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CERAMIC POSSIBILITIES OF SOME MISSOURI CLAYS AND SHALES

BY

LEONARD NATHANAEL LARSON

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A

THESIS

submitted to the faculty of the  
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the work required for the

Degree of

MASTER OF SCIENCE IN CERAMIC ENGINEERING

Rolla, Missouri

1947

MSM  
HISTORICAL  
COLLECTION

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Approved by

  
\_\_\_\_\_  
Professor of Ceramic Engineering

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## INTRODUCTION

Although many of the shales and clays of Missouri have in the past been, or are presently being used for the manufacture of heavy clay products such as face brick, common brick, sewer pipe, and tile, there is in these post-war years a significant shortage of these products within the state and country. Especially in the northwestern part of Missouri is there a distinct shortage, as only one structural clay plant is known to be operating in that area. The St. Louis and Southeastern Missouri districts are better prepared to meet construction demands, with several structural clay plants operating at peak capacity in these areas.

An investigation of the ceramic possibilities of some of the shales and clays of Northwest and Southeast Missouri would serve to indicate whether the manufacture of structural clay products such as common and face brick, tile and sewer pipe, could be accomplished.

During the past two decades, the use of light-weight aggregates, for producing concretes of unusual levity, has gained widespread use. One of the best and least expensive of these light-weight aggregates is that produced from shale by rapid heating which gives a bloated, cellular structure to the mass. With the present shortage of this commodity, tests on the bloating character of some of the shales of Missouri would indicate which could be used in the production of a good bloated-shale light-weight aggregate.

Waste clay or shale from the manufacture of clay products could be put to good use as a plasticizing agent, replacing lime, in the preparation of masonry mortar, providing commercial standards for the product could be met or exceeded. The use of clays and shales as

plasticizers in masonry mortars has been investigated by several authors, but the Missouri clays and shales have been excluded from these reports.

The plasticity, grain size distribution, amount of colloidal material, type of clay and accessory minerals, and chemical analysis of the clays and shales, and the relationship of one or more of these properties to the ceramic investigation performed on the clays is interesting from both a theoretical and practical standpoint.

## REVIEW OF LITERATURE

Ries (1) defines clay as an earthy substance of fine texture containing a mixture of hydrous aluminum silicates, with fragments of other minerals, such as silicates, oxides, carbonates, etc., and colloidal material which may be either organic or mineral character. The mass possesses plasticity (usually) when wet, and becomes rock-hard when fired to at least a temperature of redness.

Shales are a moderately hard, stratified class of clays, which have been compressed, and sometimes heated. Shales are formed by clays deposited in water and are generally offshore deposits from the borders of lakes, estuaries, or seas. Organic material is often found associated with shale, owing to the nature of its formation. It is not uncommon to find coal associated with shale to some degree. Shales are usually found in layers of considerable thickness, although some shales are deposited in layers only a few feet thick, and contain mineral impurities which usually cause the shales to fire to a red or buff color.

The Missouri shales are usually laid in beds between layers of limestone, sandstone, or other shales. It is the pressure of this overburden material that compresses the apparently homogenous clay mass into a laminated structure. A portion of the water is expelled by this constant pressure, so that the resulting shale is much less hydrous. If this pressure continues through long periods of time

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(1) Ries, H. Economic geology. 7th ed. New York, Wiley, 1942, p. 131

the shales compact more, become less and less hydrous, and undergo a change into clay slate, from which blackboard slate is derived. If metamorphism occurs, the slate will transform into metamorphic schists in which new minerals appear.

Shales form the foundation of an enormous clay industry. While they are of the most impure of clays, it is largely because of this impurity that they are so valuable when used alone, or in addition to other clays, in the manufacture of paving brick, sewer pipe, drain tile, terra-cotta, building brick and other products. Within the last two decades, certain shales have been used in the manufacture of a light-weight concrete aggregate, by virtue of their bloating characteristics when heated to, or beyond, the vitrification range.

Shales usually possess little or no plasticity as they occur in the laminated condition in the unweathered bank. However, shales exposed to weathering conditions, either before or after mining, for periods of a year or more, will develop plasticity. If ground to 10 mesh or finer, shales become more or less plastic, and the plasticity increases with finer grinding. Shales are known to contain a large percentage of clay particles of colloidal size, and the exposing of these particles from the laminated structure by mechanical means gives a plastic condition.

In most shales, incipient vitrification occurs between 1500° and 1700° F., and complete vitrification between 1700° and 1900° F. Above 1900° F., most shales begin to yield and fail as it turns into a melt or glass. It is in the upper limit of the vitrification range that bloating occurs, if the bloating materials are present in the shale,

and subsequently the manufacture of the light-weight aggregate.

Shales are nearly always impure forms of illite, kaolinite, or other clay minerals, high in fluxing and non-fluxing impurities. Free silica is always present, and the total silica is usually between 50 and 75 per cent. The alumina content can range between 10 and 25 per cent. Iron is usually high, found in the form of pyrite, limonite, or siderite, and amounting from 4 to 7 per cent, or higher, in the form of ferric oxide. Lime and magnesia are found in varying quantities, seldom as low as 2 per cent, and sometimes as high as 15 or 17 per cent. The alkalis ( $K_2O$  and  $Na_2O$ ) usually are under 5 per cent. It is the fluxes present in shales that account for its comparatively narrow vitrification range.

Shales may be separated into four general classes, depending upon their chemical and physical characteristics:<sup>(2)</sup>

- a. Siliceous Shales - shales containing free silica through all degrees of sandiness from plastic shales to sandstones. This is the most common type of shale.
- b. Calcareous Shales - shales containing calcium sulphate or carbonate in high percentage so the material approaches limestone or gypsum in composition. Of little value in clay products.
- c. Ferruginous Shales - shales of high iron content that may also be classed as iron ores.
- d. Carbonaceous Shales - shales which were deposited in swamps among growing vegetation, and which have not undergone an oxidation period will be darkened by carbon. The carbon content may be in the form of leaves, lignite, peat, graphite, wood, coal matter, etc.

Shales consist of a large number of minerals which vary greatly

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(2) Wilson, Hewitt. Clay technology. 1st ed. New York, McGraw-Hill, 1927, pp. 33-35.

in amount and kind. The clay minerals associated with shale may be kaolinite, which is a hydrous silicate of alumina ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ). According to Grim<sup>(3)</sup> many shales are composed essentially, or partially, of the clay mineral illite, which is a hydrous silicate of potash, magnesia, alumina, and iron. Ross and Kerr<sup>(4)</sup> have done much work on clay minerals and their identity, and state that the mineral beidellite occurs in many clays and shales, having a composition of  $\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ; beidellite commonly contains ferric iron to some extent which displaces the alumina, forming a hydrous ferric alumina silicate. Many plastic clays, and some shales, contain amounts of the mineral montmorillonite.

Free silica in the form of quartz sand is practically always present in shales in varying amounts, as are feldspars, minerals of iron, calcium, magnesium, and titanium. Most shales are colored by organic matter and occasionally contain fragments or very thin seams of coal or lignite. This organic coloring matter gives the shales various shades of grays, browns, reds, blues or black, and is derived from the finely divided organic matter that settled with the clay or shales at the time of deposition.

#### PHYSICAL AND CHEMICAL CHANGES OF CLAYS WITH HEAT

The firing of clay wares is ordinarily divided into three periods:<sup>(5)</sup>

##### 1. Dehydration

- a. Mechanical dehydration or "water smoking" 20° to 150° C.

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(3) Hursh, R.K., Lamar, J.E. and Grim, R.E. Illinois clays and shales as mortar mix. Ill. State Geol. Sur., Bull. no. 100, pp. 20-22, (1944)

(4) Ross, C.R., and Kerr, P.F. The clay minerals and their identity. Journal of Sed. Pet. Vol. 1, No. 1, pp. 55-64 (1931)

b. Chemical dehydration or "chemical smoking" 150° to 600° C.

2. Oxidation 350° to 950° C.

3. Vitrification 900° C. and above, followed by fusion or melt

In the dehydration period, as heat is applied, the mechanical water, or water of plasticity is first driven off. Hygroscopic water, or the moisture surrounding the clay particles, comes off with more heat, but usually is gone when 150° C. is reached. The chemically combined water of the clay and other minerals present, is usually gone at 500° C., but traces may be present at 800° C. or higher, depending upon the time and temperature of the heating.

The oxidation period, which starts near 350° C. with the oxidation of the easily ignited organic material and sulphur, continues until the last traces of carbon, or other organic matter, are burned out. The oxidation period overlaps the dehydration period, and in some cases may continue into the vitrification period or range of the clay. When the carbon content is high, the oxidation and elimination of sulphur, and the oxidation of the iron compounds will be retarded because of the CO<sub>2</sub> pressure in the pore spaces. Also, the dissociation of calcium and magnesium carbonates will be delayed. If a clay, or shale, is brought up to the vitrification temperature quickly, before the carbon and other gas-forming material has escaped, bubbles or bloating will occur within the melt; in the manufacture of "Haydite"<sup>(6)</sup> this effect is put to advantage, as will be discussed later in this paper. The dehydration

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(5) Wilson, op. cit. p. 145

(6) Hayde, S.J. Process of making brick and similar articles. U. S. Patent 1,255,878, Feb. 12, 1918.



and oxidation periods show an increase in porosity, decrease in weight, and a small increase in volume. This is explained by the loss of water, decomposition of some compounds, and the oxidation of organic matter.

The vitrification of clays begins to take place when the temperature is reached at which the eutectic composition of the clay minerals, fluxes, and free silica begins to liquify. This initial melt, which is classed as the point of "incipient vitrification", further reacts with the silicate minerals, as time and temperature are increased, to increase the fused portion of the mass until complete fusion, or the "slumping" or deformation point is reached. The fused melt flows throughout the pore spaces and voids by the action of surface tension, decreasing porosity and exterior volume until the condition of minimum pore space and volume is reached; this condition is known as "complete vitrification", and the clay has a glassy fracture, extreme toughness, and is harder than steel. Vitrification may then be said to be dependent upon the quantity and viscosity of the fused portion, and the character and amount of the voids and pore space to be filled. The vitrification range, or period, covers the heat treatment of the clay from the point of incipient vitrification to complete vitrification.

Silica in clays exists as the silicate of the clay minerals, the silicate in accessory minerals such as feldspar, hornblende, mica, etc., or as free silica of quartz sand or colloidal silica. Alumina is present in the clay mineral and the alumina-containing accessory minerals such as feldspar. Alumina is the most refractory substance in clay, and the degree of refractoriness varies with the alumina-silica ratio,

and the amount and type of fluxes present.

The iron oxides present in clay and shale are mainly derived from the iron ore minerals of hematite, magnetite and limonite. However, iron may also be introduced by ferromagnesium minerals, amphiboles, siderite, or pyrite. Iron is usually present in the ferric condition, but any ferrous iron will be oxidized to the ferric state under oxidizing kiln conditions during firing, giving rise to the red color of most clay products. The iron oxides act as active fluxes during firing, especially if free silica is present in quantity.

The presence of lime (CaO) in amounts above three per cent is detrimental to clay ware, as the eutectic of lime and some of the mineral components is suddenly formed into a low viscosity glass, which subsequently lowers the vitrification range of the clay body. The lime originates from limestone and gypsum and may be present in clay in fragments or it may be finely disseminated. If fragments of lime, which do not go into solution during firing, are present in the finished ware, they will easily hydrate and the resulting expansion will sometimes crack or spall the surface. Lime is an extremely active flux, especially in the presence of iron oxides, and has some decoloring effect on the iron oxide reds. Wheeler (7) found that a shale located in a railroad cut near Aldrich, Missouri, containing 11 per cent lime, passed from a soft, porous, unvitrified condition at 1190° C. into a fused, glassy melt at 1215° C.. Likewise, a shale found near Barrett Station in St. Louis County, which has 8 per cent lime and

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(7) Wheeler, H. A. Clay deposits, Missouri Bureau of Geology and Mines. Vol. 11, 1896, p. 72.

4.16 per cent magnesia, has a very short vitrification range.

Dolomite or magnesium limestone is the chief source of magnesia in clay, although it also may be introduced by ferro-magnesium minerals. Magnesia is considered to be a less dangerous flux than lime, as it does not produce the fluidity of lime and exerts less fluxing action. Magnesia is usually found in clays and shales, but in smaller quantities than is lime.

The alkalies ( $K_2O$  and  $Na_2O$ ) are usually present in soluble form, but feldspars and other minerals contribute soda and potash in varying amounts. The clay mineral illite contains potash in chemical combination. The alkalies are powerful fluxes, even at low temperatures. The soluble forms are more readily fused than are the silicates of sodium and potassium.

#### LIGHT-WEIGHT CONCRETE AGGREGATES

The need for lightweight building materials was created in the latter part of the nineteenth century when building design was changed from the thick, heavy, load-bearing walls, to a framework of beams and columns with thin walls, by the introduction of structural steel, followed by structural concrete and reinforced concrete. These new methods of construction made possible the erection of "skyscraper" buildings, long span bridges, and floor additions to existing buildings.

It was not until about the time of the first World War that light-weight concretes were recognized and used extensively in the erection of large buildings. The light-weight concretes soon entered the field of residential buildings and small commercial structures, in which the light-weight concretes were desirable for the manufacture of prefabricated, large-size, masonry units.

Many methods have been used by the concrete industry to produce lighter weight concretes and cement blocks. The introduction of light-aggregates, such as pumice, volcanic ash, furnace slag and cinders, sawdust, coke "breeze", and bloated shales, has been the primary method of producing light-weight concretes to date, although a cellular concrete has been developed by the use of aluminum bronze powder.

The manufacture of a light-weight concrete aggregate from shale or clay was first patented by Stephen J. Hayde <sup>(8)</sup> on February 12, 1918, and later improved as described in reissued United States Patent 1,707,395 of April 2, 1929. Hayde took advantage of the cellular structure formed by the expansion or bloating of vitrified or fused shale in this product, which he named "Haydite". Of the many light-weight aggregates used throughout the country, those made from burned clay or shale form a very important part, and have the definite advantage of being chemically inert and acid resistant.

The bloated shale type of light-weight aggregate can be made from a great variety of shales and some impure clays. The raw shale or clay is taken directly from the quarry or bank, crushed to pass a  $\frac{1}{2}$  inch screen and then fed into a rotary kiln, usually of a 50 foot length, fired by pulverized coal, oil or natural gas. The kiln temperatures are determined by the vitrification and bloating temperature of the raw material, and are usually above 2000° F. at the discharge end, and about 600° F. at the feed end. The shale or clay remains in the kiln about 45 minutes, and just before it reaches the

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(8) Hayde, S. J., op. cit.

discharge end of the kiln, incipient fusion results. At this point, the material softens, having a consistency about like chewing gum. Escaping gases are trapped and form a cellular structure with an increase in volume of approximately 50 per cent. The material is discharged, cooled, crushed and screened to various aggregate sizes. At plants using a raw material containing excess calcium oxide, water is added to the kiln-discharged material to hydrate any quicklime that may have formed during the burning process. Insufficient heating or complete fusion of the material will result in a non-uniform cellular structure and a lower crushing strength of the aggregate.

The gases evolved in the shale that cause the bloating or expansion are:

1. Entrapped air
2. Steam
3. Sulphur dioxide and trioxide
4. Carbon dioxide or monoxide
5. Oxygen
6. Hydrocarbons absorbed during firing
7. Vaporization of iron, if in excess (9)

As the shale or clay is fired to vitrification in a short period of time (40 to 45 minutes), it is impossible for the gases formed by the carbon, sulphur, and carbonates to entirely escape, and an expansion of the mass results as the point of incipient fusion is reached. Some mechanical water is also trapped by the quick firing, and the resultant steam aids the expansion. It is important to discharge the fused, expanded material quickly, or the viscosity of the fluid portion will permit the excess swelling of the gas vesicles and they will communicate with the surface, rendering a porous mass instead of a cellular

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(9) Whitlatch, Geo. L. The clays of west Tennessee. Tennessee Geological Survey. Bull. 49, 1940, p. 26.

structure.

Bloated shale aggregate concrete weighs 30 to 60 per cent less than concrete made with sand, gravel or crushed rock aggregate, and with the same amount of cement it produces concrete comparable in strength to those made with natural aggregates. Special use concrete has been made with bloated shale aggregate weighing as little as 60 lbs. per cubic foot.

Bloated shale aggregate concrete has a low heat conductivity value, the value of "K" (BTU/sq. in./in. thickness/degree difference in temperature/hour) for 80 pound concrete at 75° F. is about 2.5.<sup>(10)</sup> Because of this property, bloated shale aggregate concretes have been developed as an insulating material for temperatures as high as 2000° F.. For the high temperature insulating mixes, an alumina cement is used to insure a certain amount of refractoriness.

Other advantageous properties are those of sound absorption, resistance to fire attack, nailability, adaptability as a plaster base, ease of cutting, and a low coefficient of expansion.

Occuring in nature are many clays that will vitrify and still retain a natural levity and high porosity, rendering them valuable for light-weight clay products and aggregates for light-weight concretes. Some clays have lignitic material evenly distributed throughout the clay mass to such an extent that the ignition loss sometimes runs as high as 70 per cent, producing a porosity of 60 per cent or greater, depending on the method of brick formation.

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(10) Cleary, John B. Haydite manual. American Aggregate Co. Kansas City, Mo. 1940, p. 26.

In sections of Southeast Missouri, there is a clay which has a natural levity, both in the green and fired state, occurring in the Porters Creek formation of the lower Eocene series of the Tertiary system. The Porters Creek clay occurs in the states of Missouri, Illinois, Tennessee, and Mississippi, and the lightness, quality, and quantity varies in different localities. The fired specific gravity varies from as low as 1.07 to as high as 2.00, (11) dependent upon the locality and the cone to which it is fired. The apparent porosity is roughly inversely proportional to the specific gravity.

Although Porters Creek clay is composed essentially of montmorillonite, there is much disagreement among geologists as to the origin of the deposit, because of its widespread distribution. The Porters Creek clay breaks with a conchoidal fracture, similar to the fracture of Missouri flint clay, and is practically non-slaking in the presence of water. When ground to pass 20 mesh, and mixed with water, the clay has good plasticity, although abnormally large amounts of water are necessary to produce the plastic condition, being usually above 60 per cent water; whereas most brick clays require about 20 per cent to give the desired plastic condition.

Theories concerning the levity of Porters Creek clay have been advanced by several authors. Grim<sup>(12)</sup> states that the total porosity is increased by the random orientation of the montmorillonite flake-like particles. Whitlatch<sup>(13)</sup> believes siliceous hydrogels in the form

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(11) Whitlatch, Geo. I. op. cit. pp. 212 - 213.

(12) Grim, R. E. Petrography of the fuller's earth deposits, Olmstead, Illinois. Ill. State Geo. Sur., R.I. No. 26, 1933, p. 355.

(13) Whitlatch, Geo. I. op. cit. pp. 204 - 205.

of roughly spherical gelatinous masses may form in the voids and pore spaces between the clay particles during tempering with water. Meyer<sup>(14)</sup> advances the theory that relatively thick imbibed water films on the surfaces of the clay particles, as evidenced by the high percentage of water of plasticity, may account for a decrease of molecular particle attraction. Upon drying, the low molecular attraction is not sufficient to collapse these hydrogel masses, and the approximately spherical shape of the voids results among the clay particles, increasing the porosity of the mass.

Tests have been conducted by the Mississippi Geological Survey <sup>(15)</sup> on concretes utilizing light-weight aggregate made from Porters Creek clay. The clay was calcined to 1800° F., jaw-crushed and screened. The sized aggregate was mixed with Portland cement in from 1:4 to 1:6 ratios on a volume basis. The resulting concrete weighed as little as 70.2 lbs. per cubic foot, and had good crushing strength and workability, with a heat conductivity about one-fourth that of concrete made with sand and gravel aggregate.

Whitlatch <sup>(16)</sup> has made an extensive survey of the Porters Creek clay in Tennessee, and its possible uses in light-weight ceramic brick and structural units, and as an aggregate for light-weight concretes. Although shrinkage is usually excessive in the Porters Creek clay,

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(14) Meyer, W. W. Colloidal nature and related properties of clays. Journal of Research, U. S. Bureau of Standards. Washington. Vol. 13, No. 2, 1934, p. 256.

(15) Morse, W. C., McCutcheon, T. E., and Mandlebaum, B. F. Light-weight aggregate. Mississippi State Geological Survey. Bulletin 61, 1945, 56 pp.

(16) Whitlatch, Geo. I. op. cit. pp. 200 - 242.



making warpage and sizing difficult to control in the finished product, light-weight clay units may be dry-pressed or extruded by the stiff-mud process. The porosity of the fired product results in excellent acoustic and insulation properties, as well as an advantage in ease of handling because of its light weight.

