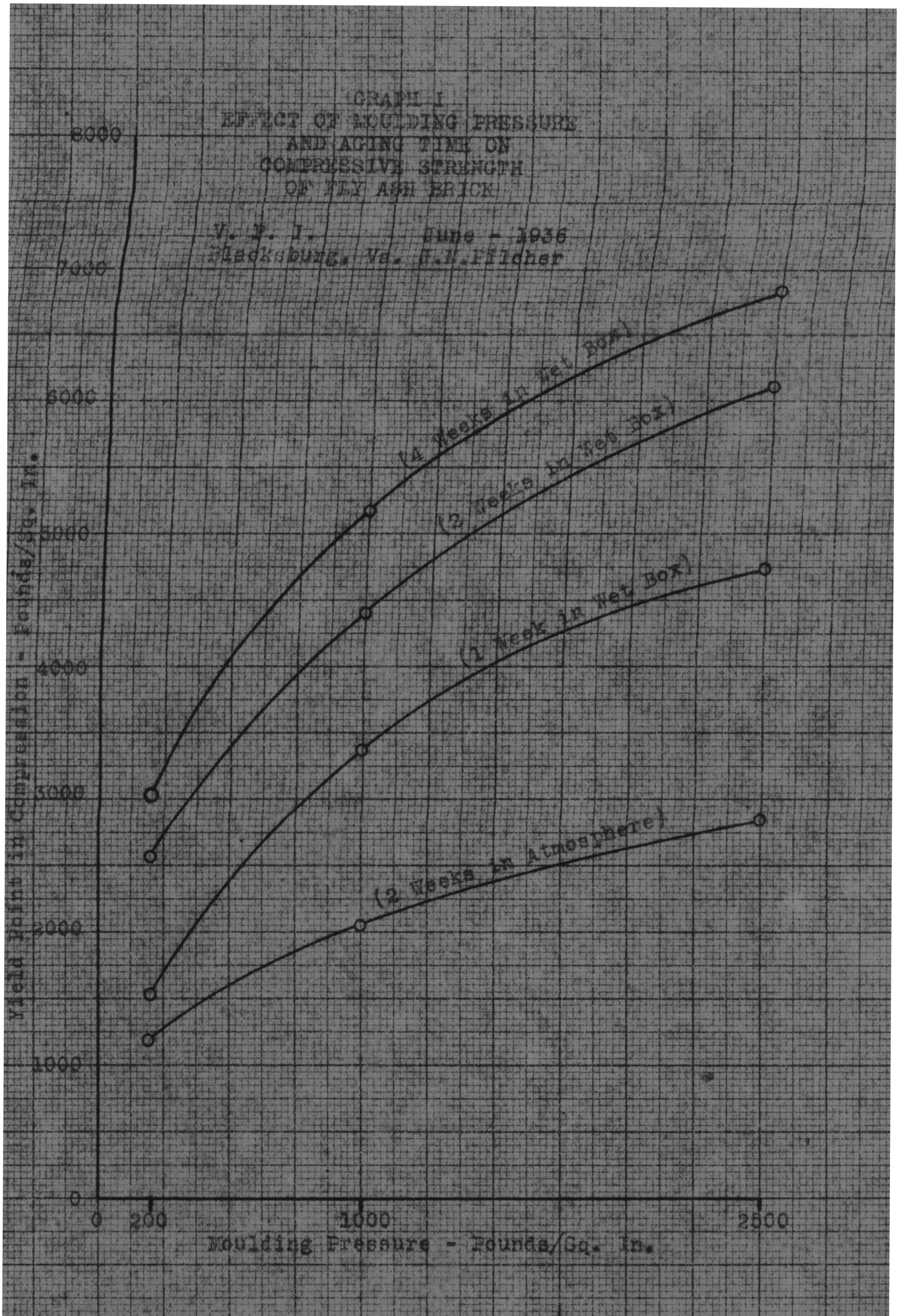
Diameter of brick = 4 inches

Weight of brick = 380 to 397 grams

Height of brick = 1 1/6 to 1 1/3 inches

| Weeks of Aging in Atmosphere | Moulding Pressure Lbs./sq. in. | Av. Yield Point Under Compression Lbs./sq. in. |
|---|---|---|
| 2 | 200 | 1180 |
| 2 | 1000 | 2060 |
| 2 | 2500 | 2845 |

These data are used in plotting the bottom curve on Graph I as indicated.



GRAPH I
EFFECT OF MOULDING PRESSURE
AND AGING TIME ON
COMPRESSIVE STRENGTH
OF FLY ASH BRICK
V. P. I. June - 1936
Blacksburg, Va.
8000
7000
6000
5000
4000
3000
2000
1000
0
0
200
1000
2500
(4 Weeks in Wet Box)
(2 Weeks in Wet Box)
(1 Week in Wet Box)
(2 Weeks in Atmosphere)
Yield Point in Compression - Pounds/Sq. In.
Moulding Pressure - Pounds/Sq. In.

TABLE IV

TEST FOR MODULUS OF RUPTURE USING RIEHLE TESTING MACHINE

Length of span = 3 1/2 inches

Width of specimen = 1 1/16 inches

Aging period = Four weeks in wet box followed by three days in atmosphere.

| Moulding Pressure Lbs./sq. in. | Height of Specimen In Inches | Total Load Taken | Modulus of Rupture Lbs./sq. in. |
|---|---|---|---|
| 200 | 1 9/32 | 70 | 210 |
| 1000 | 1 3/16 | 85 | 300 |
| 2500 | 1 3/16 | 130 | 455 |

Formula used: $M = \frac{3wl}{2bd^2}$

These specimens were prepared by taking the four inch diameter bricks and cutting them with a hack saw to the shape given above. The actual span of the testing machine was three and a half inches, that of the brick being exactly four inches with slightly rounded ends. Plate V shows one of these small specimens in place being tested.

## RESULTS OBTAINED WITH BRICKS MOULDED AT LOW PRESSURES

Mixing of Material   A batch consisting of 8,000 grams of ash, 800 grams of lime, 40 grams of rosin, and 4,000 grams of water was prepared and mixed in the large wet pan for twenty minutes. The consistency of this mix was like that of mortar with a little too much water present. It was so soft that it would not hold its shape at all when moulded. It was then left under atmospheric conditions for two days during which time it dried out considerably and formed a slight film on its exposed surface.