#### DIFFERENTIAL THERMAL ANALYSIS FOR THE DETERMINATION OF THE CLAY MINERALS

Clays and shales are primarily composed of extremely small (usually 5 microns or smaller) particles consisting of one or more of the minerals comprising the "clay material group". (17) The clay material, or clay minerals, which are predominant in nature are:

1. Kaolinite group - with related minerals dickite and nacrite
2. Montmorillonite group - bentonite, saponite, nontronite and beidellite
3. Illite group - name applied to constituents of clay materials similar to the white micas; also called hydromuscovite

As heat is applied to any of the clay minerals, endothermic and exothermic thermal reactions take place within the material, the temperature and magnitude of which are peculiar to the individual mineral. The use of these thermal reactions for studying the composition of various clays and hydrous materials was first suggested by le Chatelier in 1887. (18) Other investigators have applied this method with considerable success, and in 1913, Wohlen (19) published a paper dealing

(17) Grim, R. E. Modern concepts of clay materials. *Journal of Geology*. Vol. 50, No. 3, pp. 228-230 (1942)

(18) Le Chatelier, H. The action of heat on clays. (French). *Bull. Soc. Franc. Min.* Vol. 10, 1887, pp. 204-211.

(19) Wohlen, R. Thermal analyses of clays, bauxites and some allied materials. (German). *Sprechsaal*. Vol. 46, 1913, pp. 719-783.

with the thermal analysis of clays and associated minerals. Orcell (20) has done considerable work along these lines, and in 1939, Norton (21) presented a comprehensive paper on clay mineral determination by the differential thermal method.

The method of mineral identification by the differential thermal analysis consists essentially, of heating a small quantity of the material at a constant rate up to about 1000° C., or as near to fusion as is possible, and recording the endothermic and exothermic effects that occur. A plot of the differential temperature against the constant temperature increase will give a curve indicating the endothermic and exothermic reactions and the respective associated temperatures. Liberation of chemically combined water, compound decomposition, changes in crystal structure, or compound formation will produce endothermic or exothermic peaks on the differential thermal analysis curve. The characteristic peaks for any mineral possessing thermal properties are quite distinctive, and a mixture of minerals will give superimposed reactions corresponding to the effects of the individual minerals, as in X-ray diffraction analysis.

Grim and Rowland, in two recent investigations, (22)(23) have attempted to clarify some of the controversial issues in the works of previous authors, in addition to presenting results of non-homogeneous

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(20) Orcell, J., and Cailliere, S. Differential thermal analysis of montmorillonite (bentonite). (French). Comptes Rendus. Vol. 147, 1933, pp. 774-776.

(21) Norton, F. H. Critical study of the differential thermal method for the identification of the clay minerals. Journal American Ceramic Society. Vol. 22, pp. 54-63 (1939)

materials such as shale, brick clays, and artificial mixtures of minerals of the clay material group and clay-impurity minerals. They also used methods of recording slight differential thermal reactions, giving rise to the identification of minerals by this process, which was formerly impossible because the measurement of the differential temperature was not sufficiently accurate.<sup>(24)</sup> Berkelhamer,<sup>(25)</sup> in a thorough report gives details of construction and operation of a comparatively simple apparatus for differential thermal analysis.

Many of the authors differ somewhat on the exact amount of endothermic and exothermic reactions, and the respective temperatures in some of the minerals. Such differences are probably due to the various rates of heating used by the different investigators, and impurities occurring in the samples. To get accurate results, each set of apparatus should be checked against standard samples of interested minerals, and these curves may then be used as a standard for further unknown identification purposes.

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- (22) Grim, R. E., and Rowland, R. A. Differential thermal analysis of clay minerals and other hydrous materials. *The American Mineralogist*. Vol. 27, No. 11-12, pp. 746-761, 801-818 (1942)
- (23) Grim, R. E., and Rowland, R. A. Differential thermal analysis of clays and shales, a control and prospecting method. *Journal American Ceramic Society*. Vol. 27, No. 3, pp. 55-76 (1944)
- (24) Grim and Rowland. *American Mineralogist*. op. cit. p. 747.
- (25) Berkelhamer, L. H. An apparatus for differential thermal analysis. U. S. Bureau of Mines. Report of Investigation 3762. July, 1944.

## THE USE OF SHALES AND CLAYS IN MORTAR MIX

Ordinary masonry mortars, mixed on the site, usually consists of a mixture of Portland cement and hydrated lime. However, the cement manufacturers have undertaken the preparation of prepared masonry cements by additions of pulverized limestone, shale, or clay refuse from clay products plants. In an effort to correlate properties of the admixed material with the properties of the cement, much experimental work has been carried out in the last decade. Earliest experimental work was reported in 1891 (26) on the results of tests on mortar made with well-washed sand and cement compared with mortar made with cement and a silt-sand, which contained 18 per cent clay, with a cement-sand ratio of 1:2.

The primary function of a masonry mortar is to bind the masonry units into a strong, long-lasting structure. The ordinary lime-Portland cement mortar is adequate as far as strength is concerned, but a number of other properties are necessary in determining the utility of the masonry mortar. Schurecht (27) lists the following properties that are determining factors in the quality of brick masonry mortar:

1. The mortar should have excellent adhesion to the brick. This is especially true for the vertical joints which are not under pressure and will be subjected to tensile stresses.
2. It must produce maximum waterproofness of the wall by reason of its durability of adhesion under conditions of service.

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(26) Grant, W. H. Notes on cement, mortars, and concretes. Trans. Amer. Soc. Civil Eng. Vol. 25, Paper 498, p. 269 (1891)

(27) Schurecht, H. G., and Corbman, M. The use of New York State clays in masonry mortars. New York State College of Ceramics. Experiment Station Bulletin No. 1, 1937

3. The mortar should have such body characteristics that it will not fall away when the joints are formed, thereby saving mortar.
4. It must have a good plasticity and workability, making it possible to lay more wall per day and permitting better trowelling.
5. It should, by its attraction for moisture and the sealing of the pores in the mortar, retain water long enough and in sufficient amount for the cement to set properly.
6. It should not dry out rapidly on the mason's boards.
7. The mortar must not bleach or burn mortar colors, nor should it be injurious to the mason's hands.
8. It must not be deteriorated by heat.
9. It should be made of ingredients which do not have to be added in the form of putty, and the mixing must be easily performed.
10. It must not cause efflorescence on the brick work.
11. It should remain practically constant in volume under conditions of service.
12. The mortars should be resistant to freezing to insure a durable bond in severe climatic conditions.

The ingredients that produce the above listed properties in masonry mortars are known as plasticizers, but it is difficult to find a plasticizer that will function effectively for all mortar properties. The use of clays or shales as plasticizers in many cases will give excellent mortar properties at a minimum of cost.

Earliest use of clay as a plasticizer in mortar was in 1884 when a cement mortar was used in kiln construction at Brazil, Indiana.<sup>(28)</sup> Superiority was claimed for these mortars, over mortars made with

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(28) Straight, H. R. Clay manufacturers find a new market using waste clay for mortar mix. Brick and Clay Record. Vol. 85, No. 2, pp. 51-52 (1934)

other plasticizers, in weather resisting qualities. In 1888 a building was erected near the same town using fire clay substituted for one-half of the cement; after thirty-five years, this mortar proved to be in exceptionally good condition.<sup>(29)</sup>

The use of clays or shales as a plasticizing agent in masonry mortar has been rightly viewed with suspicion, because of the organic material present which interferes with the setting and hardening of the Portland cement.<sup>(30)</sup> Also, many clays and shales contain appreciable quantities of alkali metal compounds, which are set free in the mortar by base exchange with the calcium ion of the lime, and these alkalis are probable to appear on the wall surface as efflorescence. The efflorescence may be tested by drawing up moisture through the clay in question, and examining for alkali salt encrustation along the sides during evaporation: if practically none appear within two weeks, the clay may be regarded as satisfactory.<sup>(31)</sup>

Straight,<sup>(32)</sup> using clay to replace twenty-five per cent of the cement in 1:3 mortars, states that the use of clay "does not burn or bleach mortar colors; it gives better adhesion and suction and is therefore desirable for hollow-tile work; it seals pores and holds

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(29) Ibid.

(30) Anderegg, F. O. Mortar properties improved by adding mortar mix. Brick and Clay Record. Vol. 90, No. 5, p. 280 (1937)

(31) Smothers, W. J. The effect of clays on the properties of masonry cement. Thesis, Missouri School of Mines and Metallurgy, Rolla, Missouri.

(32) Straight, H. R. op. cit. p. 51

moisture that the mortar needs for setting and development of strength; suitable plasticity is obtained with a less amount of clay than of lime".

Spangler<sup>(33)</sup> found that clay could be used in the same proportion as hydrated lime in cement mortars, without changing the strength notably, in the range up to 30 per cent replacement of cement by clay.

Palmer and Parsons,<sup>(34)</sup> in their work with air floated shales, found that the necessary plasticity was added to mortars, with a high water-retaining capacity, and the mortars developed a maximum bond without being dried out by the porous brick.

Anderegg<sup>(35)</sup> states that finely ground shales gave good results in mortars, but do not contribute to the flexibility of mortar that lime does, although the shrinkage may be reduced. The shales also give the advantage of a low creep to the mortar, which is an essential in reinforced concrete construction.

A recent and extensive investigation of the use of clay materials as plasticizers was carried out at the New York State College of Ceramics.<sup>(36)</sup> The clays and shales, from New York and Iowa, were ground so that 80 per cent passed 100 mesh. It was found that the substitution of finely ground clays for lime in masonry mortars

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- (33) Spangler, M. G. Ground clay as a plasticizing agent. *Journal American Ceramic Society*. Vol. 13, pp. 927-928 (1930)
- (34) Palmer, L. A., and Parsons, D. A. Properties of mortar and bricks in relation to bond. *Bureau of Standards Journal*. Vol. 12, Research Paper RP683, 1934, p. 12.
- (35) Anderegg, F. O. op. cit. p. 280.
- (36) Schurecht, H. G., and Corbman, M. op. cit.

raised the adhesive strength about 55 per cent, the compressive strength was increased 36 per cent, and the average tensile strength was raised 40 per cent. The mortars, which were made with 10 and 25 per cent clay as a plasticizer, stood up very well after 30 cycles of freezing and thawing, although a slight loss of strength developed, and the shrinkage was less than corresponding mortars made with lime as a plasticizer. The water retention of clay-cement mortars was generally higher than that of lime-cement mortars on the basis of equivalent bond.

Hursh,<sup>(37)</sup> in an extensive examination of shales and miscellaneous clays, used in mortar mix, found that grinding to pass 80 or 100 mesh had little effect on the strength of the mortars, as compared with results from an 8 mesh grind of the same samples. Mortar containing shale or clay in amounts up to 40 per cent generally showed greater strength than samples of commercial masonry cement mortar.

The use of clay or shale as a plasticizing agent in mortar mix has the advantages over lime of a lower initial cost, better freezing and thawing properties, better adhesion, reduced shrinkage, and better water retention qualities.

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(37) Hursh, R. K., Lamar, J. E., and Grim, R. E. op. cit. p. 39.



## DISCUSSION

Nine shales, two Porters Creek clays, and a loess, were obtained through the Missouri Geological Survey. Representative samples were secured, in about 100 lb. lots, by selecting material, along a vertical plane, from the entire exposed face of the sedimentary outcrop. A list of the samples, and their locations, is given in Table I.

TABLE I

Number	Name and Location of Sample	Geologic Location
A - 1	Cherryville Shale - Jackson Co., Sec. 35, T48N, R33E	Pennsylvanian
B - 1	Loess - Stoddard Co., East side County Hwy. North of Bell City, Sec. 2, T27N R11E	Pleistocene
B - 2	Porters Creek Clay - Stoddard Co., channel sample of 30' face, 1/4 mile SW of Ardeola School, SW 1/4 SW 1/4 NW 1/4 Sec. 10, T27N, R11E	Tertiary, Midway Group
B - 4	Porters Creek Clay - Stoddard Co., channel section 30' face, SE side Poplar Branch Creek, 1/2 mile SW of Avert, Mo. NE 1/4 SE 1/4 Sec. 3, T26N, R11E	Tertiary, Midway Group
B - 6	Maquoketa Shale - Cape Girardeau Co., 10' face at North Main & Broadway, Cape Gir- ardeau, Mo.	Ordovician
B - 7	Chester Shale - Ste. Genevieve Co., Renault formation, West side of Hwy. 25, 1/2 mile North of St. Marys, Mo.	Mississippian, Chester Series
C - 1	Pleasanton Shale - Jackson Co., East side Noland Road, South of Little Blue Bridge, Upper 25' of lower part of Pleasanton . Shale (below Knobtown Sand.), 25' exposed of 60' bed	Pennsylvanian

TABLE I (Cont.)

Number	Name and Location of Sample	Geologic Location
C - 2	Pleasanton Shale - Jackson Co., upper 18' of Pleasanton Shale (above Knobtown Sand.), Sec. 27, T48N, R32E	Pennsylvanian
C - 3	Weston Shale - Platte Co., upper 30', one mile South of New Market, Mo., East side of U. S. Hwy. 71, SW 1/4 Sec. 14, T54N, R35E	Pennsylvanian
C - 4	Lawrence Shale - Buchanan Co., 52' between Haskell & Amazonia, old shale pit, South St. Joseph, Sec. 29, T57N, R6E	Pennsylvanian
C - 5	Lagonda Shale - Bates Co., (uppermost member of Cherokee), East side of U. S. Hwy. 71, just North of Miami Creek, 6 miles South of Butler, Mo.	Pennsylvanian
D - 1	Northview Shale - Webster Co., on North side of U. S. Hwy. 66, just West of County Road "B" spur to Northview, Mo. composite of plus & minus 20', SW SW Sec. 23, T30N, R19W	Mississippian

The physical structure of these clays is varied, as can be seen from the properties listed in Table II.

TABLE II

## Physical Structure and Related Properties

A - 1	Gray shale, indistinctly laminated. Limestone fragments present larger than 1 mm., breaks down easily on weathering.
B - 1	Tan loess, highly siliceous, slakes readily in water.
B - 2	Buff-colored clay, strong and tough, breaks with conchoidal fracture, practically non-slaking.
B - 4	Very light gray clay, strong and tough, breaks with conchoidal fracture, practically non-slaking.
B - 6	Brown shale, irregular fracture, slakes in water.

TABLE II (Cont.)

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B - 7	Dark gray shale, very thin laminations, slate-like in appearance, brittle and practically non-slaking in water.
C - 1	Hard gray shale, laminations indistinct, will slake.
C - 2	Hard gray shale, laminated, will slake.
C - 3	Hard gray shale, somewhat laminated, slakes easily.
C - 4	Hard gray shale, laminations indistinct, will slake somewhat.
C - 5	Light brown laminated shale, will slake in water
D - 1	Brownish gray shale, irregular laminations, will slake

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## RAW MATERIAL STUDIES

## CHEMICAL ANALYSIS

The advantage of using a chemical analysis to determine the amount of fluxing impurities and their effect on the clay body has been a subject of discussion by many authors over a long period of years. Regardless of the arguments concerning the merits of the chemical analysis of a clay, there are many factors which are independent of the chemical constituents which affect the green and firing properties of a clay. Among these factors are the type of accessory silicate minerals; the grain size and dissimination of limestone, calcite, and dolomite; the grain size and dissimination of iron bearing minerals and gypsum; whether sulphur is present as a sulphate (which would "scum" the fired ware) or pyrite; amount of free silica, whether iron is present as the powerful ferrous flux; or in the ferric state; and of primary importance, the type of clay minerals and their percentage in the colloidal size range.

However, when used in conjunction with clay and accessory mineral identification studies, the chemical analysis can be of great use in determining characteristics of a clay. With the view in mind of correlating the chemical analysis with other properties of the clays under investigation, chemical analyses were determined on all samples, with the results given in Table III.

TABLE III  
CHEMICAL ANALYSES

Sample	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	Sulfur	Ig. Loss
A - 1	49.70	5.82	22.96	4.94	3.55	0.18	1.97	0.77	0.57	10.50
B - 1	76.70	3.69	10.66	1.17	1.08	0.34	1.63	0.80	0.31	4.25
B - 2	66.60	4.26	17.20	0.74	2.72	0.00	1.24	0.79	0.05	6.50
B - 4	66.00	4.26	18.00	0.74	2.39	0.00	0.89	0.80	0.56	7.00
B - 6	63.60	7.65	18.25	1.17	2.10	0.00	2.29	0.60	0.12	5.20
B - 7	63.10	7.61	16.60	1.10	2.75	0.00	1.99	0.80	0.37	6.50
C - 1	63.20	5.68	20.14	0.81	1.81	0.32	1.74	0.68	0.56	5.90
C - 2	56.80	9.93	20.00	1.10	2.53	1.59	0.35	0.70	0.56	6.60
C - 3	60.40	6.39	21.69	1.07	1.58	1.06	0.48	0.70	0.56	6.90
C - 4	58.10	6.35	24.20	1.00	1.81	1.00	0.35	0.70	0.05	7.30
C - 5	63.40	6.53	21.17	1.00	2.03	0.00	0.58	0.70	0.28	5.20
D - 1	59.60	1.81	13.59	7.46	5.07	0.00	0.48	1.00	0.05	11.90

#### PARTICLE SIZE DISTRIBUTION OF CLAYS AND SHALES

The amount of colloidal, or near-colloidal, size material in a clay has a marked effect on its properties in the green state. Clays are composed of polydisperse particle systems, and a considerable

percentage of very plastic clays lie within the colloidal size range of from 0.5 micron \* down to 10 - 20 millimicrons. When mixed with water in sufficient amount to cause suspension, as in a clay slip, colloidal clay acts as a lyophilic sol, and the chemistry of colloids enters the picture as a control agent.

The material in a clay of colloidal size presents a tremendous surface area to be wetted by water as the clay is made plastic. As the measure of the water-holding capacity of a clay is closely allied to the plasticity determination by the Atterberg method, the amount of surface area, or colloidal material, will influence the plasticity of a clay. The chemistry of colloids, as applied to clays, is still in its infancy and much work is being done throughout the country to correlate the effects of base exchange, adsorption, viscosity, and other properties of the clay colloids. This paper will only try to determine the influence of grain size distribution, amount of colloidal material, and type of clay mineral, and its effect on the property of plasticity, as determined by the Atterberg method, and the effect of the clays when used in mortar mix.

The non-plastic materials in clays are detrimental to the plasticity and their particle size rarely invades the colloidal range. The nature of the clay mineral, percentage of the clay mineral of colloidal size, amount and grain size of non-plastic minerals present, amount of water added, amount and type of electrolytes present, and many other factors will influence the plasticity, or workability, of a clay.

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\* Many experts regard the upper limit of colloidal particles to be 2 microns.

## SCREEN ANALYSIS

The method used to determine the amount of coarse-sized material was the screen analysis. A preliminary examination was made on each clay or shale to determine the ease of slaking and the coarseness of the largest grains present. One hundred grams of sample, dried at 110° C., was placed in a closed jar, along with about 200 cc. of water and 10 cc. of sodium pyrophosphate, which acts as a dispersant. After standing for 24 hours, if the clay was not entirely slaked, the jar was tumbled end over end for a period of 24 hours.

The sieve series was nested, the coarsest opening sieve, as determined by the preliminary examination, at the top, and progressively smaller sieves below, to the 325 mesh (U. S. Sieve Series) sieve at the bottom. The slaked clay sample was then poured on the top sieve of the series and washed with a small jet of water. When all the finer grains had passed through the top sieve, it was removed and the jet of water applied to the next finer sieve. The process was repeated until all the portion on each of the sieves had been thoroughly washed.

The material on each sieve was then dried, weighed, and the cumulative percentage, by weight, on successively smaller sieves determined. All the material remaining on the sieves was of the heavy mineral, or accessory, type associated with clays.

## PARTICLE SIZE DETERMINATION

The sedimentation method was chosen for the determination of particle sizes from 20 microns to the upper limit of the colloidal range, 0.5 micron. Stokes' law was used in computing the settling velocities of the particles, and as this law confines itself to

spheres, while clays and the accessory minerals are of irregular shape, the results are not truly representative. However, of all the methods of particle size determination, the sedimentation method with samples withdrawn by a pipette is as accurate, and gives reproducible results, as any. The pipette method for particle size determination introduced by Andreason, and described by Loomis (38) was essentially followed in this investigation.

The Andreason pipette consists of a glass vessel about 6 cm in diameter, with a capacity of approximately 600 cc.. A 10 cc. pipette, mounted in a ground-glass stopper which fits into the mouth of the vessel, extends 20 cm. below the surface of the suspension. The pipette is equipped with a three-way stop cock and spout for drainage.

The clay sample was crushed to pass a No. 20 sieve, and then ground in a mortar and pestal to insure complete grinding, and approximately 5 grams was placed in a wide mouth bottle along with 10 cc. of sodium pyrophosphate solution (35 gm./L.) and 200 cc. of distilled water. The bottle was stoppered and then rotated end over end in a tumble mill for 24 hours to agitate and disperse the material.

The dispersed sample was transferred to the sedimentation vessel and distilled water added to the upper mark on the scale. The vessel was placed in a constant temperature bath (30° C.) for one hour, and then removed, shaken by hand for several minutes and quickly replaced in the bath. Samples of 10 cc. were withdrawn at time intervals to give maximum grain size of 20, 10, 5, 2, and 1 and 0.5 microns, ac-

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(38) Loomis, G. A. Grain size of whiteware clays as determined by the Andreason pipette. *Journal American Ceramic Society*, Vol. 21, No. 11, pp. 393 - 399 (1938)

ording to Stokes' law. The samples were collected in weighed crucibles, evaporated to dryness at 110° C., and after a correction was made for the electrolyte present, the percentage by weight of the original sample determined. These percentages represent the amount of material finer than largest particle size as calculated from Stokes' law.

The cumulative percentage of each grain size was determined, and these values combined with the cumulative percentages as determined by the screen analysis. The grain size distribution, from 1 millimeter to 0.5 micron (0.0005 mm.), is shown in the graphs of Figure 1, 2, and 3. Tabular results are given in Table IV.

The Cherryville shale, sample A-1, contains more coarse grained material than any of the other samples, having 8 per cent larger than 1 mm. and 23.7 per cent larger than 20 microns (.020mm.) The loess, sample B-1, has small percentages in both the coarse and colloidal sizes, as may be expected from a wind deposited sediment. Over 80 per cent of the loess is contained between the sizes of .044 to 0.005 mm..

The curves of the Porters Creek clays, samples B-2 and B-4, show relatively good grain size distribution throughout all size ranges from .044 to .0005 mm..

The Chester shale, sample B-7, contains 4.1 per cent material larger than 0.044 mm. and 21.9 per cent within the colloidal range of .0005 mm or less.

Practically all of the remaining shales show moderate to large percentages of material between .044 and .010 mm. with the colloidal material content 14.2 per cent or less.



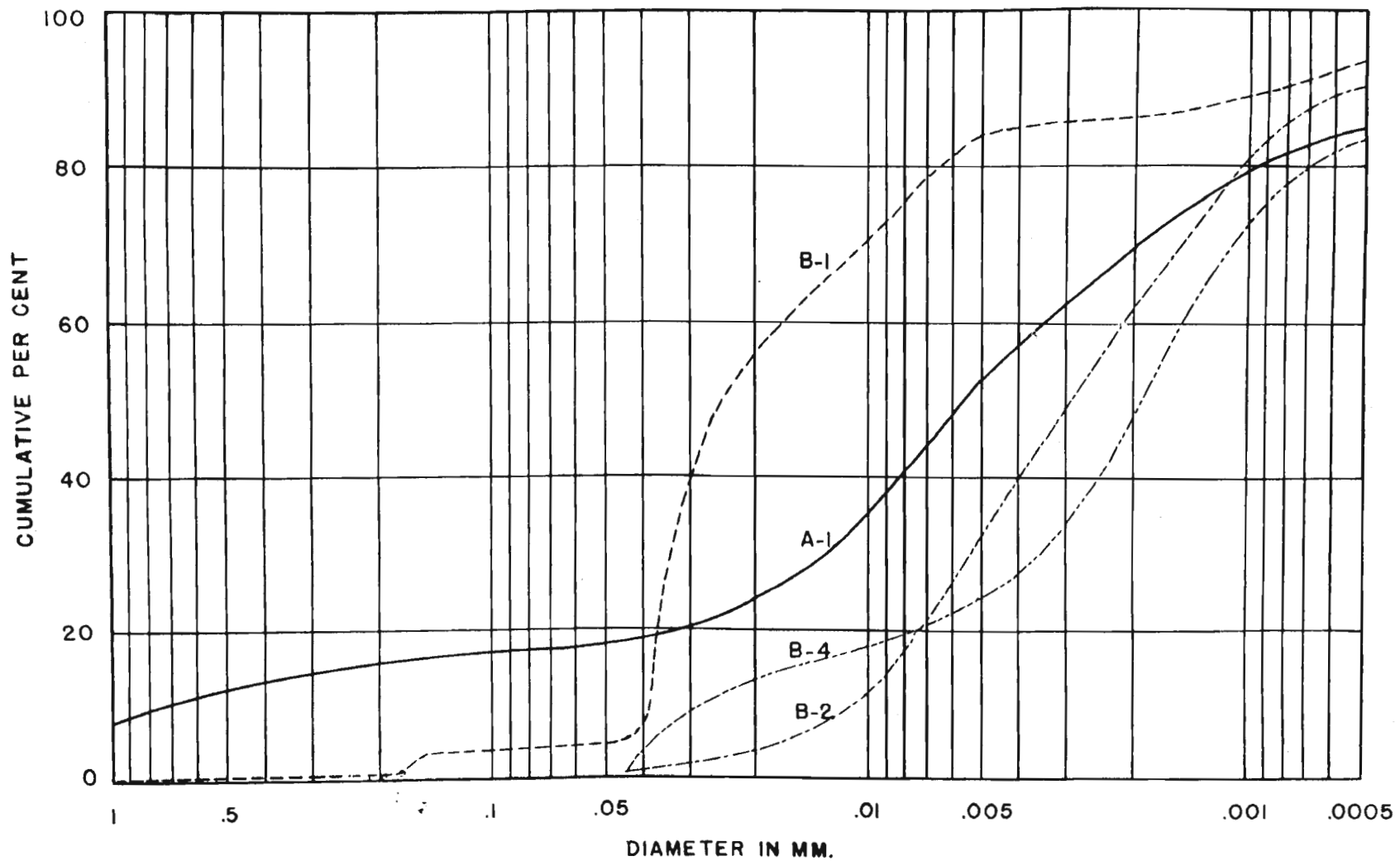


Fig. 1. Graphs showing grain size distribution in cumulative percentage for shale A-1, loess B-1, and clays B-2 & B-4

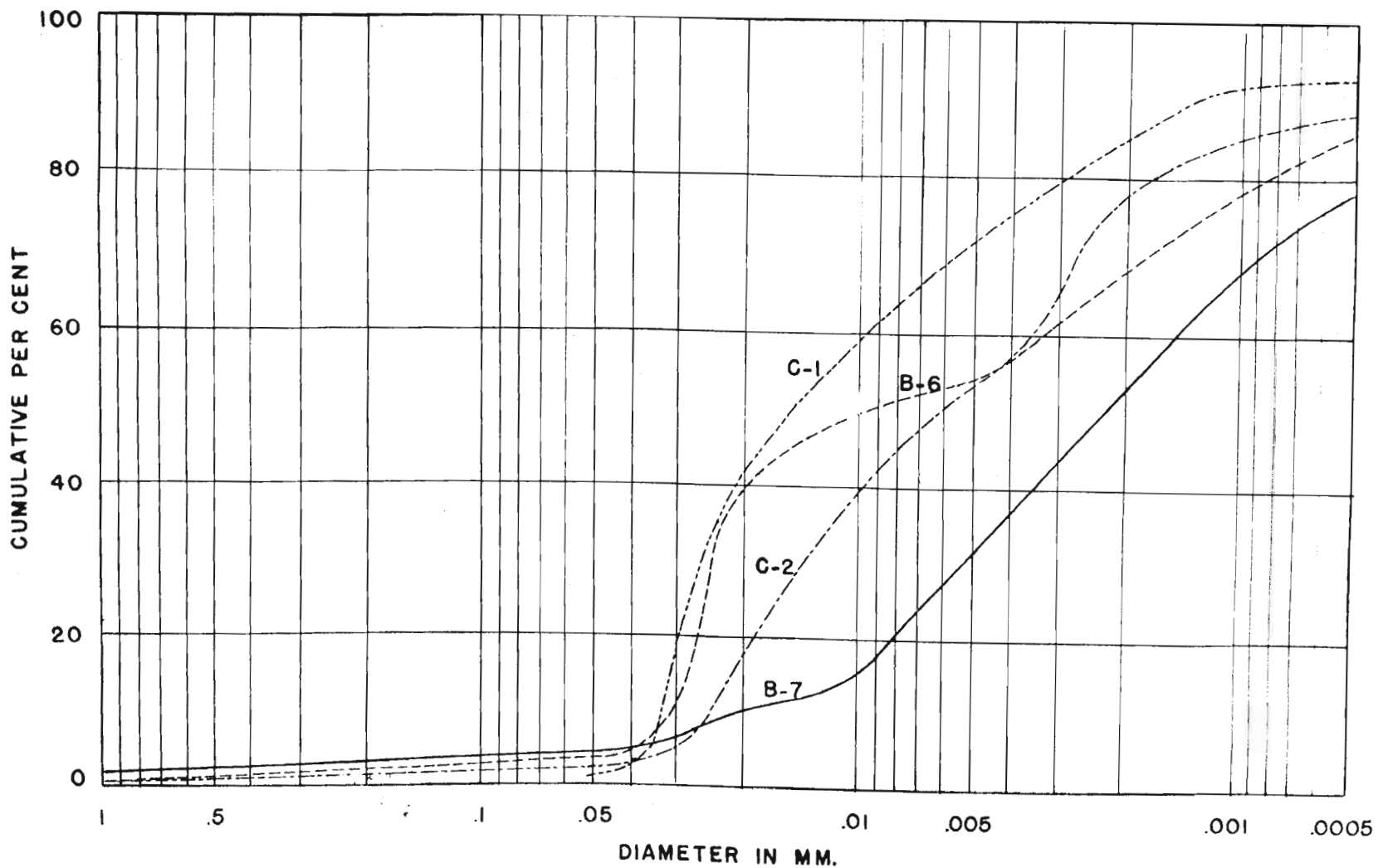


Fig. 2. Graphs showing grain size distribution in cumulative percentage for shales B-6, B-7, C-1 & C-2

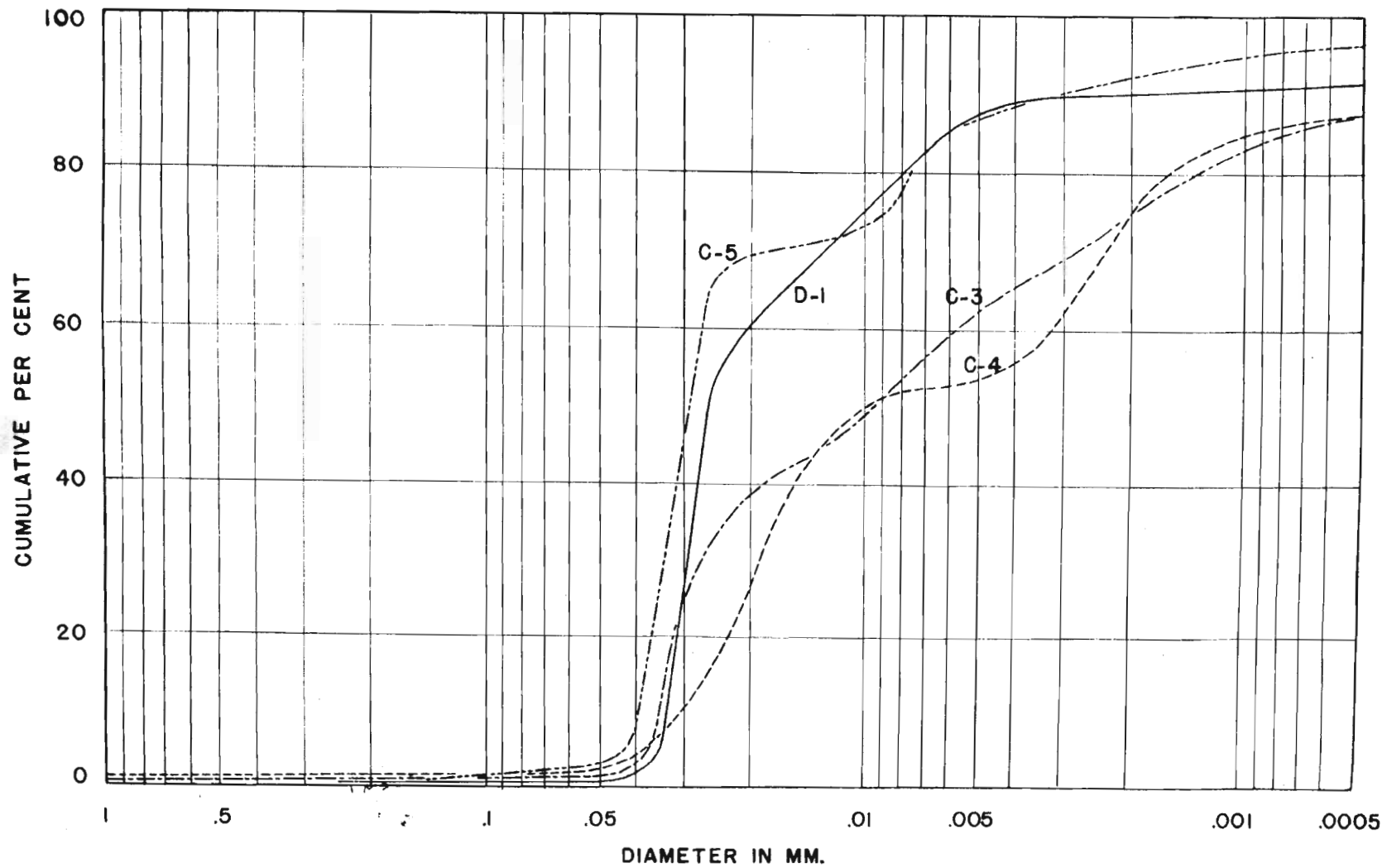


Fig. 3. Graphs showing grain size distribution in cumulative percentage for shales C-3, C-4, C-5 & D-1

TABLE IV

Grain Size Distribution of Samples, Cumulative Per Cent by Weight

Sample	Grain Size in MM.																			
	1.00	.71	.50	.35	.25	.177	.149	.105	.088	.074	.062	.053	.044	.020	.010	.005	.002	.001	.0005	
A - 1	8.00	10.2	12.7	14.0	15.1	15.6	15.9	16.2	16.4	16.5	16.5	16.6	16.7	23.7	35.2	52.3	69.4	79.3	84.7	
B - 1	0.11	0.20	0.31	0.41	0.62	1.62	3.45	3.85	4.03	4.2	4.3	4.6	5.7	55.8	70.2	81.9	86.2	89.2	93.7	
B - 2													0.64	3.8	11.6	32.2	61.6	80.4	90.5	
B - 4													0.30	13.2	17.4	24.3	47.1	72.8	83.7	
B - 6	0.65	1.06	1.35	1.67	1.9	2.16	2.4	2.70	2.95	3.1	3.2	3.4	3.7	40.1	49.4	54.1	68.4	78.1	85.8	
B - 7	1.84	2.21	2.52	2.75	2.96	3.1	3.3	3.4	3.5	3.55	3.6	3.7	4.1	10.1	15.0	32.1	53.2	68.0	78.1	
C - 1													1.0	1.7	41.8	58.8	72.1	84.8	91.8	92.4
C - 2	0.70						1.9	2.1	2.2	2.5	2.7	18.1	39.5	53.3	78.2	85.1	88.3			
C - 3	0.41						1.0	1.1	1.1	1.5	2.1	38.4	49.1	62.8	75.1	83.2	87.6			
C - 4	0.89						1.4	1.6	1.7	2.2	2.9	27.1	49.8	53.3	74.4	84.8	87.8			
C - 5							1.1	1.2	1.4	1.5	1.8	2.6	4.3	69.4	73.2	87.1	92.4	94.9	96.5	
D - 1					0.52		0.7	0.8	0.8	0.9	1.04	61.1	74.9	87.6	90.1	90.7	91.7			

## CLAY AND ACCESSORY MINERAL IDENTIFICATION

The accessory minerals associated with sediments vary greatly in quantity and type, and may be derived from the parent rock minerals or by decomposition of minerals during diagenesis and lithification. As sediments are transported, sometimes great distances, before deposition, abrasion may cause many of the softer minerals to be rounded, and in some cases the hard minerals will be abraded. This abrasion and rounding of mineral grains tends to obliterate cleavage planes, interfacial angles, and other identification features of many of the minerals.

The mineral character of the material retained on each sieve, of the screen analysis, was determined by optical methods using the petrographic and binocular microscopes. The grain size and character of the accessory minerals is of importance in determining the use of a clay or shale, and if it must be finely ground before use. As an illustration, limestone fragments of larger than 20 mesh size will calcine to  $\text{CaO}$ , and when cooled, will hydrate with such expansive force to spall or break the fired ware. However, if the limestone is in a finely divided state, it will act as a flux and enter the fused melt during vitrification. Likewise, iron bearing minerals will appear as dark spots on the fired ware, if of size larger than 1 mm., but finely divided and disseminated iron oxides are powerful coloring agents, good fluxes, and aid as a bonding component in the plastic working properties.

The accessory minerals were identified by immersion of the sieved material, after crushing if necessary, in oils of known refractive index, color, sign of elongation, extinction angle,

pleochroism, interference figure, cleavage, and crystallographic habit. A hand-magnet was used to determine any magnetic properties. The physical and optical properties of the minerals were found in texts by Rogers and Kerr,<sup>(39)</sup> Krumbein and Pettijohn,<sup>(40)</sup> and Larsen and Berman.<sup>(41)</sup>

The differential thermal method of analyzing the clay minerals was conducted with apparatus as described by Berkelhamer.<sup>(42)</sup> The sample to be tested was ground through 100 mesh, dried for 15 to 24 hours at 65° C., and placed in the sample block, packing it as dense as possible. Thermally inert alumina was placed in an adjacent hole in the sample block, and the block was heated at a constant rate to approximately 1000° C.. A differential thermocouple measured the temperature difference between the sample and alumina, and recordings were taken at 10° C. intervals of furnace temperature. A plot of the differential temperature against furnace temperature gave characteristic exothermic and endothermic peaks on the curve which indicate the dominant minerals present.

The clay and accessory minerals present in the clays and shales covered by this paper are recorded in Tables V to XVI inclusive.

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(39) Rogers, A. F. and Kerr, P. F. *Optical mineralogy*. New York, McGraw-Hill, 1942. pp. 175-376.

(40) Krumbein, W. C., and Pettijohn, F. J. *Manual of sedimentary petrography*. Appleton-Century, 1938. pp. 366-464.

(41) Larsen, E. S., and Berman, Harry. *The microscopic determination of the non-opaque minerals*. U. S. Geol. Survey. Bull. 848, 1934.

(42) Berkelhamer, op. cit.