Use of Piston Extrusion Machine   An effort was then made to exude some of this material through a hydraulic piston extrusion machine, but as it came out, it buckled up and soon ceased to run out of the machine at all. When the piston was removed and the material inspected, it was found to be tightly packed in the cylinder. A quantity of water had been squeezed out of the material leaving it only moist and packed quite hard.

Drying in Oven   As the mixture was still too moist to mould, it was placed in a drying oven for three and three-quarter hours, by which time the moisture content had decreased to such an extent that the consistency of the mixture appeared to be about correct for moulding at low pressures. The dry bulb temperature of the oven was 205° F and the wet bulb temperature 190°F.

<u>Moulding</u> The material was not packed in the mould very much by the hands this time, and eight bricks were made, two at each of the following pressures: 50, 100, 200, and 400 pounds per square inch. A little water trickled out of the mould when the 400 pounds per square inch pressure was used. These bricks were placed in the wet box and tested for yield point under compression two weeks after they were mixed, twelve days after moulding. The results are shown in Table V and Graph II which follow:

TABLE V

RESULTS OBTAINED WITH BRICKS MOULDED AT

LOW PRESSURES

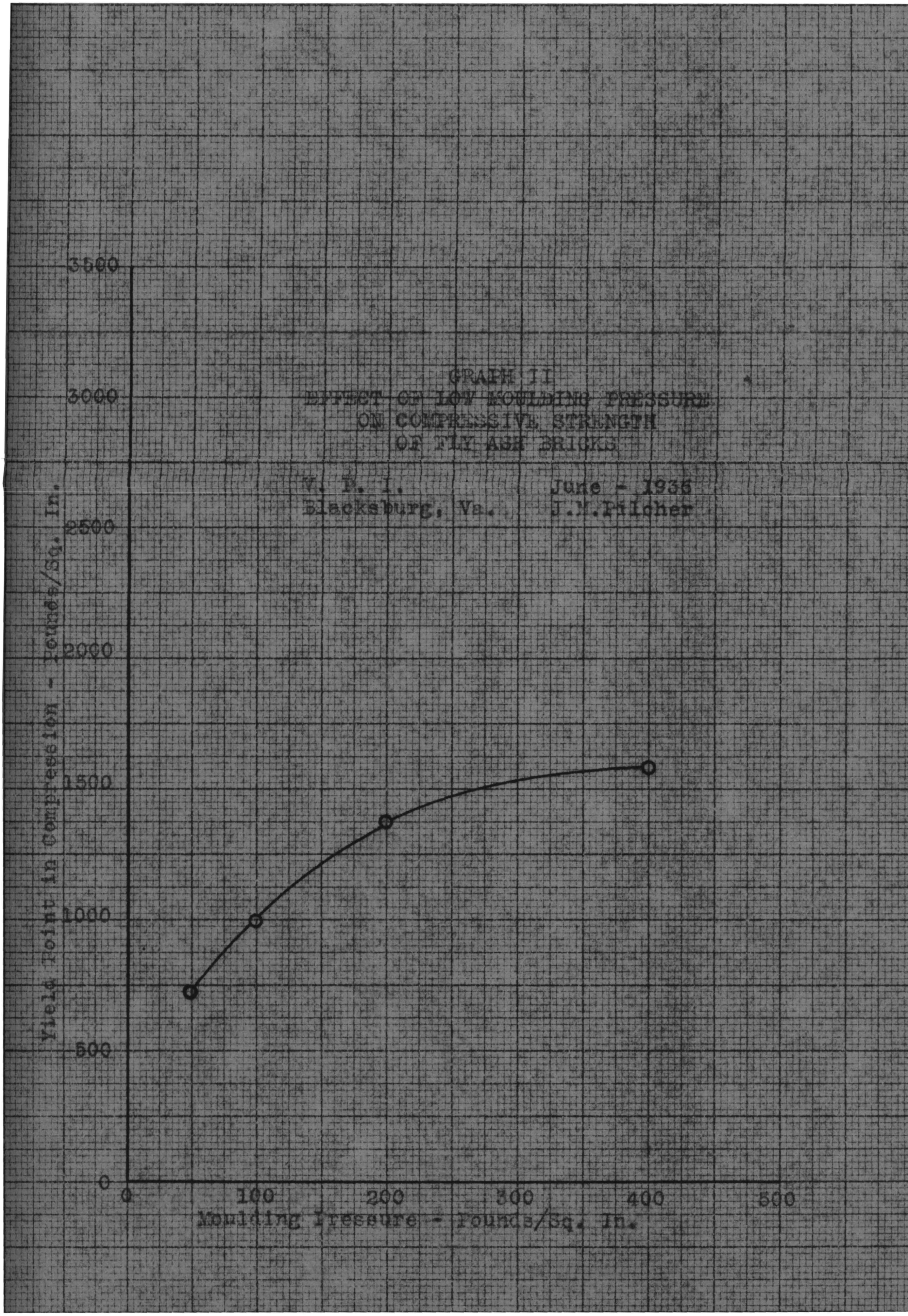
Diameter of brick = 4 inches

Weight of brick = 357 to 393 grams

Height of brick = 1 1/8 to 1 1/3 inches.

| Moulding Pressure Lbs./sq.in. | Av. Yield Point Under Compression Lbs./sq. in. |
|---|---|
| 50 | 730 |
| 100 | 1000 |
| 200 | 1370 |
| 400 | 1590 |

These data are used for plotting the curve on Graph II.


GRAPH II
EFFECT OF LOW MOULDING PRESSURE
ON COMPRESSIVE STRENGTH
OF FLY ASH BRICKS
V. P. I.
Blacksburg, Va.
June - 1935
J.M.Pilcher
Yield Point in Compression - Pounds/Sq. In.
3500
3000
2500
2000
1500
1000
500
0
0
100
200
300
400
500
Moulding Pressure - Pounds/Sq. In.

## EFFECT OF COMPOSITION ON COMPRESSIVE STRENGTH

Plan In order to determine the effect of composition on the compressive strength, a set of three bricks were made under standard conditions, and then additional sets were made with one constituent at a time, varied from this standard. Sets were also made using various proportions of calcium sulfate.