TABLE V - Mineral Constituents of Cherryville Shale, Sample A-1

Screen No.	Quartz	Chalcedony	Limestone & Calcite	Dolomite	Orthoclase	Pyrite	Magnetite	Limonite	Gypsum	Hornblende	Olivine	Apatite	Titanite	Zircon	Remarks
18	X	X	X		X	X				X					Granite fragments on 18 mesh composed of orthoclase, hornblende and quartz
25	X		X			X									
35	X		X			X									
45	X		X			X	X	X		X					Pyrite associated with quartz in size larger than 45 mesh
60	X		X			X	X	X	X	X					
80	X		X			X	X	X	X	X	X				
100	X		X		X	X	X	X	X	X	X				
140	X		X	X	X			X			X	X			
170	X		X	X				X			X	X	X		
200	X		X	X				X			X	X	X		
230	X		X	X				X			X		X		
270	X		X	X				X			X		X		
325	X		X	X				X			X		X	X	

Clay Mineral: Illite, with lesser montmorillonite

TABLE VI - Mineral Constituents of Loess, Sample B-1

Screen No.	Quartz	Chert	Orthoclase	Microcline	Limonite	Magnetite	Hematite	Biotite	Hornblende	Zircon	Remarks
18	X		X	X	X	X					
25	X		X	X	X	X	X				
35	X	X	X	X	X	X	X				Granite fragments in size larger than 35 mesh composed of orthoclase, microcline and quartz
45	X	X	X		X	X	X				
60	X	X	X		X	X	X				
80	X		X		X	X	X				
100	X		X		X		X				
140	X		X		X		X				
170	X		X		X		X	X			
200	X		X		X		X	X			
230	X		X		X		X	X			
270	X		X		X		X	X		X	
325	X		X		X		X		X	X	

Clay Mineral: Illite



TABLE VII - Mineral Constituents of Porters Creek Clay, Sample B-2

Screen No.	Quartz	Orthoclase	Microcline	Limonite	Magnetite	Gypsum	Muscovite (sericite)	Biotite	Pyrite	Apatite	Zircon	Remarks
325	X	X	X	X	X	X	X	X	X	X	X	A few grains of pink orthoclase of 10 mesh size are present
	Clay Mineral: Essentially Illite, with a small quantity of kaolinite											
	The minuteness of coarse material makes it impracticable to run screen analysis with any accuracy											

TABLE VIII - Mineral Constituents of Porters Creek Clay, Sample B-4

Screen No.	Quartz	Orthoclase	Limonite	Magnetite	Pyrite	Muscovite (sericite)	Biotite	Gypsum	Apatite	Remarks
325	X	X	X	X	X	X	X	X	X	The minuteness of coarse material makes it impracticable to run screen analysis with any accuracy
	Clay Mineral: Illite									

TABLE IX - Mineral Constituents of Maquoketa Shale, Sample B-6

Screen No.	Quartz	Hematite	Limonite	Magnetite	Pyrite	Limestone & Calcite	Dolomite	Aragonite	Orthoclase	Gypsum	Apatite	Zircon	Remarks
18	X	X	X		X	X	X	X					Limestone composed of calcite, dolomite, and aragonite
25	X	X	X			X			X				
35	X	X	X			X			X				Quartz grains sparse in size larger than 80 mesh; abundant below 140 mesh
45	X	X	X	X		X			X				
60	X	X	X	X		X			X				
80	X	X	X	X		X			X				
100	X	X	X	X		X			X				
140	X	X	X	X		X			X	X		X	
170	X	X	X	X		X			X	X	X	X	
200	X	X	X	X		X			X	X	X	X	
230	X	X	X	X		X			X	X	X	X	
270	X	X	X	X		X			X	X	X	X	
325	X	X	X	X		X			X	X	X	X	

Clay Mineral: Illite

TABLE X - Mineral Constituents of Chester Shale, Sample B-7

Screen No.	Quartz	Limestone & Calcite	Dolomite	Orthoclase	Limonite	Hematite	Magnetite	Aragonite	Gypsum	Siderite	Zircon	Remarks
18	X	X	X		X							The limestone is composed of calcite, dolomite, and amorphous calcium carbonate
25	X	X		X	X							
35	X	X		X	X							
45	X	X		X	X	X						The quartz grains are concentrated largely in sizes less than 45 mesh
60	X	X		X	X	X	X					
80	X	X				X	X	X				
100	X	X				X	X	X				
140	X	X				X	X	X	X			
170	X	X				X	X	X	X	X		
200	X	X				X	X	X	X	X		
230	X	X				X		X	X	X		
270	X	X				X			X	X		
325	X	X				X			X	X	X	

Clay Mineral: Essentially illite with appreciable montmorillonite

TABLE XI - Mineral Constituents of lower Pleasanton Shale, Sample C-1

Screen No.	Quartz	Calcite	Aragonite	Orthoclase	Microcline	Oligoclase	Limonite	Hematite	Pyrite	Siderite	Muscovite	Biotite	Gypsum	Chlorite	Glauconite	Zircon	Remarks
270	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	A more complete screening impossible because of small quantity of sample
325	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
Clay Mineral:		Illite															

TABLE XII - Mineral Constituents of upper Pleasanton Shale, Sample C-2

Screen No.	Quartz	Limonite	Hematite	Pyrite	Siderite	Orthoclase	Limestone & Calcite	Dolomite	Chlorite	Remarks
18		X								
60	X	X	X	X		X	X			
100	X	X	X	X		X	X			
170	X	X	X	X		X	X	X		

TABLE XII (Concluded)

Screen No.	Quartz	Limnite	Hematite	Pyrite	Siderite	Orthoclase	Limestone & Calcite	Dolomite	Chlorite	Muscovite	Biotite	Microcline	Apatite	Remarks
230	X	X	X			X	X	X	X					
270	X	X	X		X	X	X	X	X	X	X	X	X	
325	X	X			X	X	X	X	X	X	X	X	X	

Clay Mineral: Illite and some Montmorillonite

TABLE XIII - Mineral Constituents of Weston Shale, Sample C-3

Screen No.	Quartz	Limestone & Calcite	Limonite	Pyrite	Siderite	Orthoclase	Microcline	Albite	Dolomite	Muscovite	Biotite	Zircon	Chlorite	Remarks
18		X												Limestone composed of calcite and dolomite
60	X	X	X	X		X	X							
100	X	X	X	X	X	X			X			X		Zircon present as inclusions
170	X	X	X	X	X	X			X	X		X		
230	X	X	X		X	X		X	X	X	X	X		Zircon freed
270	X	X			X			X	X	X	X	X	X	
325	X	X			X			X	X	X	X	X	X	

Clay Mineral: Illite, with some montmorillonite

TABLE XIV - Mineral Constituents of Lawrence Shale, Sample C-4

Screen No.	Quartz	Limestone & Calcite	Dolomite	Limonite	Siderite	Muscovite	Biotite	Talc	Chlorite	Glauconite	Zircon	Remarks
18	X	X	X		X	X	X	X		X	X	Limestone composed of calcite, dolomite, quartz, glauconite, siderite, zircon, talc, biotite, and muscovite
60	X	X		X								
100	X	X		X								
170	X	X		X				X				
230	X	X	X	X	X	X		X				
270	X	X	X	X	X	X	X	X				
325	X	X	X	X	X	X	X	X	X			

Clay Mineral: Illite with montmorillonite

TABLE XV - Mineral Constituents of Lagonda Shale, Sample C-5

Screen No.	Quartz	Calcite	Dolomite	Limonite	Magnetite	Pyrite	Orthoclase	Muscovite	Biotite	Gypsum	Chlorite	Apatite	Zircon	Remarks
100	X	X	X	X	X									
140	X	X	X	X	X	X								
170	X	X	X	X	X			X					X	Zircon present as inclusions in quartz
200	X	X	X	X	X		X	X	X				X	
230	X	X	X	X	X		X	X	X	X	X		X	
270	X	X	X	X			X	X	X	X	X		X	
325	X	X	X	X			X	X	X	X		X	X	

Clay Mineral: Unidentifiable by the differential thermal analysis method



TABLE XVI - Mineral Constituents of Northview Shale, Sample D-1

Screen No.	Quartz	Limonite	Hematite	Calcite	Dolomite	Orthoclase	Zircon	Remarks
60	X	X	X	X				
100	X	X	X	X	X			
170	X	X	X	X	X		X	Zircon present as inclusions in quartz
230	X	X	X	X	X		X	
270	X	X	X	X	X	X	X	
325	X	X	X	X	X	X	X	

Clay Mineral: Unidentifiable because reactions masked by calcite, dolomite and other minerals

## DISCUSSION OF CLAY AND SHALE MINERAL CONSTITUENTS

The Cherryville shale, sample A-1, would need fine grinding to disseminate the limestone and pyrite present in sizes larger than 1 mm.. Any grinding of raw clay introduces additional cost into ceramic heavy-clay production, and for this reason the presence of these large size detrimental accessory minerals may prohibit the use of this clay for structural clay products.

The Maquoketa shale, sample B-6, contains limestone and iron ore minerals in size larger than 45 mesh which must be disseminated, by some method, throughout the clay. Dry pan grinding of the material would probably give satisfactory distribution of these minerals, as they appear to be fairly loosely aggregated.

The Chester shale, sample B-7, would need grinding to both grind and scatter the limestone and limonite, which are present in large grain sizes, and to crush the "slate-like" laminated sheets of shale. The shale is practically non-slaking in water, and grinding is necessary to produce plasticity.

The upper Pleasanton shale, sample C-2, would also need grinding because of the presence of limonite in large size. Dry panning would probably complete the grinding process satisfactorily. The Lawrence shale, sample C-4, should also be fed into the dry pan before mixing with water, for the same reason.

The other clays and shales need no special grinding preparation before use if the extrusion process is used for manufacture. However, if brick is to be made by the dry press process, grinding, and later addition of water, should be accomplished in the dry pan.

Accessory minerals, other than iron ore minerals and limestone, are not present in sufficient quantity to cause any detrimental effect in the fired clay product. The firing behavior examination of the clays, which is discussed later, will show if the limestone and iron oxides interfere with the good firing of the clays.

All the shales contained illite as the clay mineral, and some of the shales had additional amounts of montmorillonite, as evidenced by the differential thermal analysis identification method. This is as would be expected, for the laminations of shale, and therefore the classification of the clay as a shale, is believed largely due to parallel orientation and diagenesis of minerals of the illite group. The formation of illite is best accomplished in marine or brackish waters, where potash is not being removed significantly,<sup>(43)</sup> as clays have higher adsorptive powers for potassium than any other metallic ion. According to Keller,<sup>(44)</sup> if illite is being formed in an environment in which potash is in excess, the particles will orient themselves giving rise to the laminated structure, and upon lithification it will be a shale.

Illite also proved to be the dominant mineral present in the Porters Creek clay samples, although all previous investigators have indicated the clay mineral to be montmorillonite. X-ray and petrographic analysis of Missouri Porters Creek clay indicate it to be composed of quartz, montmorillonite, with small amounts of kaolinite.\*

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(43) Ross, C. S. Clays, and soils in relation to geologic processes. Jour. Wash. Acad. Sci. Vol. 33, p. 231 (1943)

(44) Keller, W. D. Evidence of texture on the origin of the Cheltenham fireclay of Missouri and associated shales. Jour. Sed. Petrology. Vol. 16, No. 2, p. 70 (1946)

Further investigation into the clay minerals of the Porters Creek clay is not within the scope of this paper, but the possibility that illite is present in the clay which has always previously been considered to be composed of montmorillonite cannot be ruled out entirely. Ross<sup>(45)</sup> states "the gradual inversion of montmorillonite to bravaisite or mica-like minerals ("illite") seems to offer an explanation of this preferential fixation of potassium and the dominance of potassium minerals in marine deposits." Perhaps the original montmorillonite of the Porters Creek clay has been altered to minerals of the illite group in localized areas, from which the samples were gathered.

#### PLASTICITY INDEX AND DRYING PROPERTIES

Practically all clays show a decrease in size upon drying, which may be attributed to the loss of mechanical water, which fills the pore spaces between the particles holding them apart. As the water evaporates from the plastic clay mass, the particles are drawn closer together by surface tension, until sufficient particles are in contact to stop all further shrinkage. The finer grained clays would have greater shrinkage than the clays of coarse grain size, because more water is necessary, in the fine grained types, to wet the surfaces of the larger quantity of fine particles having greater surface area. Additions of sand, or other non-plastics, will serve to reduce the shrinkage of a "fat" clay, while additions of plastic clay to "lean" clays will increase the plasticity and shrinkage. The plasticity and green strength of a clay will largely control the proposed use

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\* Conversation with Dr. O. R. Grawe and Dr. G. A. Muilenburg

(45) Ross, C. S. op. cit.

for various types of clay products. Excessive shrinkage, caused by clays of great plasticity, will tend to cause warping and cracking of the dry product, and size will be difficult to control.

#### PLASTICITY INDEX

All tests used in the determination of the plasticity index were carried out as described by A. S. T. M. Specifications D423-39<sup>(46)</sup> and D424-39,<sup>(47)</sup> which cover the "liquid" and "plastic limits" of soils and clays.

The mechanical device for testing the liquid limit consists essentially of a brass cup, suspended on a carriage, which is dropped through a distance of 1 cm. upon a hard rubber base by turning a crank through one revolution. The plastic sample is placed in the bottom of the cup, smoothed off to a depth of 1 cm., and grooved by a special tool to an angle of 60°.

"For the liquid limit determination, the sample is ground to pass a No. 40 sieve, and the sieved material is thoroughly mixed with distilled water in an evaporating dish until the mass assumes a putty-like consistency. A portion of this sample is then transferred to the cup of the liquid limit device, leveled off to a depth of 1 cm., and divided by means of the grooving tool. The cup is then dropped onto the rubber base two times per second until the two sides of the sample come into contact along the bottom of the groove along a distance of about one-half inch. The number of shocks are recorded and the moisture content of the sample determined on a dry basis.

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(46) A. S. T. M. Standards, Part II. Standard method of test for liquid limit of soils. pp. 626-628 (1944)

(47) A. S. T. M. Standards, Part II. Standard method of test for plastic limit and plasticity index. pp. 629-630 (1944)

The above operation is repeated for two additional portions of the sample to which sufficient water has been added to bring the clay to a more fluid condition requiring less than 25 shocks for sample contact.

A flow curve is plotted on semilogarithmic graph paper, with moisture contents on the arithmetical scale and the number of shocks on the logarithmic scale. The moisture content corresponding to the intersection of the flow curve with the 25 shock ordinate shall be taken as the liquid limit of the clay."

"For the plastic limit determination the ground sample (40 mesh) is placed in an evaporating dish and mixed with distilled water until the mass becomes plastic enough to be shaped into a ball. The ball of clay is then rolled between the palm of the hand and a ground-glass plate with just sufficient pressure to form the plastic mass into a thread. When the diameter of the thread becomes 1/8 inch the soil shall be kneaded together and again rolled out. This process is continued until the crumbling of the clay prevents the formation of the thread. The plastic limit of the clay is the moisture content, expressed as a percentage of the weight of the oven dried clay, of this crumbled sample.

The plasticity index of a soil, or clay, is the difference between its liquid limit and its plastic limit:

$$\text{Plasticity Index} = \text{Liquid limit} - \text{plastic limit}''$$

Results of plasticity measurement as determined by the plasticity index method are reproducible and easily obtained. Data for the plasticity index of each sample is listed in Table XVII.

PREPARATION OF TEST PIECES AND DRYING SHRINKAGE<sup>(48)</sup>

The raw clay, or shale, was thoroughly dried at about 70° C., passed through a jaw crusher, and the crushed material ground in a hammer mill to pass a No. 20 sieve. The ground material was then fed into a Simson Mixer, and water slowly added, until stiff mud consistency was obtained.

The plastic mass was forced into a metal mold, 1 1/8 x 1 1/8 x 12 inches in size, with the aid of a wooden plunger. The excess was struck off with a spatula and the surface smoothed. Test pieces two inches in length were then cut from the molded plastic bar of clay. The pieces were numbered for identification and placed on a smooth wooden pallet.

The plastic pieces were weighed to the nearest 0.1 gram. The plastic volume was determined immediately on each piece by the suspended weight method. Kerosene, with a specific gravity of 0.800 was used as the measuring fluid. The test pieces were lightly dried with a soft cloth to remove the kerosene film, and allowed to dry at room temperature. The test pieces were then dried at a temperature of approximately 70° C. for five hours, and then at 110° C. until constant weight was obtained, then allowed to cool in a dessicator.

The dry test pieces were weighed to the nearest 0.1 gram, and then soaked in kerosene for at least 12 hours, after which the volume of each was determined by the suspended weight method.

From the data obtained by the above tests, calculations were made to determine the linear shrinkage and volume shrinkage on drying, and the water of plasticity. The results of these determinations are listed in Table XVII.

TABLE XVII - Plasticity Index, Water of Plasticity, and Drying Shrinkage

Sample No.	Liquid Limit	Plastic Limit	Plasticity Index	Volume Shrinkage	Linear Shrinkage	Water of Plasticity
A-1	28.4%	18.7%	9.7%	15.5%	4.9%	21.8%
B-1	28.3	19.0	9.3	9.3	3.0	20.6
B-2	104.5	76.6	27.9	46.1	13.5	61.5
B-4	125.8	60.6	65.2	46.9	13.7	66.4
B-6	32.6	23.0	9.6	18.5	6.1	24.0
B-7	39.3	26.8	12.5	29.8	9.1	25.6
C-1	30.0	23.3	6.7	13.8	4.4	24.0
C-2	37.0	23.1	13.9	18.5	5.8	23.2
C-3	32.6	23.7	8.9	16.8	5.3	23.6
C-4	35.6	24.2	11.4	20.7	6.5	23.7
C-5	30.3	23.8	6.5	13.7	4.4	22.2
D-1	27.0	19.8	7.2	13.3	4.3	21.3

## DISCUSSION OF PLASTIC AND DRYING PROPERTIES

A comparison of the plasticity indices with the amount of material within the colloidal size range, for the clays as a whole, reveal that no definite relationship results between colloidal material present and plasticity. Likewise, a comparison of the amount of material finer than 5 and 10 microns, which would probably contribute somewhat to the plasticity of a clay, gives no proportional relationship with

(48) Journal American Ceramic Society. Standards report. Preparation of clay trials. Vol. 11, pp. 445, 449-451 (1928)



the plasticity index. This may be explained by the fact that variables enter the picture in the form of coarse-grained non-plastics, non-plastics of colloidal size, and the amount and type of clay mineral present. Also, the plasticity of a clay is not only a measurement of the water it holds at certain stages of plasticity, but rather a combination of several properties, of which the water-absorbing capacity is but one part. The plasticity index method of plasticity determination of a clay indicates a relative measurement by the difference in water content between two plastic conditions, and when used in this relative sense is satisfactory in the comparison of two or three clays.

It is interesting to note that the Chester shale, sample B-7, has nearly 22 per cent material within the colloidal range of less than 0.5 micron, yet exhibits a plasticity index of only 12.5. The clay mineral is illite, with some montmorillonite, which would normally be expected to give greater plasticity than the index obtained for this shale. However, the physical consolidation of the Chester shale into thin, hard, slate-like laminated sheets makes it difficult for water to reach the individual particles even after grinding the shale through 40 mesh. Undoubtedly, finer grinding, or pulverization, of the material would produce greater plastic properties in that a greater area of clay particles would be exposed to wetting.

In the Cherryville shale, sample A-1, although some montmorillonite is present with the illite, plasticity is low, with a value of 9.7 for the plasticity index. The large quantities of coarse, non-plastic accessory minerals restrict the formation of plasticity.

The Porters Creek clays, samples B-2 and B-4, have negligible quantities of non-plastics larger than 44 microns, and exhibit

excessive plasticity even to the point of stickiness. The clay is geologically classified as a fuller's earth, and it absorbs huge amounts of water before the liquid limit is reached. On the basis of plasticity alone, it would seem that montmorillonite makes up the bulk of the clay mineral, even though the clay mineral identification showed illite to be present. Sample B-4, with 16.3 per cent colloidal material, had much greater plasticity than B-2, which had less than 10 per cent within the colloid range. Some kaolinite was indicated to be present in Sample B-2 which would replace the highly plastic clay mineral and reduce the plasticity somewhat, as kaolinite is considered to be not greatly plastic.

The grain size distribution curves for the loess, sample B-1, and Northview shale, sample D-1, are very similar and the percentages of material at respective particle sizes are nearly equal. As may be expected, the plasticity indices are closely identical, with 9.3 for the loess and 7.2 for the Northview. Although the clay mineral was unidentifiable in the latter, it follows that illite is probably present because of the allied grain size and plastic properties between it and the loess.

The relatively larger amount of material present above 20 microns in the Maquoketa shale, sample B-6, and the lower Pleasanton shale, sample C-1, and the Weston shale, sample C-3, probably accounts for the respective plasticity indices of 9.6, 6.7 and 8.9, as the dominant clay mineral in each shale is illite.

The upper Pleasanton shale, sample C-2, and the Lawrence shale, sample C-4, exhibit closely allied and similar grain size distribution curves, with minor amounts of coarse material and about 12 per cent

of colloidal material present. Each contains illite, and some montmorillonite, and resultant plasticity indices are 13.9 and 11.4 respectively.

The Lagonda shale, sample C-5, was the least plastic of all the samples, with a plasticity index of 6.5. This is not surprising, as the grain size distribution curve for Lagonda shale shows it to contain about 70 per cent material larger than 20 microns, and only 3.5 per cent material within the colloidal range.

All of the shales have good drying properties, and exhibit no cracking, checking or warping. The drying linear shrinkage varies from 4.4 to 9.1 per cent, and on the basis of the drying properties the shales are satisfactory for various types of heavy clay product production. The loess sample had the least shrinkage of all the samples tested, with a value of 3.0 per cent linear shrinkage. This low shrinkage value is largely due to the large quantity of non-plastic silica sand present in the loess.

The shrinkage of the Porters Creek clays is excessive, and some warping was evident in the dry test pieces but no cracking or checking was visible. The forming of brick, using these clays, by the extruded process would present drying difficulties and exact sizing would be hard to control. Further experimentation, using the dry press process of forming brick, with additions of non-plastic clay, sand, or grog, would undoubtedly reduce the drying shrinkage considerably and make for better and more easily controlled drying properties.