Standard Run The composition of the standard run was as follows:

1000 grams of fly ash

100 grams of lime

250 grams of water

5 grams of rosin

This was mixed for twenty minutes in the small wet pan and three bricks moulded with 1,000 pounds per square inch pressure, and placed in the wet box for two weeks. The three bricks made under the same conditions were placed on top of each other, but separated with oil paper. The tables and graphs which follow show the proportions of the constituent varied and the compressive strengths of the resulting bricks.

TABLE VI

EFFECT OF VARIOUS PROPORTIONS OF

LIME ON COMPRESSIVE STRENGTH

Diameter of brick = 4 inches

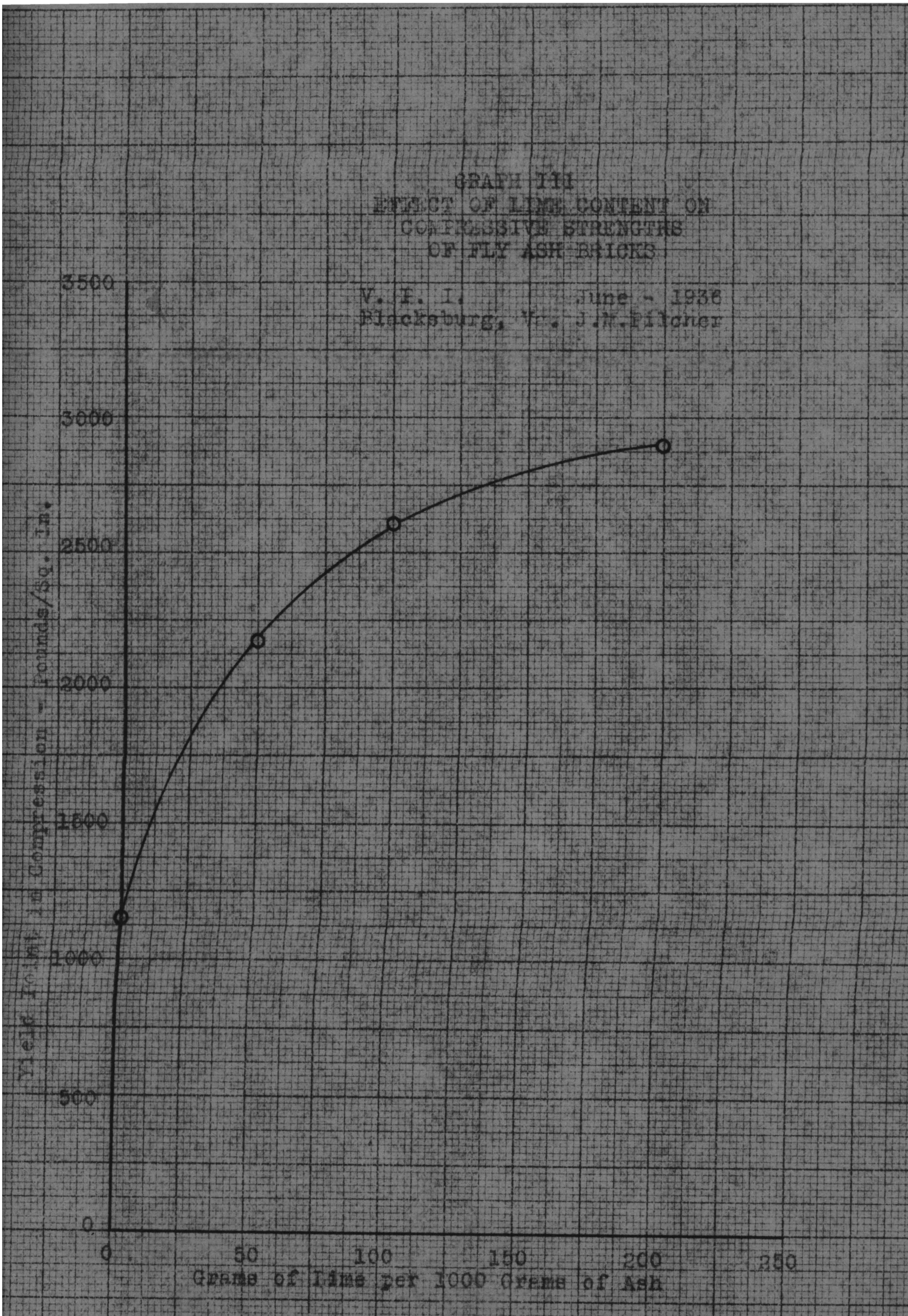
Weight of brick = 406 to 432 grams

Height of brick = 1 1/6 to 1 1/3 inches

All conditions except lime content standard.

| Grams of Lime per 1000 grams of Fly Ash | Av. Yield Point Under Compression Lbs./sq. in. |
|---|---|
| 0 | 1168 |
| 50 | 2174 |
| 100 | 2603 |
| 200 | 2897 |

Comments on Visible Condition of Bricks The three bricks containing no lime at all were considerably cracked around the edges, and the top brick of the three had cracks running all the way across the top. The set containing only 50 grams of lime per 1000 of ash had very slight cracks around the outer upper edges of the bricks, while the other two sets had no apparent defects.



GRAPH III
EFFECT OF LIME CONTENT ON
COMPRESSIVE STRENGTHS
OF FLY ASH BRICKS
V. P. I. June - 1936
Blacksburg, Va. J.M.Pilcher
3500
3000
2500
2000
1500
1000
500
0
Yield Point in Compression - Pounds/Sq. In.
0
50
100
150
200
250
Grams of Lime per 1000 Grams of Ash

# TABLE VII

## EFFECTS OF VARIOUS PROPORTIONS OF ROSIN ON COMPRESSIVE STRENGTH

Diameter of brick = 4 inches

Weight of brick = 393 to 447 grams

Height of brick = 1 1/6 to 1 1/3 inches

All conditions except rosin content standard

| Grams of Rosin per 1000 grams of Fly Ash | Av. Yield Point Under Compression lbs./sq. in. |
|---|---|
| 0 | 3187 |
| 5 | 2603 |
| 10 | 1602 |
| 20 | 196 |

Comments on Visible Condition of Bricks All three bricks of the last batch, (20 grams of rosin per 1000 grams of ash) were badly cracked around the outer portion. These started failing under practically no load and never did take much. None of the other bricks had any apparent defects.

TABLE VII

EFFECTS OF VARIOUS PROPORTIONS OF

ROSIN ON COMPRESSIVE STRENGTH

Diameter of brick = 4 inches

Weight of brick = 393 to 447 grams

Height of brick = 1 1/6 to 1 1/3 inches

All conditions except rosin content standard

| Grams of Rosin per 1000 grams of Fly Ash | Av. Yield Point Under Compression Lbs./sq. in. |
|---|---|
| 0 | 3187 |
| 5 | 2603 |
| 10 | 1602 |
| 20 | 196 |

Comments on Visible Condition of Bricks All three bricks of the last batch, (20 grams of rosin per 1000 grams of ash) were badly cracked around the outer portion. These started failing under practically no load and never did take much. None of the other bricks had any apparent defects.

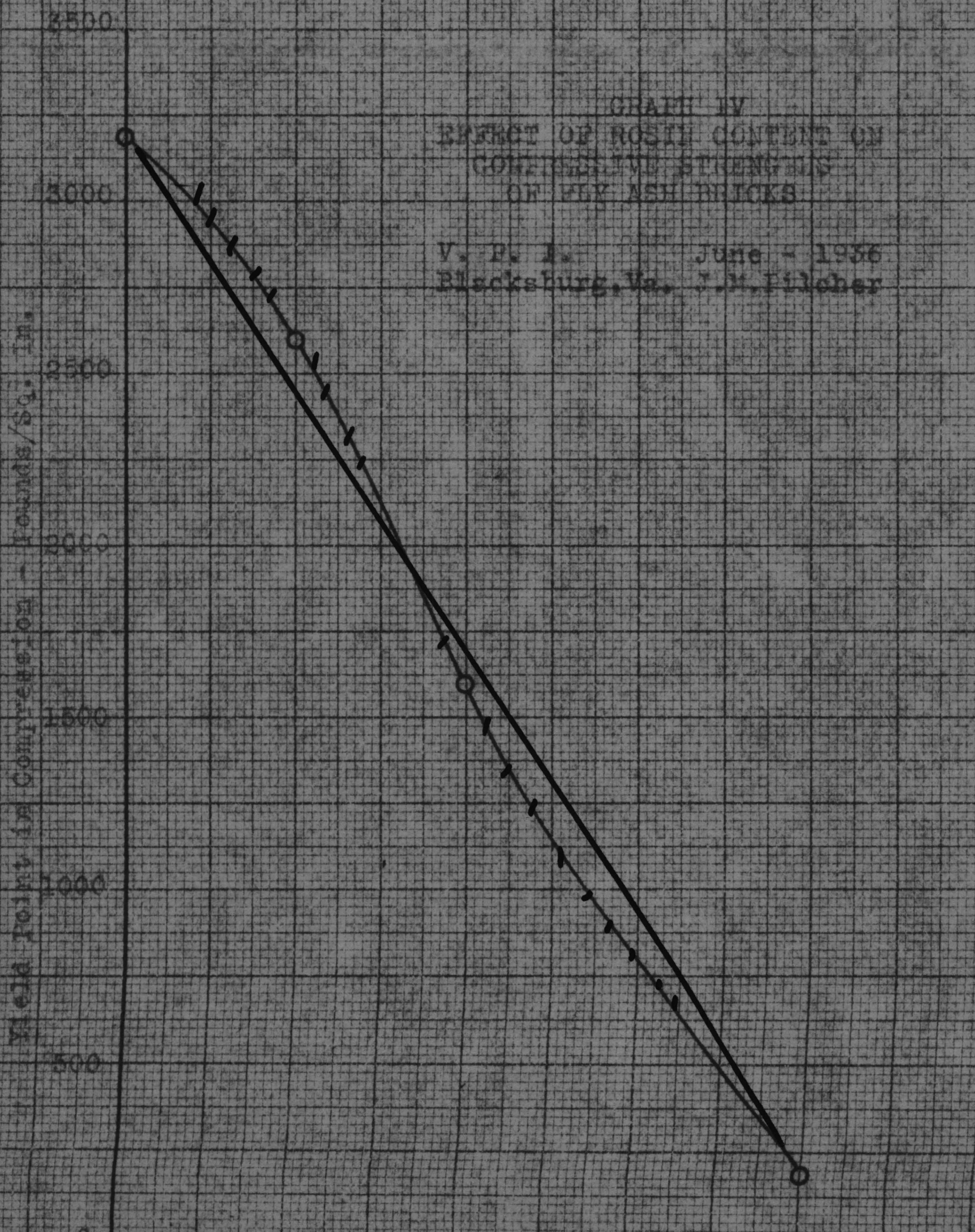

GRAPH IV
EFFECT OF ROSIN CONTENT ON
COMPRESSIVE STRENGTH
OF FLY ASH BRICKS
V. P. I.
June - 1936
Blacksburg, Va.
J. M. Pilcher
3500
3000
2500
2000
1500
1000
500
0
0
5
10
15
20
25
Yield Point in Compression - Pounds/Sq. In.
Grams of Rosin per 1000 Grams of Ash