## FIRED PROPERTIES

## PYROMETRIC CONE EQUIVALENT

The standard method of test for the pyrometric cone equivalent (PCE), as specified by A. S. T. M. Specification C24-42, (49) (50) was used for all determinations.

"The sample is thoroughly mixed, then quartered and ground to pass a No. 70 sieve. The sieved sample is then calcined, in an oxidizing atmosphere, to from 925° to 980° C., for not less than 30 minutes.

The calcined sample is mixed with an organic binder, such as dextrine, and water, and the mass formed into truncated trigonal pyramids, 1 1/8 inch in height.

When dry, the test cones are mounted on a plaque of refractory material, along with Standard Pyrometric Cones. The plaque is placed in a suitable furnace and heated in an oxidizing atmosphere at a specified rate. The softening of the test cone will be indicated by the top bending over and the tip touching the plaque. The PCE is reported in terms of Standard Pyrometric Cones and is (than) Standard Cone which most nearly corresponds in time of softening with the test cone."

A Remmy Lab. Kiln, No. 2150, was used in all PCE determinations.

## BEHAVIOR IN FIRING

The method of determining the fired properties of a clay is the

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(49) op. cit. Pyrometric cone equivalent of refractory materials. pp. 294-296.

(50) op. cit. Tentative Revision, Pyrometric cone equivalent of refractory materials. pp. 1604-1605.

firing behavior, which involves the progressive changes in porosity, volume, hardness, strength, and color at regular and frequent intervals during vitrification.

In the firing behavior of each clay or shale, progressive firing properties were determined on test pieces drawn from the furnace at two cone intervals within a 20 cone range, with the upper limit of the heat treatment range being three or four cones below the PCE of the material. This long heat treatment range of 20 cones insures a complete coverage of the firing range, for practically all clays and shales, from the point of incipient vitrification to excessive heat treatment as indicated by over-firing or bloating.

The method followed in the firing behavior determinations is essentially that as listed in the Journal American Ceramic Society. (51)

Three test pieces (from the drying shrinkage determinations) of each shale or clay, were placed on each of ten refractory splits, along with standard pyrometric cones, which were selected to give an interval of two cones between each successive refractory split.

The firing was accomplished in an oil fired, rectangular down-draft kiln of approximately 30 cubic foot setting volume. Temperature control was maintained by means of a platinum-platinum-10% rhodium thermocouple, and an oxidizing atmosphere was assured by a CO<sub>2</sub> indicator. The heating was at a rate of 50° C. per hour, from the start of the firing until the heat treatment was reached corresponding to the softening point of the third consecutive cone below that at which the first trial is to be drawn, and 20° C. per hour thereafter, until

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(51) Journal American Ceramic Society. Standards report. Standard method for behavior in firing. Vol. 11, pp. 454-457 (1928)

the firing was completed.

When the standard pyrometric cone, corresponding to the lowest heat treatment desired, went down, the refractory split containing the first set of test pieces was withdrawn from the kiln. At intervals of two cones thereafter, the splits containing the test pieces were withdrawn from the kiln. Immediately after removal from the kiln, the test pieces were covered with hot sand and allowed to cool.

When cool enough to handle, the fired test pieces were placed in a dessicator until room temperature was reached. They were then carefully examined for any adhering material, cleaned and weighed.

The weighed test pieces were placed in distilled water and boiled for two hours, then cooled to room temperature while still immersed in the water. The saturated test pieces were weighed to the nearest 0.1 gram after being dried lightly with a damp cloth to remove excess water. The fired volume was determined on each test piece by the suspended method.

The apparent porosity and volume shrinkage was determined on each test piece by the following calculations:

#### Apparent Porosity

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

AP - per cent apparent porosity  
 Sf - weight of the saturated fired test piece  
 Wf - weight of the fired test piece  
 Vf - fired volume of test piece

#### Volume Fired Shrinkage

$$VS = \frac{Vd - Vf}{Vd} \times 100$$

VS - per cent volume fired shrinkage  
 Vd - dry volume  
 Vf - fired volume

The results of the firing behavior are listed in both graphic and tabular form for each of the clays under investigation in following pages. The apparent porosity (AP) and volume shrinkage (VS) are plotted against the PCE to which the test pieces were fired.

The firing behavior is the final test for determining the suitability of a clay, or a shale, for face brick, common brick, or other heavy clay products, before actual pilot plant production. The requisites of a clay, or shale, to be used for face brick manufacture are:

1. At least moderate plastic working properties to minimize loss in defective green ware.
2. Drying and firing shrinkages should not be high in order to retain shape and avoid warping.
3. The porosity should be low to insure low absorption of water and good freezing and thawing resistance when exposed to weather.
4. The color should be pleasing and attractive.
5. Vitrification should take place at a low temperature and the brick must fire to a dense, hard and strong structure. A minimum of 5 cone vitrification range is necessary, but a greater range is desirable to insure low loss in a periodic kiln where temperature control is not too accurate.

Common, or back-up, brick can be made from the same material that is used for face brick, but more impure clays are usually used for this purpose because the degree of vitrification and color are not of primary importance as these brick are seldom exposed to the weather.

Sewer pipe clays should have medium to good plasticity to give good flow characteristics; the green strength should be high; drying and firing shrinkages should be moderate to low to avoid warping; the vitrification range should be long; and the clay must contain free silica to take a good salt glaze at about 2000° F..

## RESULTS AND DISCUSSION OF FIRING BEHAVIOR DATA

## Cherryville Shale - Sample A-1

TABLE XVIII - Firing Behavior of Cherryville Shale

Pyrometric Cone Equivalent - Cone 2 (1135° C. approximately)			
Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
014	30.5%	1.1%	Buff
012	30.3	1.5	Buff
010	30.3	2.7	Reddish Buff
08	29.2	---	Red
06	14.7	---	Red
04	8.9	---	Deep Red
02	2.2	---	Deep Red

The Cherryville shale, although ground to pass 20 mesh, still contained enough calcareous material of detrimental grain size to cause breakage of the test pieces, fired to higher than cone 06, when immersed in water. The limestone present in the test pieces that were fired to cone 04, or less, evidently was not sufficiently calcined to cause absorption of water and consequent test piece breakage by expansion due to hydration. Because of the breakage, no volumes were obtainable on the trials fired to a higher heat treatment than cone 08. The hydrated calcium oxide appeared as small white spots on the surface, and within the test pieces.

As indicated by the apparent porosity curve in Fig. 4, incipient vitrification takes place at cone 014 and the shale is not overfired at cone 02. Between cones 08 and 02, there is a decrease in porosity



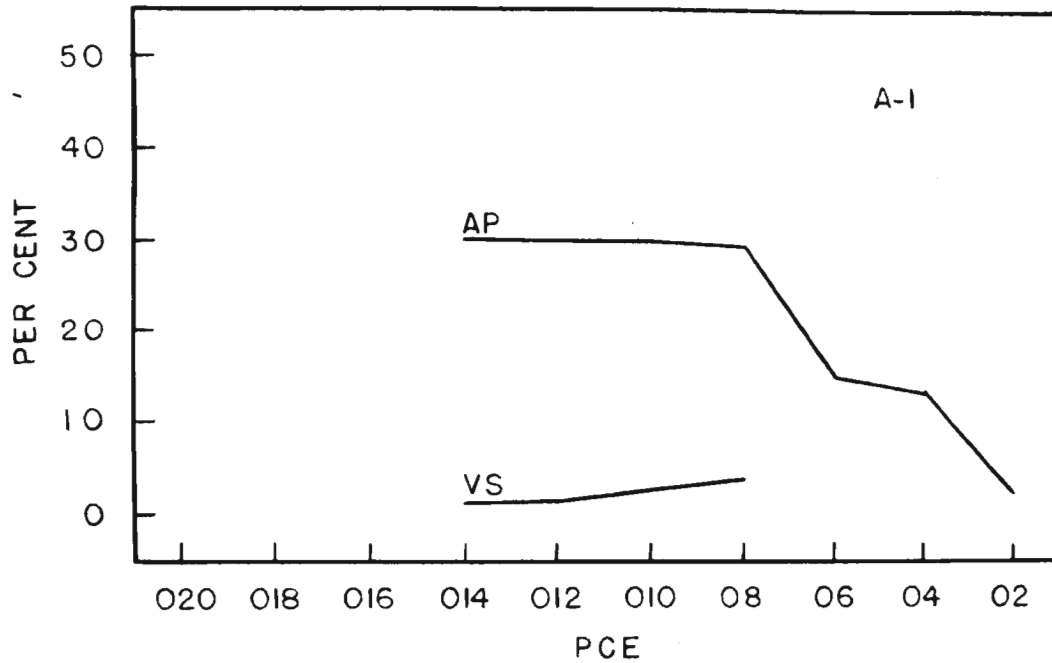


Fig. 4 Progressive changes in apparent porosity (AP) and volume shrinkage during firing of the Cherryville shale, sample A-1.

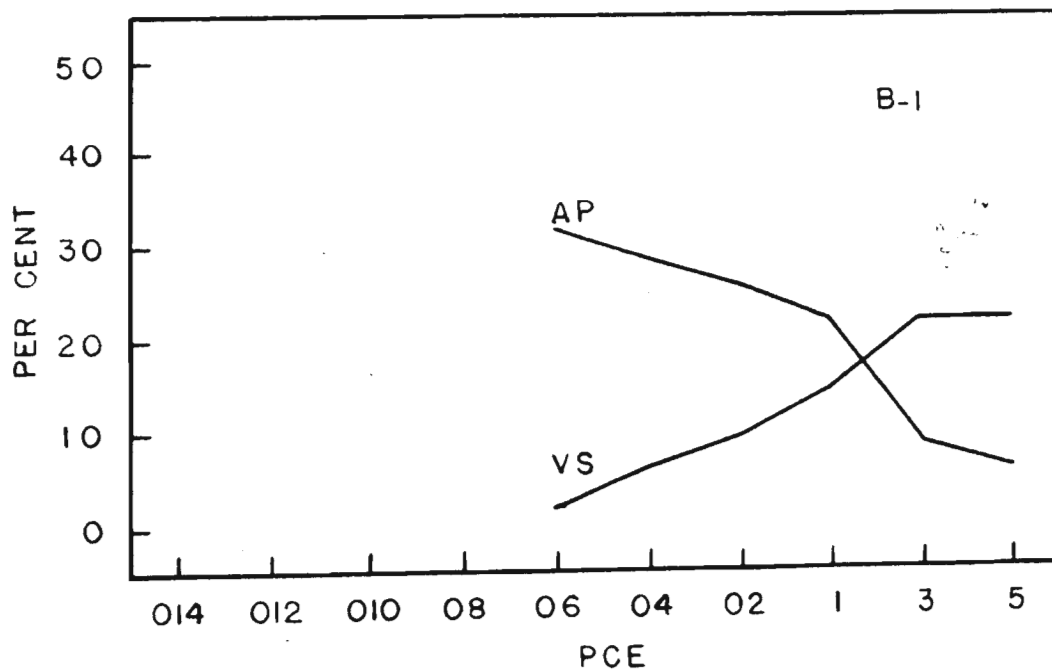


Fig. 5 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of loess, sample B-1.

from about 30 to 2 per cent, which is excessive for brick production in a 4 cone range. Because of this short firing range, and the necessity for finer grinding than 20 mesh to thoroughly disseminate the calcareous material throughout the clay to avoid hydration, the clay may be considered unsatisfactory for any heavy clay production.

Loess - Sample B-1

TABLE IXX - Firing Behavior of Loess

Pyrometric Cone Equivalent - Cone 7-8 (1210° - 1225° C. app.)			
Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
06	32.3%	1.5%	Dark Salmon
04	28.4	5.6	Reddish Brown
02	25.4	9.0	Reddish Brown
1	21.5	13.9	Dark Reddish Brown
3	8.6	21.6	Deep Red - Overfired
5	5.5	22.2	Deep Red - Overfired

The firing behavior of the loess indicates incipient vitrification at cone 06 (1005° C.), with overfiring beginning about cone 3 (1145° C.). The porosity at cone 06 is above 30 per cent, and a first quality face brick should have less than 30 per cent porosity to give good weathering properties. The firing range, based on color and porosity, of brick made of loess would be between cones 04 and 2. This would give brick having a total linear shrinkage (drying and firing) of between 4.8 and 8.0 per cent. Brick, within this range, have the hardness of steel and have greater compression strength than the required 3000 pounds per square inch. (52) The color would be the

standard brick-red and would be fairly uniform throughout the entire range.

The only difficulty in producing brick of this loess would be the relatively short firing range, making close temperature control an absolute necessity for uniform size and color.

Porters Creek Clay - Sample B-2

TABLE XX - Firing Behavior of Porters Creek Clay

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Pyrometric Cone Equivalent - Cone 9-10 (1250° - 1260° C.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
012	44.3%	9.5%	Buff
010	44.3	10.3	Buff
08	44.0	10.4	Buff
06	39.1	16.9	Buff
04	38.1	18.9	Buff
02	37.2	20.4	Buff
1	35.9	21.5	Buff
3	30.6	27.3	Dark Buff
5	26.6	30.7	Brown
7	12.7	39.1	Dark Reddish Brown

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Vitrification begins in this clay below a temperature of 840° C. and continues to near cone 5 (1180° C.), where overfiring results.

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(52) A. S. T. M. Standards, Part II. Tentative specifications for building brick. pp. 1087-1091 (1944)

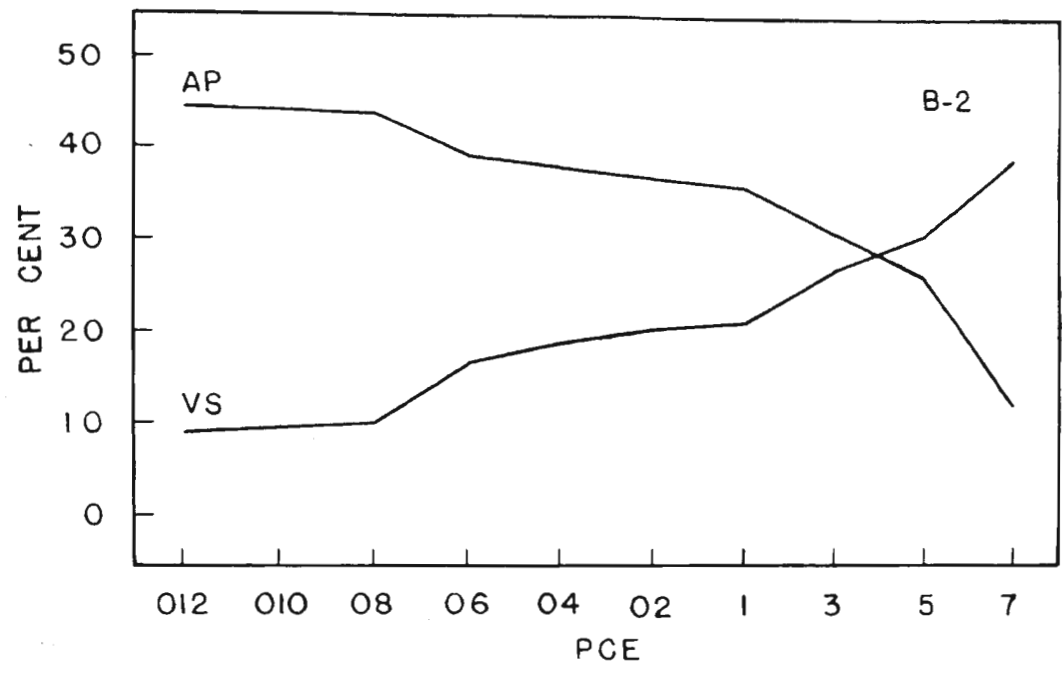


Fig. 6 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of Porters Creek Clay, sample B-2.

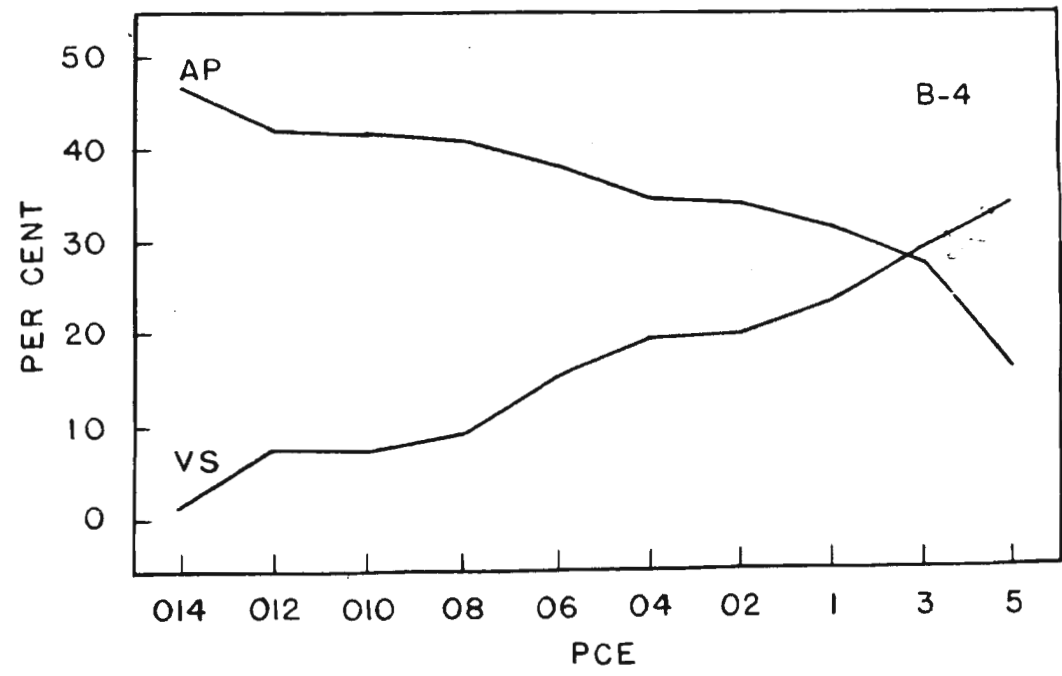


Fig. 7 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of Porters Creek Clay, sample B-4.

Shrinkage is excessive, the total linear value being 16.6 per cent at cone 012, and 25.1 per cent at cone 5. Warping was evidenced above cone 1, with excessive warpage and shrinkage at cone 5 and higher.

Because of the excessive porosity (above 35 per cent) of this clay when fired up to cone 1, it could not be used for face brick. Back-up, or common, brick could be manufactured from this clay if fired in the range from cones 012 to 1, as high porosity and accurate dimensions are of only minor importance for this type brick. However most back-up brick are obtained from lots of face brick which do not meet specifications as to size and porosity.

Because of the unusual levity of this clay, when fired to about 1800° F., it has special use in the field of light-weight concrete aggregates which will be discussed later.

Porters Creek Clay - Sample B-4

TABLE XXI - Firing Behavior of Porters Creek Clay

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Pyrometric Cone Equivalent - Cone 8 (1225° C.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
014	46.3%	1.5%	Light Buff
012	42.2	7.5	Light Buff
010	41.4	7.8	Light Buff
08	41.0	9.3	Light Buff
06	38.3	15.3	Buff
04	34.5	19.9	Buff

TABLE XXI - Firing Behavior of Porters Creek Clay (cont.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
02	34.2%	20.1%	Dark Buff
1	31.7	22.7	Brown
3	24.9	29.1	Light Chocolate
5	16.1	34.3	Brownish Black

The firing behavior of this sample of Porters Creek clay is very similar to sample B-2, as a comparison of Fig. 6 and Fig. 7 shows. This sample has a somewhat longer firing range than sample B-2, as the percentages of magnesia and potash are lower, thus reducing the fluxing power. The porosity decreases gradually between cone 014 and cone 02 from 46.2 per cent to 34.2 per cent, with a firing linear shrinkage from 0.5 to 6.3 per cent. Above cone 1, overfiring results, and this is indicated by the rapid decrease in porosity, increase in shrinkage, and progressive darkening in color.

This clay could be used in the manufacture of common brick, as in the case of sample B-2, and limitations would be of the same character.

Maquoketa Shale - Sample B-6

TABLE XXII - Firing Behavior of Maquoketa Shale

Pyrometric Cone Equivalent - Cone 5-6 (1180° - 1190° C.)			
Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
012	26.7%	2.8%	Buff
010	24.8	5.3	Buff
08	22.3	6.9	Dark Buff

TABLE XXII - Firing Behavior of Maquoketa Shale (cont.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
06	5.8%	21.7%	Reddish Brown
04	2.0	23.0	Reddish Brown
02	2.3	21.5	Chocolate Red
1	1.6	16.1	Chocolate Red
3	2.6	2.6	Dark Chocolate

The Maquoketa shale has a short firing range, as can be seen from the apparent porosity and volume shrinkage curves in Fig. 8. Incipient vitrification is at cone 012, and the changes in apparent porosity and volume shrinkage are gradual until cone 08. Beyond cone 08 the porosity decreases rapidly, and the shrinkage increases inversely proportional to the porosity, until cone 04, where the curves level off. The abrupt changes in slope in the porosity and shrinkage curves, beyond cone 08, are indicative of the sudden formation of a low viscosity melt in the body, which quickly flows into the visicles and pore spaces of the body through the action of surface tension, thus decreasing the porosity and increasing the shrinkage.

Further proof of this low viscosity melt is indicated at temperatures beyond cone 04, where bloating of the mass results from gases which have not been fully liberated from the body during the heating. The volume shrinkage decreases from 23 per cent, at cone 04, to 2.6 per cent at cone 3, while the apparent porosity remains practically constant at around 2 per cent. This increase in volume, without a proportional increase in apparent porosity, indicates that a large

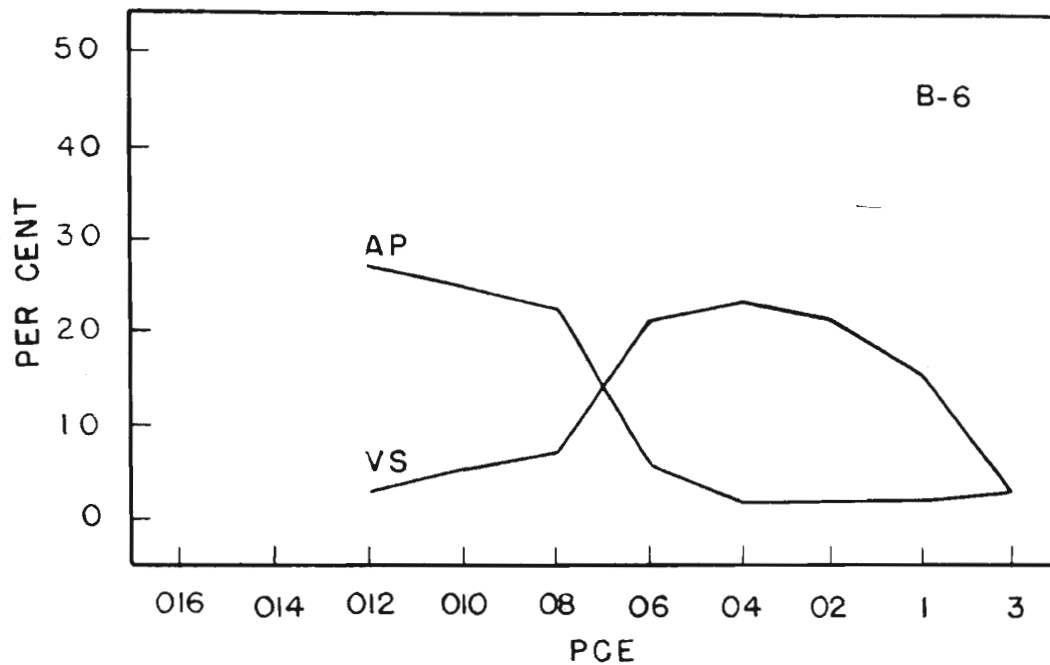


Fig. 8 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of Maquoketa shale, sample B-6

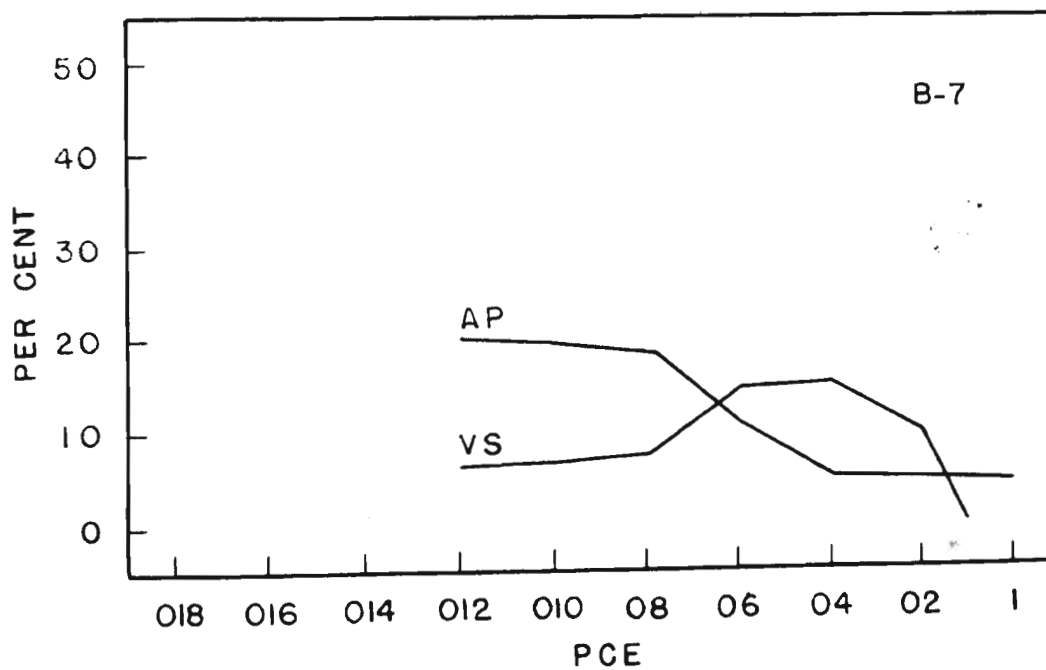


Fig. 9 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of Chester shale, sample B-7.



percentage of the voids produced by the bloating are of the non-connected cellular type, caused by entrapped gas bubbles.

The Maquoketa shale has little economic possibility for structural clay products because of the short firing range, which occurs at temperatures below the sudden changes in apparent porosity and volume shrinkage, and subsequent bloating action.

Another difficulty encountered is the fact that the sudden formation of the melt causes a reduction of the compression strength of the clay body during firing. Brick near the bottom of the setting would tend to deform under the overlying weight, and much low quality or worthless product would result.

Chester Shale - Sample B-7

TABLE XXIII - Firing Behavior of Chester Shale

Pyrometric Cone Equivalent - Cone 5 (1180° C.)			
Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
012	20.7%	6.8%	Buff
010	19.8	6.9	Buff
08	18.7	7.9	Dark Buff
06	10.7	14.4	Dark Red
04	5.0	14.8	Dark Red
02	4.7	11.2	Dark Red
1	4.5	5.7% Expansion	Very Dark Red

The apparent porosity and volume shrinkage curves in Fig. 9 indicate that incipient vitrification is at cone 012, with a high percentage of melt formation just beyond cone 08. With heat treatment

beyond cone 04, bloating occurs, resulting in progressive volume increase to the point at cone 1, where the test pieces have greater volume than in the unfired state.

As in the case of the Maquoketa shale, sample B-6, the bloating causes a cellular structure to form within the mass, resulting in a large number of entrapped voids, impervious to water penetration.

Because of the short firing range, from cone 010 to cone 08, and increased melt with subsequent bloating, this shale can be termed practically worthless for ceramic structural clay product manufacture.

#### Lower Pleasanton Shale - Sample C-1

TABLE XXIV - Firing Behavior of Lower Pleasanton Shale

---

Pyrometric Cone Equivalent - Cone 9 (1250° C.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
08	33.1%	1.7%	Buff
06	28.9	8.7	Red
04	22.4	14.1	Reddish Brown
02	17.2	18.9	Reddish Brown
1	13.1	21.6	Reddish Brown
3	1.1	26.1	Dark Chocolate
5	1.1	26.4	Chocolate Black

---

The lower Pleasanton shale has good firing properties with incipient vitrification at cone 08, and maximum shrinkage and minimum porosity at cone 3 and higher, as indicated by the curves in Fig. 10. Face brick of the first quality could be manufactured from this shale, using either the stiff-mud extrusion, or the dry press method of forming

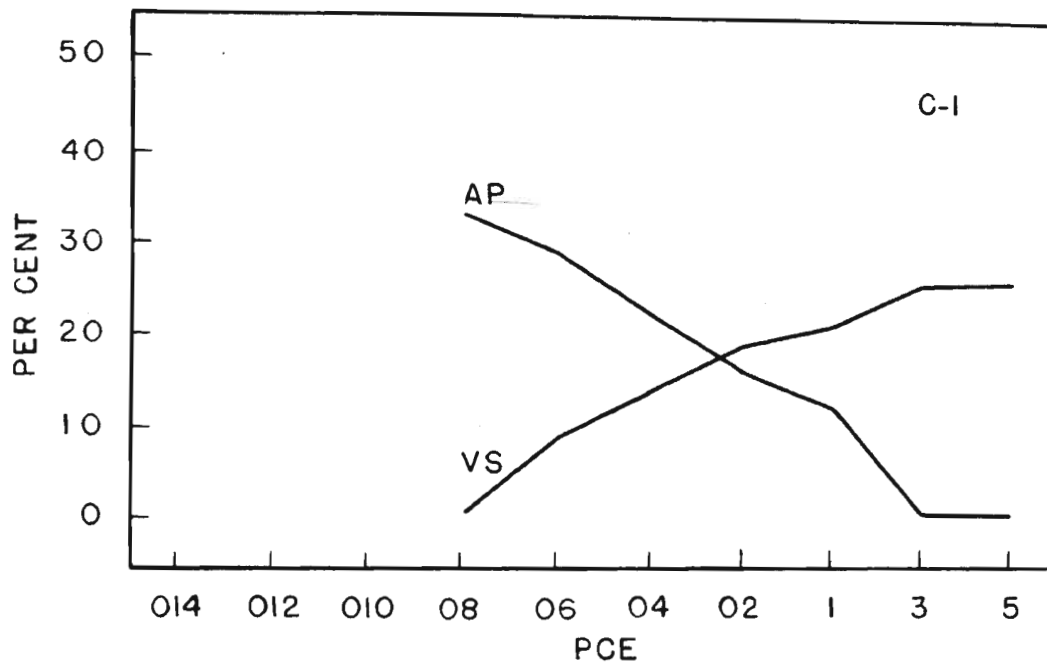


Fig. 10 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of lower Pleasanton shale, sample C-1.

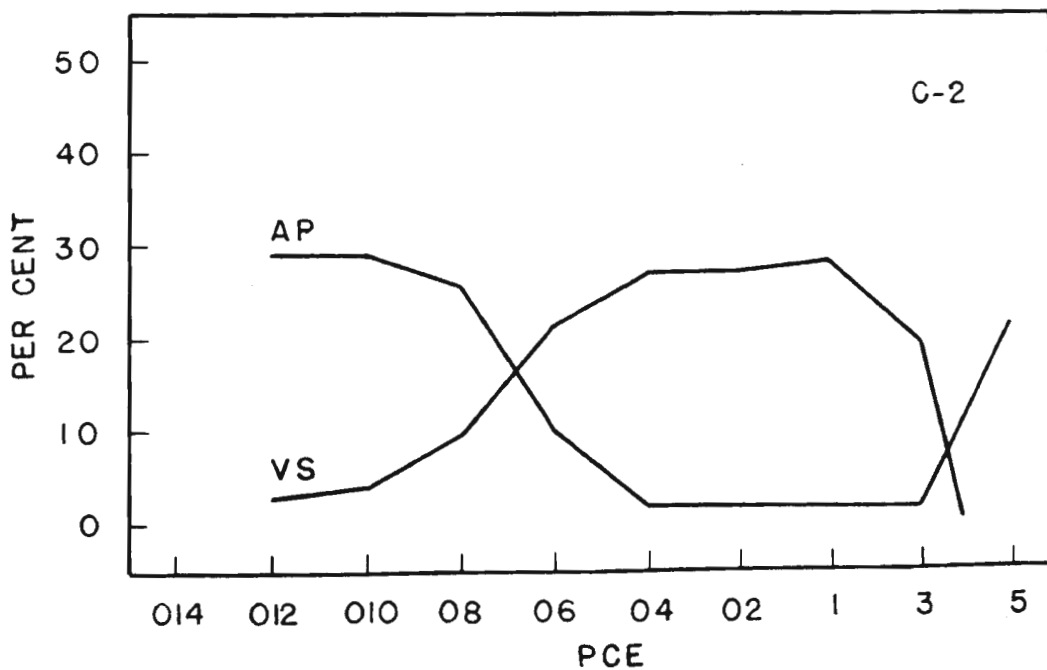


Fig. 11 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of upper Pleasanton shale, sample C-2.

the brick. The firing range of this shale lies between cones 06 and 2, where the burned material has the hardness of steel and the pleasing red brown color. Porosity varies between the limits of 28.9 per cent, at cone 06, and about 8 per cent at cone 2. The total drying and firing shrinkage (linear) of brick fired within this range would be from 7.2 to 11.8 per cent, which is not excessive. No warping, cracking, or checking occurs during firing, and the minimum compression strength is somewhat greater than 4500 pounds per square inch.

This shale also satisfies all the properties necessary for the manufacture of sewer pipe except that the plasticity is low, having a plasticity index of 6.7. An extrusion test would show if the plasticity were sufficient to allow shaping of the pipe bell without cracking the ware. Green strength is high enough to withstand handling of the product in sewer pipe form.

Upper Pleasanton Shale - Sample C-2

TABLE XXV - Firing Behavior of Upper Pleasanton Shale

---

Pyrometric Cone Equivalent - Cone 9 (1250° C.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
012	29.5%	2.8%	Buff
010	29.2	4.1	Buff
08	25.8	9.6	Buff
06	10.9	21.9	Reddish Buff
04	1.5	27.1	Dark Reddish Buff
02	1.5	27.3	Dark Red
1	1.5	28.9	Dark Red
3	1.3	19.0	Dark Red
5	21.5	28% Expansion	Chocolate

The upper Pleasanton shale has fair firing behavior properties for face brick, as indicated by the apparent porosity and volume shrinkage curves in Fig. 11. Incipient vitrification is at cone 012, and sufficient vitrification is present at cone 010 to give a steel-hard body. The face brick firing range of this shale is between the cones of 010 and 06, giving a temperature range of 115° C., which is long enough to maintain accurate firing in almost any type of kiln. The apparent porosity would range from 29.2 to 10.9 per cent, and the total linear shrinkage from 6.1 to 12.8 per cent within this range. Color would be a medium to reddish buff in the finished product.

Sewer pipe could not be made from this shale as the desirable porosity and shrinkage fall at too low a temperature, where a minimum temperature equivalent of about cone 02 is necessary for satisfactory salt glazing.

Although paving brick are seldom used anymore for street surfacing, some are manufactured for miscellaneous uses and for repair of existing facilities. This shale furnishes an excellent raw material for the manufacture of paving brick, as it has a long firing range, with low porosity and minimum volume change, between cones 04 and 1. The porosity remains constant at 1.5 per cent, and the volume shrinkage increases only from 27.1 to 28.9 per cent within this range. The paving brick could be brought to any heat treatment within this 75° C. temperature range, which would produce a dark color, a strong, dense, durable structure, free from cracks, voids and laminations, with extremely low porosity and uniform size.

As this shale occurs in an area in which no igneous rock outcrops, another profitable use may be assigned, which is that of railroad

ballast. In the manufacture of this product, the raw clay would need no special treatment, except to break it into lumps of smaller than fist size. It may be fired in tunnel, periodic, or scove kilns, or in a heap in which fire boxes have been placed with a layer of crushed coal covering the entire clay heap surface. Requirements for railroad ballast are a dense structure that is strong and durable, with a porosity of less than 3 per cent.

At temperatures higher than the equivalent of cone 1, a low viscosity melt forms with resultant bloating and volume expansion. At cone 5, the volume is 28 per cent greater than that of the shale in the unfired state.

Weston Shale - Sample C-3

TABLE XXVI - Firing Behavior of Weston Shale

Pyrometric Cone Equivalent - Cone 10-11 (1260°-1285° C.)			
Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
08	30.6%	1.8%	Dark Buff
06	22.5	13.3	Dark Buff
04	16.5	16.2	Dark Reddish Brown
02	13.6	20.6	Dark Reddish Brown
1	11.0	23.7	Chocolate Brown
3	4.3	25.8	Chocolate Brown
5	1.4	25.3	Dark Chocolate Brown
7	13.3	11.9	Black Chocolate Brown

The Weston shale has a long firing range, as indicated by the firing behavior curves in Fig. 12, and could be used for face brick

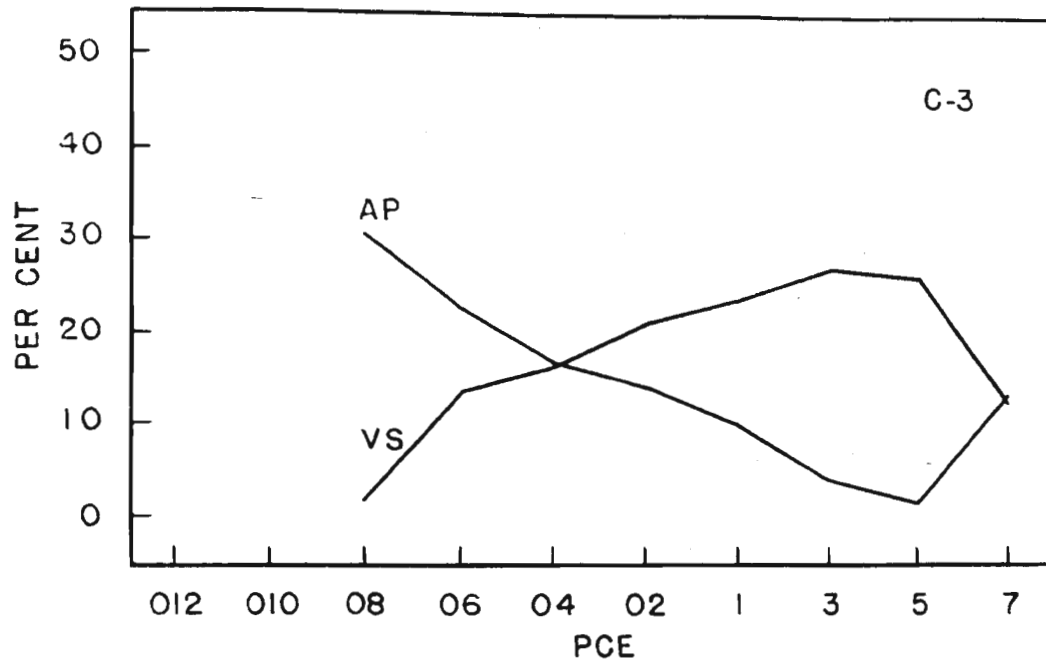


Fig. 12 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of Weston shale, sample C-3.

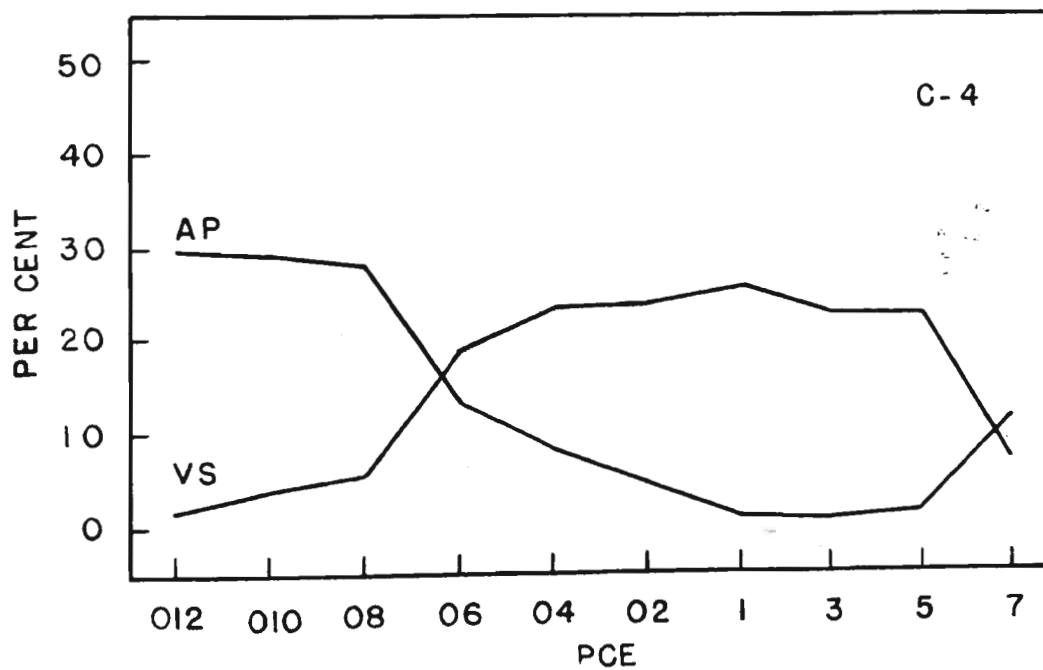


Fig. 13 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of Lawrence shale, sample C-4.

and sewer pipe manufacture. Incipient vitrification is at cone 08, with the lower limit of the firing range at about cone 05. The products may be fired to near cone 4, but a maximum of cone 3 would be better as original bloating occurs between these two cones. In this firing range of 115° C., the apparent porosity varies from 19.5 to 4.3 per cent, and the total linear shrinkage from 10.0 to 13.3 per cent. The color of the brick would grade from dark buff through chocolate brown. The brick is steel hard and easily meets the minimum compression requirements.