TABLE VIII

EFFECTS OF VARIOUS PROPORTIONS OF CALCIUM SULFATE ON COMPRESSIVE STRENGTH

Diameter of brick = 4 inches

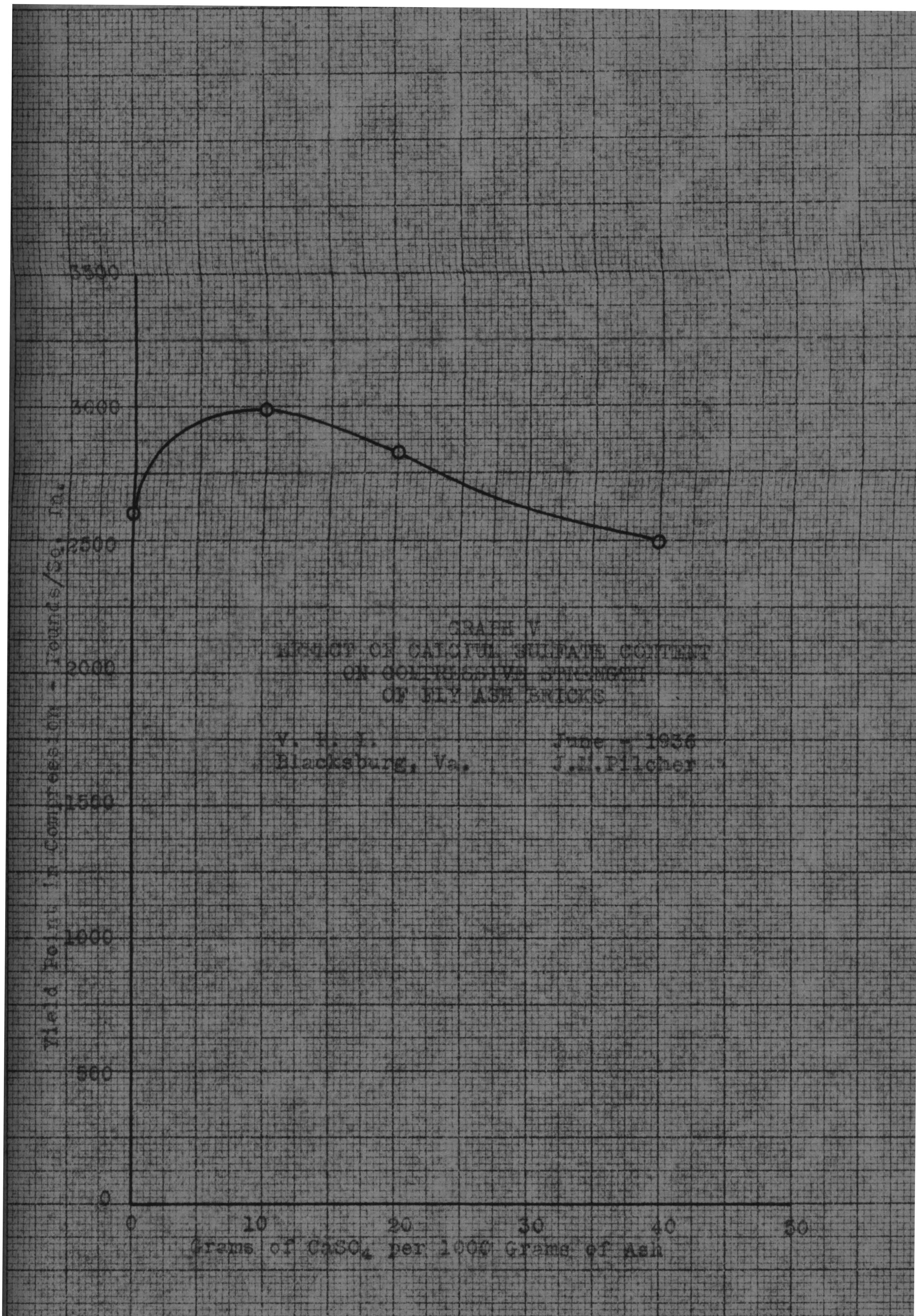
Weight of brick = 413 to 455 grams

Height of brick = 1 1/6 to 1 1/3 inches.

All conditions except $CaSO_4$ content standard.

| Grams of $CaSO_4$ per 1000 grams of Fly Ash | Av. Yield Point Under Compression Lbs./sq. in. |
|---|---|
| 0 | 2603 |
| 10 | 2990 |
| 20 | 2820 |
| 40 | 2500 |

Comments on Visible Condition of Bricks None of these bricks had any visible defects before testing, however, after fracture, small white specks could be seen in the bricks.




GRAPH V
EFFECT OF CALCIUM SULFATE CONTENT
ON COMPRESSIVE STRENGTH
OF FLY ASH BRICKS

V. P. I.
Blacksburg, Va.

June - 1936
J.M.Pilcher

## CHEMICAL AND SIEVE ANALYSIS OF FLY ASH

Chemical Analysis The analysis of the New York Steam Company fly ash and that from the base of the stack of the power plant at the Virginia Polytechnic Institute was made according to the procedure given in " Quantitative Analysis" by F. H. Fish (12) with the following results:

TABLE IX

CHEMICAL ANALYSIS OF FLY ASH

| | V. P. I. | N. Y. S. |
|---|---|---|
| Moisture | 0.17 | 0.15 |
| Combustible | 74.00 | 16.36 |
| $SiO_2$ | 23.35 | 53.25 |
| $Al_2O_3$* | 1.10 | 8.20 |
| CaO | 1.25 | 5.95 |
| MgO | 0.59 | 3.90 |
| $Fe_2O_3$ | 0.80 | 12.20 |

* The per cent $Al_2O_3$ was determined by getting the per cent $R_2O_3$ and $Fe_2O_3$, the difference being $Al_2O_3$.

## CHEMICAL AND SIEVE ANALYSIS OF FLY ASH

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| CaO | 1.25 | 5.95 |
| MgO | 0.59 | 3.90 |
| $Fe_2O_3$ | 0.80 | 12.20 |

* The per cent $Al_2O_3$ was determined by getting the per cent $R_2O_3$ and $Fe_2O_3$, the difference being $Al_2O_3$.

Sieve Analysis Two hundred grams of each ash was used to make the sieve analysis as given by the following table. The set of sieves was placed in the shaker for eight minutes with tapper operating.

TABLE X

SIEVE ANALYSIS OF FLY ASH

| | V. P. I. | N. Y. S. |
|---|---|---|
| Retained on 100 mesh | 89.9 | 7.6 |
| Retained on 200 mesh | 7.7 | 16.6 |
| Retained on 325 mesh | 0.7 | 11.1 |
| Passing thru 325 mesh | 1.9 | 64.1 |

Appearance of Fly Ash The N. Y. S. ash was medium grey in color and felt about like talcum powder. The V. P. I. ash was black and granular with a few light white particles present.

## SOUNDNESS TEST ON LIME USED

Mixing The mix consisted of 30 grams of hydrated lime, 25 grams of Berkshire Silica Sand passing a U. S. Standard 200 mesh sieve and 25 grams passing a 100 mesh sieve (instead of 50 grams passing a B. S. Sieve No. 150), and 120 grams of Standard Ottawa Sand (instead of 120 grams of silica sand passing a B. S. Sieve No. 18 and retained on a B. S. Sieve No. 25), and 13.0 c. c. of water. This was thoroughly mixed with a spatula and tamped into the LeChatelier mould (see Plate VI) by hand. A reference was given to this procedure in the "Literature Review" under the heading of "Soundness Test" (6). The distance between the ends of the indicator arms was measured and found to be 16.0 m.m.

Autoclave Operation The autoclave was heated up for about half an hour by allowing steam to run through it while empty. The filled mould, held together as shown in Plate VI, was hung inside the autoclave with the indicator arms free, not touching anything. Steam was allowed to enter the chamber slowly so that after half an hour the steam pressure had reached 15 pounds per square inch and 100 pounds at the end of one hour. The steam pressure was then maintained at 100 pounds (within 5 pounds) for a little over four hours, after which one hour was allowed for the pressure to gradually go down, the total time in the autoclave being a little over six hours. The distance separating the ends of the indicator arms was again measured and found to be 16.5 m.m., an increase of 0.5 m.m.

## IV DISCUSSION OF RESULTS

Mixing The ball mill cannot be used for mixing this material due to the fact that the mixture packs in the corners of the porcelain jars where it remains until scraped out. This mixing operation is probably the most important part of the process for, unless the lime is uniformly distributed among the fine particles of fly ash the great advantage resulting from the large surface area of small particles will be lost, because it is the area of contact between the two reacting particles which is important. Fine particles make it possible to have this large contact area, but good mixing is also necessary. This fact is clearly brought out when the compressive strength of a brick mixed in the large wet pan is compared with that of a brick mixed in the small wet pan, all the conditions of composition, aging and moulding being the same. Under the standard conditions as described under the heading "Effect of Composition on Compressive Strength" the mix prepared in the large wet pan gave a brick with an average yield point under compression of 4,400 pounds per square inch (Table II), while the brick mixed in the small wet pan for the same time gave a strength of only 2,603 pounds per square inch (Table VI). Also, upon fracture of the brick in the compression tests, white specks could be noticed in those bricks mixed in the small wet pan. This was not noticed in the case of those mixed in the large wet pan.

Moulding The importance of the moulding operation has a significance similar to that of the mixing operation in that its ultimate purpose is to bring the reacting particles in greater proximity with one another. It is, of course, also necessary to give the brick some definite shape by moulding, but this could be accomplished with very little pressure. The increase in strength with increase in moulding pressure as shown by Graphs I and II is due to the greater area of contact over which the particle may react as the moulding pressure is increased and the particles fit in with each other better.

All difficulties in obtaining good results in the moulding operation were overcome when the combination of an oil film and the destruction of the brickplug interface by twisting, was employed. Lycopodium would have worked due to its non-wetting property, but it is expensive and not necessary when the above condition can be easily substituted.

In pressing bricks in a very close fitting mould like the one used in this investigation it is necessary that sufficient time be allowed for the air trapped within to escape. If the reduction in volume is too rapid the air will compress inside the brick and tend to pop the brick open when the top of the mould is removed. If, however, the material were packed in by hand first, the volume reduction during moulding would hardly be enough to cause this trouble. The only time trouble from this source occurred in this work was when the material was dumped into the mould without any packing at all.

<u>Autoclaving</u> The purpose of the autoclave operation is to speed up the reaction between the active ingredients, the oxides and combinations of oxides of calcium, silica, and aluminum. It is quite possible in this steaming operation that the reaction approaches completion more closely in two hours than it would in the wet box in a month, but the economics of the problem must also be considered. The failure of attempts to autoclave bricks as described under "Investigation" was due to mechanical conditions, that is, a lack of the proper design of autoclave. If it had been one in which heat was applied to a closed chamber containing an inch or so of water in the bottom until the desired pressure was reached, it is quite possible the attempt would have been successful. In this work, the steam was taken off a 175 pound (gauge) steam line with the result that it came in at a tremendous velocity causing a splashing of condensed water and considerable damage to the bricks.

If exhaust waste steam were available, which is often true in the summer time, it could be used with the proper type of autoclave very economically.

<u>Wet Box Step</u> The effect of using a wet box as compared with aging in the atmosphere is made significant by two facts: First, Tables II and III and Graph I show that a brick aged two weeks in a wet box is more than twice as strong as the same brick aged the same length of time in the atmosphere. Second, the bricks which were not placed in the wet box had a white powdery formation on the outer portion of the top surface, while none of those placed in the wet box

had this formation. The fact that this occurred only on the top and not the bottom is significant, indicating that it was the rapid drying which caused it. As soon as the brick dries, the reaction stops due to the lack of the catalytic action of the moisture. It is quite possible that a brick which ages in the atmosphere does not appreciably increase in strength after it dries (about one week), while those aged in the wet box continue to increase in strength as shown by Graph I.

Effect of Composition. I Lime. Graph III shows how the strength of the brick continued to increase with increase in lime content, but this increase was not so much past the concentration of 100 grams of lime per 1000 grams of ash. As small cracks began to appear in bricks containing 50 grams of lime and less per 1000 grams of ash, the proportion selected as the standard must be about best.

II Rosin. Considering Graph IV it is readily observed that the less rosin used, the stronger the brick, with 20 grams of rosin per 1000 grams of ash giving a brick of practically no strength at all. For this reason only enough rosin absolutely necessary to check efflorescence should be used, with 5 grams per 1000 of ash considered the upper limit.

III Calcium Sulfate. Calcium sulfate, according to Graph V, increases the strength of the brick when used in small proportions, about one per cent. The cost of this gypsum would be the determining factor as to how much less than one per cent should be used or whether any should be used at all. The results here are comparable with those obtained from the literature (9) in which gypsum was used in natural cement.

<u>Theory of Chemical Reaction</u> The Ternary System of CaO, $Al_2O_3$, $SiO_2$ as prepared by Rankin and Wright in "Chemistry and the Cement Industry" (29) is reproduced on the following plate.