Sewer pipe made from this shale would have good plastic flow characteristics and green strength. Shrinkage is moderate, but grog additions would reduce this value considerably. The firing range is long enough to give good yield and falls in the temperature range of salt glazing.

The long firing range of the Weston shale may be attributed to moderately low amounts of lime, magnesia, and the alkalies, as shown by the chemical analysis, which total to about 5 per cent. The iron is probably largely present in the ferric oxide state, and does not produce powerful fluxing action until about cone 5 where bloating occurs.

Lawrence Shale, Sample C-4

TABLE XXVII - Firing Behavior of Lawrence Shale

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Pyrometric Cone Equivalent - Cone 10-11 (1260° - 1285° C.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
012	29.3%	1.4%	Dark Buff
010	28.9	3.7	Dark Buff
08	27.8	5.7	Dark Buff



TABLE XXVII - Firing Behavior of Lawrence Shale (cont.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
06	13.5%	18.9%	Light Reddish Brown
04	8.9	23.3	Light Reddish Brown
02	4.5	24.0	Dark Reddish Brown
1	1.0	25.8	Chocolate Brown
3	0.5	23.5	Dark Chocolate Brown
5	1.4	23.5	Dark Chocolate Brown
7	16.8	6.7	Black

This shale has a very long vitrification range, shown in graphic form in Fig. 13, with incipient vitrification at cone 012, or before, and overfiring at cone 3 and beyond. This long range may be attributed to the comparatively low amounts of lime, magnesia, and alkalies, which total under 5 per cent, considerably higher alumina content and lower silica, than the previous shales tested. Under these conditions, the changes in porosity and shrinkage are low with progressive firing. The shale has medium to good plasticity, and would readily flow, under the stiff mud process, without deformation, cracks, or other plastic defects.

Face brick manufactured with this shale should be fired within the firing range of cones 06 and 1, where the 120° C. temperature differential would make firing control comparatively simple. The porosity would vary from 13.5 to 1.0 per cent, and the total drying and firing linear shrinkage from 12.4 to 14.4 per cent. Although the porosity variation is moderate, shrinkage is practically constant, and uniform size ware

would result. The compression strength is well above 4500 pounds per square inch, and the color varies from reddish brown through chocolate brown.

Sewer pipe could be possibly made from this shale, although close temperature control is necessary above cone 02, where sufficient melt is formed to give deforming tendencies under load conditions.

Initial bloating occurs at temperatures just above cone 1, and continues for the remainder of the firing. The increase in porosity above cone 5 indicates that the voids, produced by bloating action, are of the connected vesicular type, rather than entrapped gas cells.

Lagonda Shale - Sample G-5

TABLE XXVIII - Firing Behavior of Lagonda Shale

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Pyrometric Cone Equivalent - Cone 11-12 (1285°-1310° C.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
010	30.4%	0.5%	Dark Buff
08	28.8	3.2	Dark Buff
06	25.7	8.7	Brownish Red
04	18.5	14.8	Dark Red
02	15.0	17.9	Dark Red
1	6.9	24.5	Very Dark Red
3	0.9	22.0	Chocolate Brown
5	0.9	22.0	Dark Chocolate Brown

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The Lagonda shale is a long burning clay, as indicated by the porosity and shrinkage curves in Fig. 14, with incipient vitrification at cone 010. The porosity change, with increasing heat treatment, is

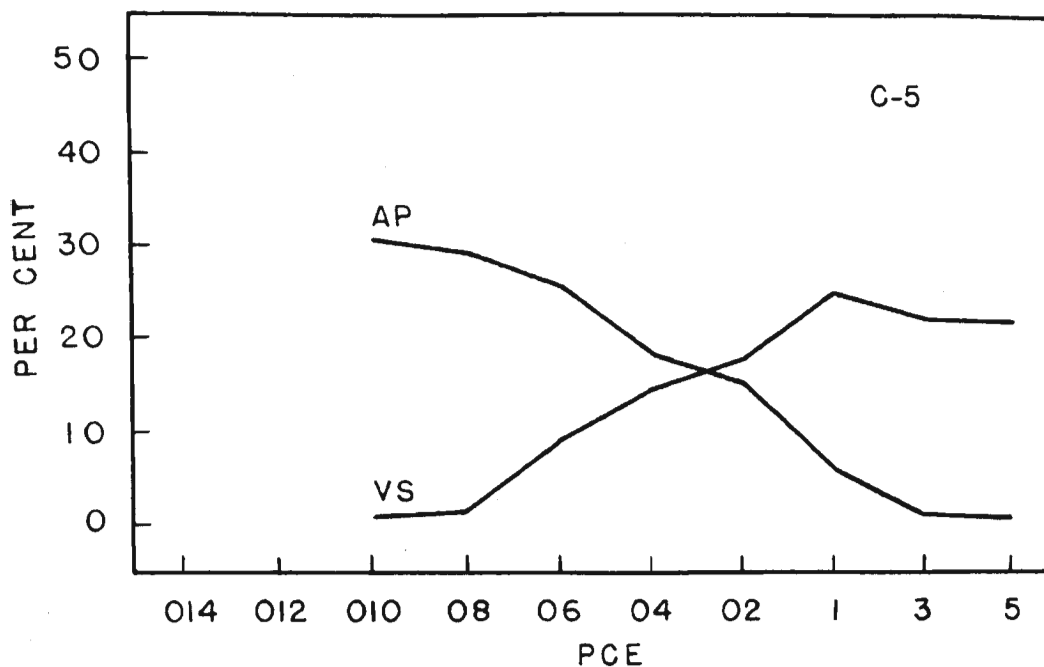


Fig. 14 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of Lagonda shale, sample C-5.

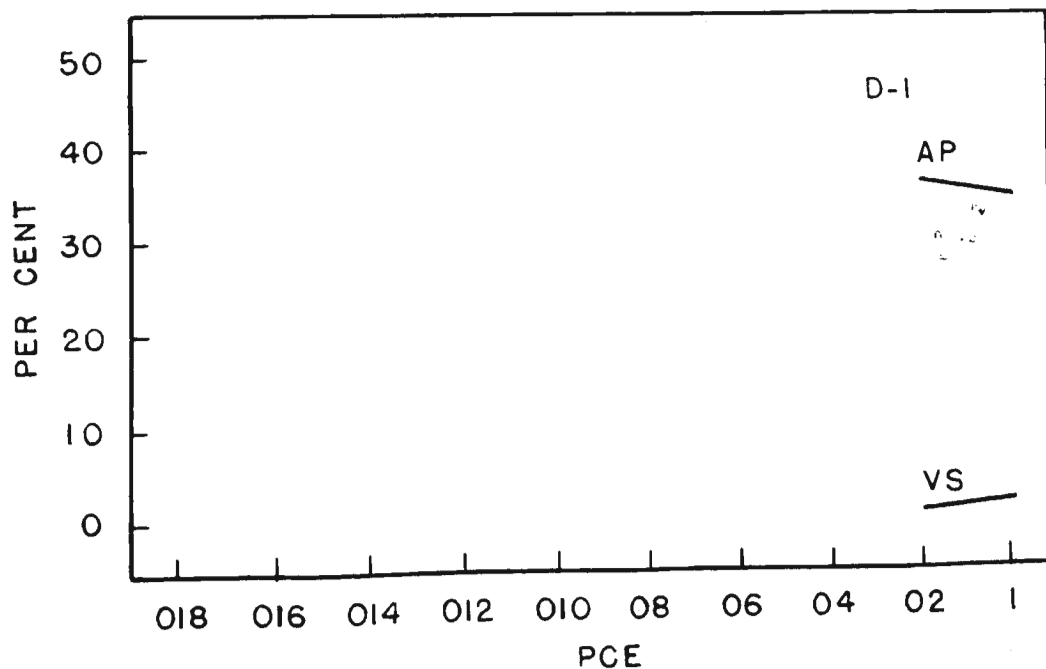


Fig. 15 Progressive changes in apparent porosity (AP) and volume shrinkage (VS) during firing of Northview shale, sample D-1.

gradual, and maximum firing shrinkage is at cone 1. Good quality face brick, brownish red to dark red in color, can be obtained between cones 06 and 01, a 105° C. temperature interval. The apparent porosity would vary from 25.7 to 11.0 per cent, and total shrinkage (linear) from 6.9 to 10.0 per cent. The strength is good and no imperfections are visible in the fired ware.

The shale has only poor to fair plastic properties, and therefore could not be used for sewer pipe manufacture.

At cone 1, and above, the volume increases with a decreasing porosity, which indicates that bloating of the cellular, entrapped voids, type is present.

Northview Shale - Sample D-1

TABLE XXIX - Firing Behavior of Northview Shale

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Pyrometric Cone Equivalent - Cone 5 (1180° C.)

Fired to Cone	Apparent Porosity	Volume Shrinkage	Color of Test Piece
02	36.6%	0.9%	Dark Cream
1	35.1	1.9	Dark Cream

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The Northview shale has a vitrification range of only 6 cones, a temperature interval of 85° C. This is due to an excessive amount of lime and magnesia, totaling more than 12 per cent in the chemical analysis. With incipient vitrification at 1095° C., and fusion at 1180° C., this clay is worthless for any type of heavy clay production.

## BLOATING CHARACTERISTICS OF THE SHALES

## PROCEDURE OF BLOATING TEST

Each of the raw shale samples were broken and shaped into test pieces approximately 1" X 3/4" X 3/4" in size. About a dozen of the test pieces were placed in a Remmy Lab. Kiln, No. 2150, and spaced so that none of the pieces were touching. Using a platinum platinum-10% rhodium thermocouple to measure the temperature, the kiln was heated at a rate of 25° C. per minute until 1000° C. was reached, and 10° C. per minute thereafter, for the remainder of the test.

The test pieces were withdrawn at intervals of 5° to 15° C., depending upon the rate of bloating of the particular shale in question. The first test piece was withdrawn when expansion was first evidenced by viewing the samples through a hole in the refractory door.

Immediately after withdrawal from the kiln, the test piece was broken apart to expose the nature of bloating inside the hard outer shell of vitrified clay. When the bloating had reached the excessive stage, where the test pieces were sticky and semi-fluid, the bloating tests were discontinued.

## DISCUSSION OF BLOATING TEST

## Lawrence Shale - Sample C-4

The progressive bloating encountered with an accurately controlled temperature increase is indicated by photomicrographs (Fig. 16 - 19) of the bloated products resulting from the Lawrence shale. This shale has a long bloating range between the initial bloating temperature and the stage of excess bloating or "burning."

In Fig. 16, the Lawrence shale at 1090° C., bloating has just

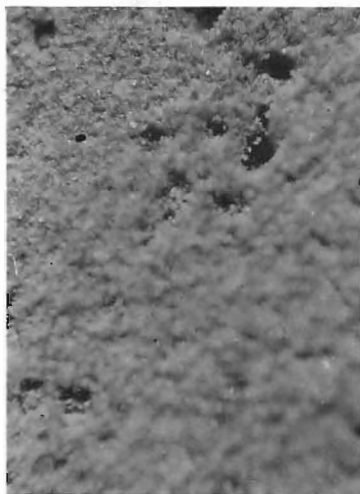


Fig. 16 Photomicrograph of initial bloating of Lawrence shale,  $1090^{\circ}$  C. 60 diameters.

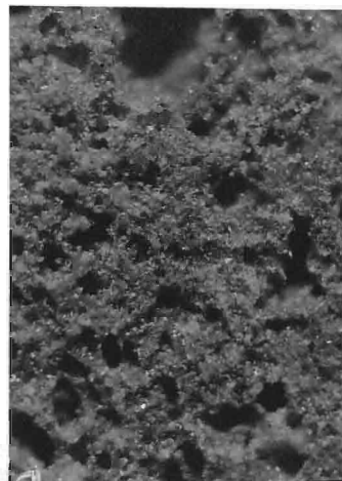


Fig. 17 Photomicrograph of bloating of the Lawrence shale at  $1110^{\circ}$  C. 60 diameters.

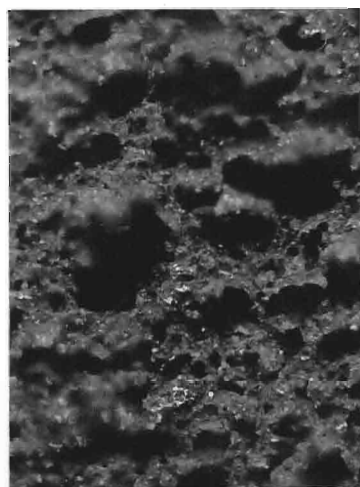


Fig. 18 Photomicrograph of good bloated product of Lawrence shale at  $1140^{\circ}$  C. 60 diameters.

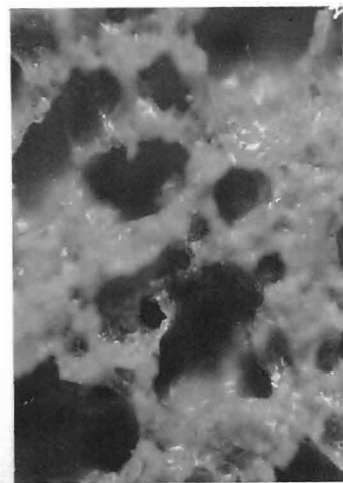


Fig. 19 Photomicrograph of Lawrence shale at upper limit of good bloated product.  $1160^{\circ}$  C. 60 diameters.

begun with the formation of small voids irregularly spaced throughout the mass. At  $1110^{\circ}$  C., Fig. 17, the bloating is fairly evenly distributed within the shale, the voids being somewhat larger and many times more numerous than at the lower temperature. This further expansion has resulted in numerous unconnected voids, or cells, along with some that are linked to one another by vesicles or gas tubes. Fig. 18, taken of the bloated shale at  $1140^{\circ}$  C., shows the further extent of bloating with more temperature increase. The typical light-weight bloated shale product has resulted, having a cellular structure, with practically all of the cells being the individual type, and very few of them interconnected. The size of the cells ranges up to about 0.3 mm.

Further increase in temperature enlarges the size of the cells, and at  $1160^{\circ}$  C., the upper limit of a good bloated shale product is reached. At this temperature, the groundmass is fused thoroughly, and the glassy portion is evident in Fig. 19. The upper limit of the cell size has increased to larger than 0.4 mm. Above this temperature, the melt becomes so fluid that the gas pressure within the cells causes rapid expansion of the cells themselves, and large voids of the interconnected type result. The extreme fluidity of the melt also decreases the strength of the groundmass, resulting in a collapse of the bloated mass within the stronger shell of the clay test piece.

Initial bloating of the Lawrence shale is below  $1090^{\circ}$  C., with the light-weight aggregate bloating range from  $1110^{\circ}$  -  $1160^{\circ}$  C. This bloating range, of  $50^{\circ}$  C., is the longest of any shale tested, and would make possible an optimum amount of quality product without too precise operating conditions. The product is strong and hard throughout this temperature range, and the cellular structure contains no

excessively sized voids and is uniform from center to shell. This is an excellent shale to use for the manufacture of a light-weight bloated shale concrete aggregate.

#### Cherryville Shale - Sample A-1

Initial bloating of the Cherryville shale occurs at a temperature of 1070°, and continues until past 1145° C., where excessive bloating or "burning" is pronounced. The temperature range for a good light-weight aggregate product is between 1115°-1140° C.

Calcining of the limestone fragments during the firing results in lime nodule formation in the finished product. The lime thus formed absorbs water after cooling, expands and breaks down the bloated product somewhat. This is not an unsatisfactory condition as the bloated shale product is crushed and screened before use as an aggregate in concrete.

A photomicrograph of the bloated product is shown in Fig. 20.

#### Maquoketa Shale - Sample B-6

The Maquoketa shale initially bloats at about 1120° C., and the bloating continues until burning takes place at temperatures above 1175° C. The bloating characteristics of the shale are good, but a product results, within the bloating range of 1145-1175° C., that has a higher specific gravity than the good light-weight concrete aggregate demands, being about 1.4. A comparison of the photomicrograph of this shale's bloated product, taken at 1165° C., with the excellent product shown in Fig. 18, shows that the numerous small cells within the groundmass are lacking; this may explain the higher specific gravity. Only a fair type of commercial bloated shale product could be obtained.



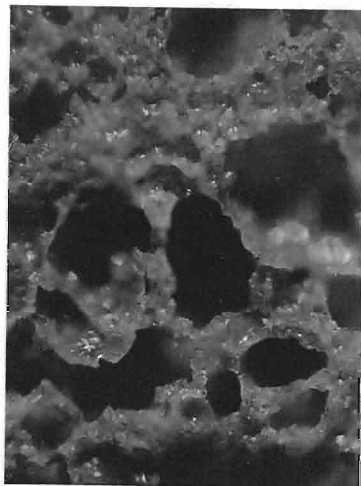


Fig. 20 Photomicrograph of bloated shale product produced from Cherryville shale. 1130° C. 60 diameters

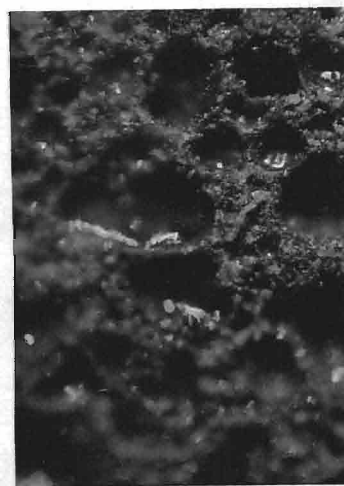


Fig. 21 Photomicrograph of bloated Maquoketa shale. 1165° C. 60 diameters



Fig. 22 Photomicrograph of Chester shale bloated product at 1185° C. 60 diameters

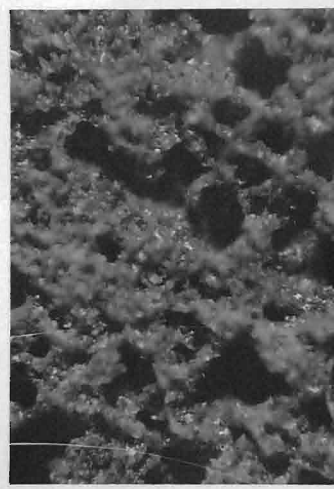


Fig. 23 Photomicrograph of bloated shale product from lower Pleasanton shale at 1135° C. 60 diameters

#### Chester Shale - Sample B-7

The Chester shale has good bloating characteristics within the temperature range of 1175°-1205° C., although the quality of product would be considered commercially low, owing to the thinly laminated form of the raw shale. As the bloating action occurs inside the material, surrounded by a hard, dense shell of vitrified clay, only a limited portion of each individual laminated piece bloats as the shell takes up the majority of the product.

If the Chester shale were ground, mixed with water, then molded and dried before the bloating operation, a good product would result as the laminated form is no longer present. Laboratory tests prove this to be true, and good bloating resulted from molded-shale fragments.

Fig. 22 is a photomicrograph of the Chester shale at 1185° C.

#### Lower Pleasanton Shale - Sample C-1

A good bloated shale light-weight aggregate results from the lower Pleasanton shale at temperatures of from 1110° C. to 1145° C. Within this range, a light, thin-shelled, and strong product is produced entirely suitable for concrete aggregate. The cellular structure is uniform and the specific gravity is less than 1. Initial bloating is at 1100° C. and burning at temperatures of 1150° C. and above.

At 1135° C., a cellular structure is produced as shown in Fig. 23.