Now, considering the standard run of 1000 grams of fly ash to 100 grams of lime, it is found with the aid of Table IX and the specifications of the lime used as described in "Materials," that the ratio of the components is 532.5 grams of $SiO_2$ to 82 grams of $Al_2O_3$ to 139.5 grams of CaO which means that the percentages of each are 70.6% $SiO_2$, 10.9% $Al_2O_3$, and 18.5% CaO.

The dotted lines on Plate VIII indicate these percentages. From this it is apparent that the most probable ternary compound formed is Anorthite ($CaO.Al_2O_3.\ 2SiO_2$). It is hardly possible that much if any Gehlenite ($2\ CaO.\ Al_2O_3.\ SiO_2$) is formed. A considerable amount of Cristoballite and Wallostonite are probably formed. It seems, however, that most of the reaction in the process is in the formation of the monocalcium silicate just as is true in the manufacture of sand-lime brick (10,18) very little ternary compound being found.

<u>Plasticity of Mixture</u> The results obtained with the hydraulic piston extrusion machine indicate that this material has very little plasticity, not enough to be moulded like ordinary clay brick.

<u>Use of V. P. I. Power Plant Ash</u> Referring to Table IX, it is seen that this ash from the base of the stack of the Power Plant of

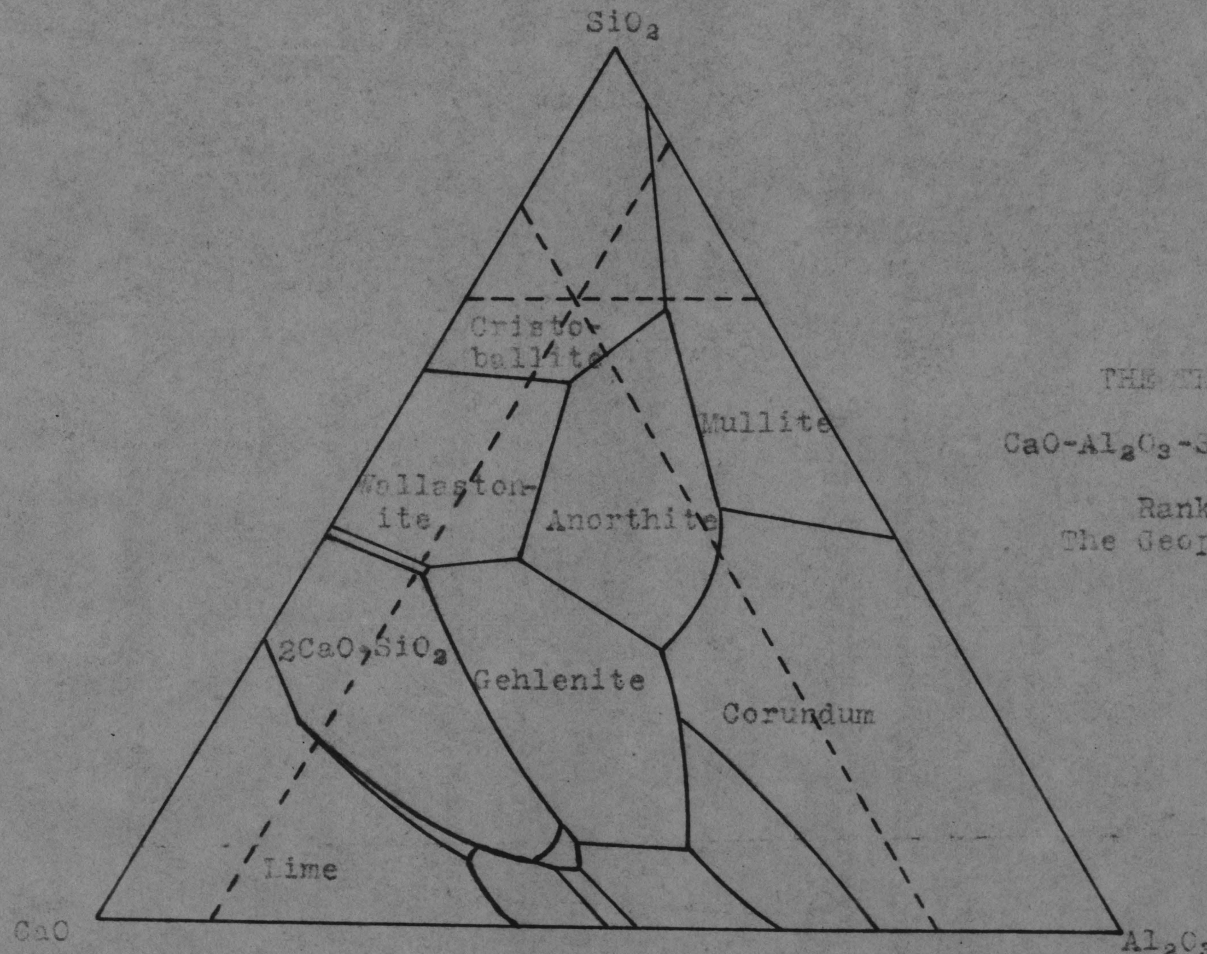


PLATE VIII

THE TERNARY SYSTEM OF

$CaO$-$Al_2O_3$-$SiO_2$. Am. J. Sci 39,1(1915)

Rankin and Wright
The Geophysical Laboratory

the Virginia Polytechnic Institute contains 74 per cent combustible material. This would, in itself, make the ash unsuitable for use as a building material, but might make it suitable as a fuel. However, its use as a fuel is beyond the scope of this investigation. It might be possible, however, if the necessary changes were made in the plant, and fly ash collectors of some sort installed, that the ash could be used for such a purpose as discussed in this problem. It is highly probable that the ash collected at the base of the stack has a higher per cent of combustible material than that which goes out the top, for otherwise practically none of the fuel value would be used.

Economic Considerations All of the raw materials used in this brick are very cheap or else have no value at all except for the rosin which is used in very small proportions. By-product hydrated lime could most likely be used as the source of calcium oxide.

There is the possibility that fly ash might develop a market value in the event that brick made from this material became popular, but as some plants can collect as much as 500 tons a day there is little fear from this angle. Another economic consideration along this same line is that, should it prove desirable to steam treat the bricks, you would, in many cases, be able to use exhaust steam which would otherwise be wasted.

Compressive Strength of Brick According to Emley (11) and to common practice in this country, a brick should be placed in the testing machine so that the load will be distributed uniformly over the specimen. This is usually accomplished by setting the specimen in plaster of Paris before testing, but due to the smooth surfaces of the samples tested in this investigation it was considered necessary to only place the bare brick on a perfectly smooth surface (the mould top) and apply the pressure.

An important fact which must be kept in mind during a consideration of this study is that these bricks were quite thin in comparison to the cross-section area and, roughly speaking, the compressive strength of a specimen will vary directly as the square root of its area, but inversely as its height. However, this will in no way affect the relative results. That is, it does not detract from the value of the information obtained concerning the most desirable composition and conditions of manufacture. In order to compare these compressive strengths with those of ordinary clay bricks as tested by standard A. S. T. M. specifications, the value on the fly ash brick would have to be cut approximately in half, as they are only about half as high.

Modulus of Rupture The modulus of rupture of the brick aged 4 weeks in the wet box and 3 days in the atmosphere, and moulded at a pressure of 2500 pounds per square inch was 455 pounds per square inch, according to Table IV. According to the New York City building code of 1905 this value should average 450 pounds per square inch, with no brick falling below 350. However, Committee C-3 of A. S. T. M. (11) showed that in 1915 Class A bricks had an average modulus of rupture 1482 pounds per square inch; Class B, 668; Class C, 621; and Class D,

469. These last specifications put the best brick of this investigation in Class D, slightly below the average.

Soundness of Lime Used  Although the procedure for determining soundness of lime as described by Bessey and Eldridge (6) was not followed to the letter (because of the lack of B. S. sieves), it was so nearly duplicated that the results may be justly compared. Since these men (6) found that every lime which gave an expansion of less than 5 m. m. produced a sand-lime brick with a compressive strength of at least 3,000 pounds per square inch, this lime appears to be desirable for use in structural material, as its expansion was only 0.5 m. m.

## V CONCLUSIONS

1. The results of this investigation indicate that a brick of fairly high strength can be made by using fly ash as the main constituent.

2. The most desirable ratio of components which should be used in making this brick is approximately 1000 grams of ash to 100 grams of hydrated lime to 250 grams of water to 2 (or less) grams of rosin.

3. Five grams of gypsum may be added to the above if economical and necessary for specified strength.

4. Only enough rosin to control efflorescence should be used.

5. As moulding pressure increases, up to 2500 pounds per square inch, the strength of the resulting brick increases.

6. As aging time in the wet box increases, up to four weeks, the strength of the resulting brick increases; and wet box aging is at least twice as effective as atmospheric aging.

7. The curves indicated that the strength of the brick was still increasing at the maximum moulding pressure and maximum aging time used.

8. The completeness of the mixing operation is a most important consideration in the manufacture of this type of building material.

9. The ash from the power plant of the Virginia Polytechnic Institute is not, at present, suitable for making a fly ash brick.

10. The strength of fly ash brick made under the conditions herein described do not compare very favorably with the strength of ordinary clay brick.

11. The autoclave as described under "Apparatus" is not suitable for steam treating fly ash bricks.

12. The lime used in this investigation was "sound."

## VI SUMMARY

In recent years there has been considerable development along the lines of using powdered coal as the source of energy in the larger power plants. One of its greatest disadvantages, however, is that a very fine ash termed "fly ash" is formed, a large portion of which goes up the stack and out over the surrounding territory. The high per cent of fine silica ($SiO_2$) classifies this "fly ash" as a silicosis health hazard, and the tons which settle over the community make it a nuisance as far as cleanliness is concerned. To prevent these undesirable results, the ash must be collected and disposed of in some way. The purpose of this investigation is to study a process for manufacturing a product, of which "fly ash" is the main constituent, which will be suitable for structural purposes.

The standard run used in this investigation was as follows: first, the mixture consisting of 100 parts of "fly ash," 10 parts of hydrated lime, 0.5 parts of rosin, and 25 parts of water was mixed in a wet pan for 20 minutes; second, the mix was pressed at 1,000 pounds per square inch in a polished steel mould; third, the bricks thus formed were aged in a wet box for two weeks. Each factor in the standard run was then varied above and below the standard, and curves plotted to determine the optimum conditions for each variable. The yield point under compression was the chief criterion for determining

the best value of each variable.

The most desirable composition was found to be 100 parts of fly ash to 10 parts of lime (to 25 parts of water) to 0.2 parts (or less) of rosin and one part of calcium sulfate.

The results of aging tests showed that the strength of the brick continued to increase steadily through the period of time considered, four weeks. The results of varying the moulding pressure showed that the strength of the brick increased with increase in the moulding pressure through the entire range employed, a brick under 200 pounds per square inch pressure being about twice as strong as one moulded at 50 pounds and about half as strong as one moulded at 1000 pounds per square inch.

The lime used in this investigation was tested for "soundness" and found to be in good in this respect.

## VII RECOMMENDATIONS

1. The effect of adding substances commonly used to increase the strength of building materials as, for example, sodium chloride, should be investigated.

2. The possibility of autoclaving the bricks with other equipment than that used here should be studied. There is an autoclave available in the Ceramics Department of V. P. I. which might prove successful. It is the type in which water is placed at the bottom of a closed container to which heat is applied. An attached gauge indicates the pressure developed.

3. The values of moulding pressure and aging time necessary to produce a Class A or B brick should be determined.

4. Data should be obtained to show the relative advantages, from both an economic and technical standpoint, of curing bricks by autoclaving, wet box aging, and atmospheric aging.

5. A survey of the amount of fly ash available and at what cost should be made. The cost of other raw materials should be considered.

6. In any future study of this problem, the sample bricks made should be so shaped that the height of the specimen is nearly the same as its diameter (or width, in the case of a square brick).

7. Data should be obtained to see how the compressive strength varies with height when the cross-section area of the specimen is

maintained constant.

8. The modulus of rupture test should be used more extensively.

9. Data should be obtained to show how these bricks withstand acids, alkali, freezing, exposure to weathering conditions, abrasion, hot flames, and shock. Information on the absorption value would also be desirable.

10. Study the possibility of using fly ash and furnace ash combined.

11. Develop flow sheet, make calculations, and design a plant for making a building material from fly ash.

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