#### Upper Pleasanton Shale - Sample C-2

Bloating starts in the upper Pleasanton shale at about 1050° C., but a good bloated shale product does not result until about 1090° C., and continues to 1130° C. Fig. 24 shows the cellular structure formed at 1110° C.. This is a very good lightweight aggregate, having a

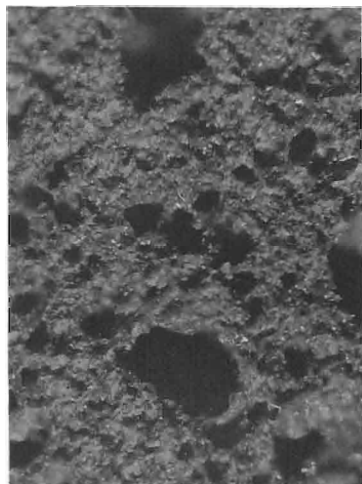


Fig. 24 Photomicrograph of the bloated shale product produced by upper Pleasanton shale at  $1110^{\circ}$  C. 60 diameters

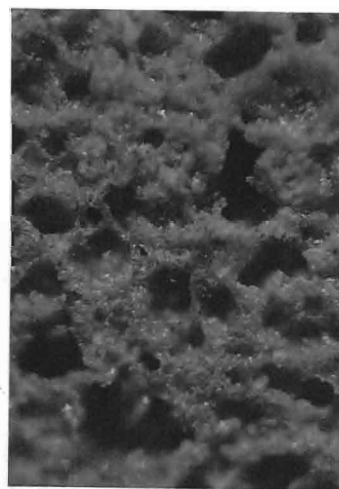


Fig. 25 Photomicrograph of the bloated shale product resulting at  $1130^{\circ}$  C. from the Weston shale. 60 diameters

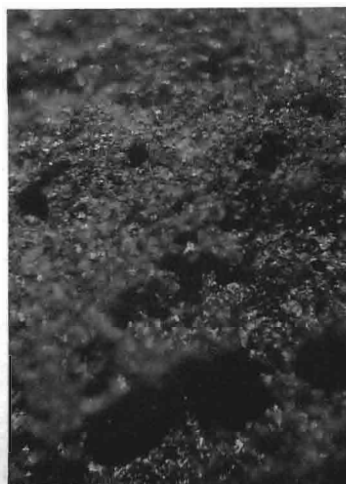


Fig. 26 Photomicrograph of the bloating occurring at  $1240^{\circ}$  C. with the Lagonda shale. Note the grading of the cellular structure into a vitrified product. 60 diameters

specific gravity of less than 1, with a very thin shell, therefore giving a maximum amount of light-weight product from the material fed into the kiln. The structure is uniform, strong and hard.

#### Weston Shale - Sample C-3

This shale also gives a very good bloated shale light-weight aggregate product within the range of  $1110^{\circ}$  -  $1150^{\circ}$  C. Initial bloating occurs at  $1090^{\circ}$  C. and burning at temperatures above  $1150^{\circ}$  C. The cellular structure produced at  $1130^{\circ}$  C. is shown in Fig. 25. The specific gravity of the product is about 1, and a uniform cellular structure is formed within the bloating range.

#### Lagonda Shale - Sample C-5

Bloating of the Lagonda shale occurs only at the fusion temperature,  $1225^{\circ}$  C., and higher. The bloating action is very slight, and occurs only at the very center of the test pieces. Fig. 26 shows the bloated area, at the bottom of the photomicrograph, which grades rapidly into the dense, vitrified area away from the center of the test piece.

#### Northview Shale - Sample D-1

No bloating action was evident in the Northview shale even at temperatures of  $1230^{\circ}$  C., which is  $50^{\circ}$  C. above the PCE of the shale.

#### CONCLUSIONS AS TO CAUSES OF BLOATING

The character of the bloating of a shale is directly related to the amount of alkalies present, the color of the raw shale which is an indication of the amount of organic material, and the amount of the clay mineral present, as indicated by the alumina content. These, and other properties related to the bloating action, are listed in tabular form in Table XXX.

TABLE XXX - Bloating, and Related Properties of Shales

Sample No.	Character of Bloating	Bloating Range	Raw Color	Ig. Loss	Incipient Vitrification	% Alkalies
A-1	Good	1115-1140°C.	Gray	10.50%	795°C.	2.15%
B-6	Fair	1145-1175	Brown	5.20	840	2.10
B-7	Good	1175-1205	Gray	6.50	840	1.99
C-1	Good	1110-1145	Gray	5.90	945	2.06
C-2	Very Good	1090-1130	Gray	6.60	840	1.94
C-3	Very Good	1110-1150	Gray	6.90	945	1.54
C-4	Excellent	1110-1160	Gray	7.30	840	1.35
C-5	Poor	At Fusion	Brown	5.20	890	0.58
D-1	None	None	Brownish Gray	11.90	1095	0.48

The alkalies present in shale determine, to a large extent, the temperature at which incipient vitrification takes place. At the point of incipient vitrification, a eutectic is formed of the most fusible constituents, and as the alkalies are fused at lower temperatures than the iron oxides, lime, and magnesia, their presence in moderate quantity will enable the eutectic to form. As soon as this eutectic, or initial melt, is formed, the other less fusible oxides are drawn into solution, resulting in progressive increase in melt with temperature rise.

As shown by the colors of the raw shales, those which tend to be gray are better bloating shales than those which are brown or brownish gray. Although the number of samples tested is not large, this would seem to indicate that those shales which contain carbonaceous matter in the finely disseminated form will give better bloating characteristics. The liberation of CO or CO<sub>2</sub> from the organic or free carbon is largely the cause of good bloating.

The action of decomposing carbonates may be considered secondary to bloating reaction. This conclusion is evident by a comparison of the Cherryville shale, sample A-1, with shale samples C-1 to C-4. All of these shales are gray in color, and, with the exception of the Cherryville shale, contain approximately 4 per cent, or less, lime and magnesia. The Cherryville shale contains 8.49 per cent lime and magnesia, yet it's bloated product is of no better quality than the other samples which contain lesser quantity of these materials. in the form of limestone and dolomite. The rapid heating rate, necessary to produce the cellular structure for light-weight aggregate, undoubtedly retards the decomposition of the limestone and dolomite, which allows the gases formed by the oxidation of the carbonaceous matter to primarily cause the bloating, as carbon is oxidized as low as 200° C..

Another reaction which influences the bloating is the decomposition of the clay mineral, with resultant liberation of the chemically combined water in the form of steam. Although the loss of the chemically combined water begins at about 150° C., the rapidity of the temperature increase in the bloating test insures that the reaction is not fully complete at bloating temperatures. The alumina content is a measure of the amount of clay mineral from which the chemically combined water is evolved. In general, the greater the clay content, the better the bloating character of the shale.

A summary of the shale properties that make for good bloating characteristics are as follows:

1. Alkalies should be present in moderate amount to form the base of the eutectic composition. The degree of melt formation should be gradual with increasing temperature; this will give a long, progressive bloating range.

2. The oxidation of finely disseminated carbonaceous matter, with resultant CO and CO<sub>2</sub> liberation within the melt, is of a primary importance after the melt has reached the proper viscosity to give good bloating.
3. The decomposition of limestone and dolomite, liberating CO<sub>2</sub> is of secondary importance in the production of a good bloated shale lightweight aggregate.
4. The amount of clay mineral present has a definite bearing on the bloating characteristics of a shale, as the chemically combined water, liberated as steam, aids the bloating effect.

#### PORTERS CREEK CLAY LIGHT-WEIGHT AGGREGATE

The Porters Creek clays of Southeast Missouri could be used for light-weight concrete aggregate production, for a low specific gravity material results when the clay is vitrified. The burned material is moderately strong and hard, and is abrasion resistant. Although the clays tested in this investigation do not possess the levity of Porters Creek clays of other localities, the specific gravity is sufficiently low for light-weight concrete aggregate. (53)

The Sample B-2, of Porters Creek Clay, will give a light-weight product suitable for aggregate within the temperature range of 840° 1005° C.. The specific gravity within this range varies from 1.31 to 1.41, and the burned material is moderately hard and strong. Porosity is about 40 per cent.

The Porters Creek clay, sample B-4, when fired to between 795° and 945° C., gives a light-weight product which has a specific gravity of from 1.27 to 1.37 and is strong and moderately hard. The porosity of the light-weight product varies from 46.5 to 41.0 per cent.

For the manufacture of a light-weight aggregate, the Porters Creek

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(53) Whitlatch, G. I. op. cit. pp. 212-213.

clays only need be mined, broken into lumps, and air dried before firing. A rotary kiln would be most economical for firing, but periodic kilns of the downdraft type could probably be profitably used. The burned material needs only crushing and screening to the desired sizes before use. As the firing range is long for both clays, good light-weight aggregate could be obtained without a maximum of temperature control.

The sampling and testing of the Porters Creek clay from other locations in the state would undoubtedly uncover clays which would produce a product of better levity.



## MORTAR PROPERTIES USING MISSOURI CLAYS AND SHALES AS PLASTICIZERS

## METHOD OF INVESTIGATION

All of the mortars were prepared in the proportion, by weight, of one part of the cement-clay mixture to three parts of sand. The amount of clay, or shale, used in the mortar was varied by substitution of clay for 20, 30, and 40 per cent, by weight, of the cement portion of the mortar. Equal substitution was used for the mortars containing hydrated lime instead of clay. Standard silica sand from Ottawa, Ill., graded to pass a No. 20 and be retained on a No. 30 sieve, was used in the preparation of all mortars.

The dry materials were all kept in storage at approximately 70° F., and the mixing water was maintained at the same temperature.

Enough Portland cement, of a standard brand, was obtained to meet the requirements of the entire investigation. Before use, the cement was screened through a No. 40 sieve to thoroughly break up the lumps and eliminate any foreign material. The clays and shales were ground to pass a No. 20 sieve. Before addition to the mixing water, the cement-clay portion of the mortar was thoroughly dry mixed in the correct proportions.

The mortars were prepared by the method specified for masonry cement, A. S. T. M. Designation C91-40.<sup>(54)</sup>

"The mortars shall be mixed in a nonabsorbent bowl of about 1 gal. capacity. A measured quantity of water shall be poured into the bowl which has previously been wiped with a damp cloth. A 500 gram portion

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(54) A. S. T. M. Standards, Part II. Standard specifications for masonry cement. pp. 7-13 (1944)

of the cement shall then be added and stirred into the water with the fingers of one hand until all the cement is wetted. Approximately 800 gram of sand shall then be added and the stirring continued for 30 seconds. The remainder of 1500 grams of sand shall then be added and the mortar mixed for 75 seconds by vigorous and continued stirring, squeezing, and kneading with one hand. The mortar shall then be allowed to stand for 60 seconds, and then mixed for another 60 seconds. During the operation of mixing, the hands shall be protected by rubber gloves."

The consistency of the mortars used in the water retention and compression cube tests is standardized by means of a flow table, which is described in Section 21c of A. S. T. M. Designation C91-40:

"The flow table apparatus consists of a rigid frame with a flat circular top, so mounted on a vertical shaft that it can be raised and dropped through a fixed height of  $1/2$  plus or minus  $1/32$  inch by means of a rotated cam. The top shall be of noncorrodible metal 10 inches in diameter and with the attached shaft shall weigh 9 lb. plus or minus 1 ounce. The frame shall be attached rigidly to a concrete pedestal, which in turn shall be attached rigidly to the floor. The mold shall be of a noncorrodible material, 4 inches in inside diameter at the base,  $2\ 3/4$  inches at the top, and 2 inches in height."

"The top of the flow table shall be carefully wiped dry and the flow mold placed at the center and filled with mortar. In filling the mold, the mortar shall not be rammed, but gently smoothed off level with the top of the mold by aid of a trowel and the mold then removed. Immediately, the table shall be dropped through a height of  $1/2$  inch, 25 times in 15 seconds. The flow is the resulting increase in diameter

of the mortar mass, expressed as a percentage of the original diameter. Trial mortars shall be made with varying percentages of water until the standard consistency is obtained. Each trial shall be made with fresh mortar. The quantity of water shall be expressed as a percentage of the weight of the combined dry materials. The mortar shall be of standard consistency for the molding of compression cubes when the flow is 65 to 80 per cent. Mortars for the water retention test shall be mixed to a flow of 100 to 115 per cent."

#### WATER RETENTION TEST

The water retention of a mortar is a test designed to measure the resistance of the mortar to being dried out by absorption of the water by the porous brick, tile, or unit being laid up. The removal of the water from the mortar results in a weakening of the bond between the mortar and the structural units. Most structural units are saturated with water before the wall is laid up to partially eliminate this effect.

The water retention apparatus, as described by Section 26a, A.S.T.M. Designation C91-40, is described as follows:

"This apparatus consists of a water aspirator controlled by a mercury column relief and connected by a way of a three-way stopcock to a funnel upon which rests a perforated dish about 15.5 cm. in diameter and 2 cm. in height. A mercury manometer indicates the vacuum. A rubber gasket shall be sealed to the top of the funnel and shall be kept wet during a test to insure a seal between the funnel and dish. The perforated dish shall be made of nonabsorbent material. Hardened filter paper equivalent to Carl Schleicher and Schull filter paper No. 575 shall be used. It shall be of such diameter that it will lie flat and completely cover the bottom of the dish."

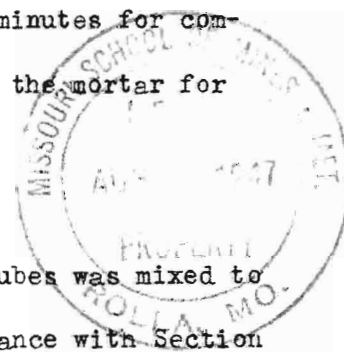
The method of test, as described by Section 26b, A. S. T. M. Designation C91-40, is as follows:

"The mortar shall be mixed to a flow of from 100 to 115 per cent in accordance with the outlined procedure. Immediately after making the flow test, the mortar on the flow table shall be mixed for 30 seconds with that remaining in the mixing bowl, and then evenly distributed without compacting over the sheet of dampened filter paper in the perforated dish and the surface leveled off flush with the rim of the dish by drawing a straightedge across the dish with a slightly sawing motion. The dish shall then be seated on the wetted gasket, and with the mercury column set at 2 inches, the stopcock shall be turned to apply the vacuum to the funnel for 60 seconds. The contents of the dish shall then be immediately removed by a putty knife or square end spatula and placed in the mold on the flow table, each portion being puddled with the fingers. The flow is again determined and this water retention value expressed as a percentage of the original flow for the water retention test mortar. The entire operation shall be carried out without interruption and as quickly as possible and shall not require more than 7 minutes for completion, starting from the completion of the mixing of the mortar for the first flow determination.

#### COMPRESSION STRENGTH OF MORTARS

The mortar used for making the compression test cubes was mixed to a consistency of from 65 to 80 per cent flow in accordance with Section 21b, A. S. T. M. Designation C91-40. The procedure for the molding and testing of the compression cubes is briefly described as follows:

The mortars were mixed to a standard consistency of 65 to 80 per cent flow, and 2-inch cube specimens were formed in accurately dimensioned



molds of noncorrodible metal. Immediately after molding, the molds were placed in a damp closet, maintained at a relative humidity of 90 per cent or more and about 70° F., for 48 to 52 hours. The cubes were then removed from the molds and left in the damp closet until 7 days have elapsed from the time of molding. After aging in the damp closet for 7 days, the cubes for the 28-day compression check were immersed in clean, running water. The compression tests on the cubes aged for 7-days were made immediately after removal from the damp closet, and the compression tests on the 28-day specimens immediately after removal from the storage water.

The 7-day and 28-day aged cubes were tested on a compression strength machine of the screw type, applying the load at not less than 1000, nor more than 6000 lbs./sq.in./minute.

Three cubes of each mortar for each aging period were tested, and the compression strength was taken as the average of these three specimens.

#### DISCUSSION OF MORTAR PROPERTIES

The results of the water retention and compression tests for each of the clays and shales under investigation are given in Table XXXI. Additional mortars, using lime instead of clay, were also tested to show comparative results of the lime mortars to the clay mortars, as the lime mortars are more or less standard in use.

#### WATER REQUIREMENTS OF MORTARS

The water requirements of the mortars to produce the standard consistency is largely dependent upon the plasticity of each clay or shale as indicated by the plasticity index. This relationship is to be expected, as both the plasticity index and the mortar's water requirement are measures of the water necessary to produce a standard consistency.

TABLE XXXI Water Retention and Compression Strength of Mortars

Per cent replacement of cement by clay & sample No.	% water content of mortar for 65-80% flow	Compression strength in psi		% water content of mortar for 100-115% flow	% water retention
		7-day	28-day		
20 A-1	15.6 %	1493	2510	16.9 %	40.6 %
30 A-1	16.9	1035	1629	18.7	56.8
40 A-1	17.5	683	1060	19.1	62.9
20 B-1	13.6 %	1908	3071	14.4 %	36.4 %
30 B-1	14.0	1566	2659	14.8	42.4
40 B-1	14.1	1181	2072	15.1	43.8
20 B-2	18.1 %	1062	1996	19.3 %	37.5 %
30 B-2	19.4	735	1389	21.9	40.6
40 B-2	21.9	448	864	24.2	50.0
20 B-4	18.9 %	801	1523	20.6 %	39.4 %
30 B-4	21.0	529	1083	23.0	40.0
40 B-4	22.5	409	861	24.2	50.0
20 B-6	14.4 %	1513	2303	16.2 %	41.2 %
30 B-6	15.0	1225	1789	17.1	38.2
40 B-6	15.6	851	1387	18.1	38.9
20 B-7	14.4 %	1403	2215	16.3 %	35.3 %
30 B-7	15.3	893	1637	17.1	38.3
40 B-7	15.8	642	1163	18.1	47.1
20 C-1	14.5 %	1517	2446	16.2 %	45.7 %
30 C-1	15.0	1254	2080	17.0	50.0
40 C-1	15.6	821	1354	17.6	57.2

TABLE XXXI Water Retention and Compression Strength of Mortars (cont.)

Per cent replacement of cement by clay & sample No.	% water content of mortar for 65-80% flow	Compression strength in psi		% water content of mortar for 100-115% flow	% water retention
		7-day	28-day		
20 C-2	14.6 %	1604	2262	17.0 %	47.3 %
30 C-2	15.5	1233	1819	17.8	58.8
40 C-2	16.5	895	1223	18.7	59.8
20 C-3	14.6 %	1657	2540	16.6 %	48.6 %
30 C-3	15.4	1477	2067	17.6	62.3
40 C-3	16.1	897	1216	18.4	65.8
20 C-4	14.7 %	1478	2403	16.6 %	47.6 %
30 C-4	15.5	1237	1958	17.6	61.8
40 C-4	16.2	895	1305	18.4	61.4
20 C-5	14.7 %	1845	2418	16.5 %	40.0 %
30 C-5	15.2	1297	1930	17.5	46.2
40 C-5	15.9	919	1299	18.1	48.5
20 D-1	14.0 %	1542	1976	15.5 %	43.8 %
30 D-1	14.5	1256	1575	16.5	51.8
40 D-1	15.0	950	1363	17.5	53.3
20 Lime	15.1 %	1921	2900	17.0 %	57.1 %
30 Lime	16.1	1580	2176	18.6	68.7
40 Lime	17.1	1150	1472	23.0	77.1
100% Cement	13.6 %	2577	3560	14.6 %	32.3 %

The water required to give 65-80 per cent flow varies from 0.8 to 2.5 per cent less than the water requirement for 100-115 per cent flow. Each increase of 10 per cent of clay substituted for cement required water increase of from 0.1 to 0.4 per cent for the loess, 1.3 to 2.5 per cent for the Porters Creek clays, and 0.5 to 1.4 per cent for the shales, with the 65-80 per cent flow. The water required, for 100-115 per cent flow, with increase of 10 per cent of clay substituted for the cement, was somewhat higher than the requirement for 65-80 per cent flow.

#### WATER RETENTION OF MORTARS

Of all the clays and shales tested, only one of these, the Weston shale, sample C-3, met the minimum requirements of 65 per cent for water retention, as specified by A.S.T.M. specifications,<sup>(55)</sup> and this value is obtained at nearly 40 per cent replacement of the cement by clay. Graphic presentation of the relationship of the water retention values to the per cent clay replacement of the cement is shown in Figures 27, 28 and 29.

The water retention of a mortar, using shale or clay as a plasticizer, depends on (1) the amount of fine grained particles and those of colloidal size, which exhibit a tremendous surface area to be wetted, (2) the type and amount of adsorbed salts on the clay material particles, and the degree to which these adsorbed ions are hydrated, and (3) the tenacity which the water films have for the clay material particles, which is related to the type of surface of the clay particles and the type of clay mineral.

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(55) A. S. T. M. Standards, Part II. Standard specifications for masonry cement. pp. 7-8 (1944)



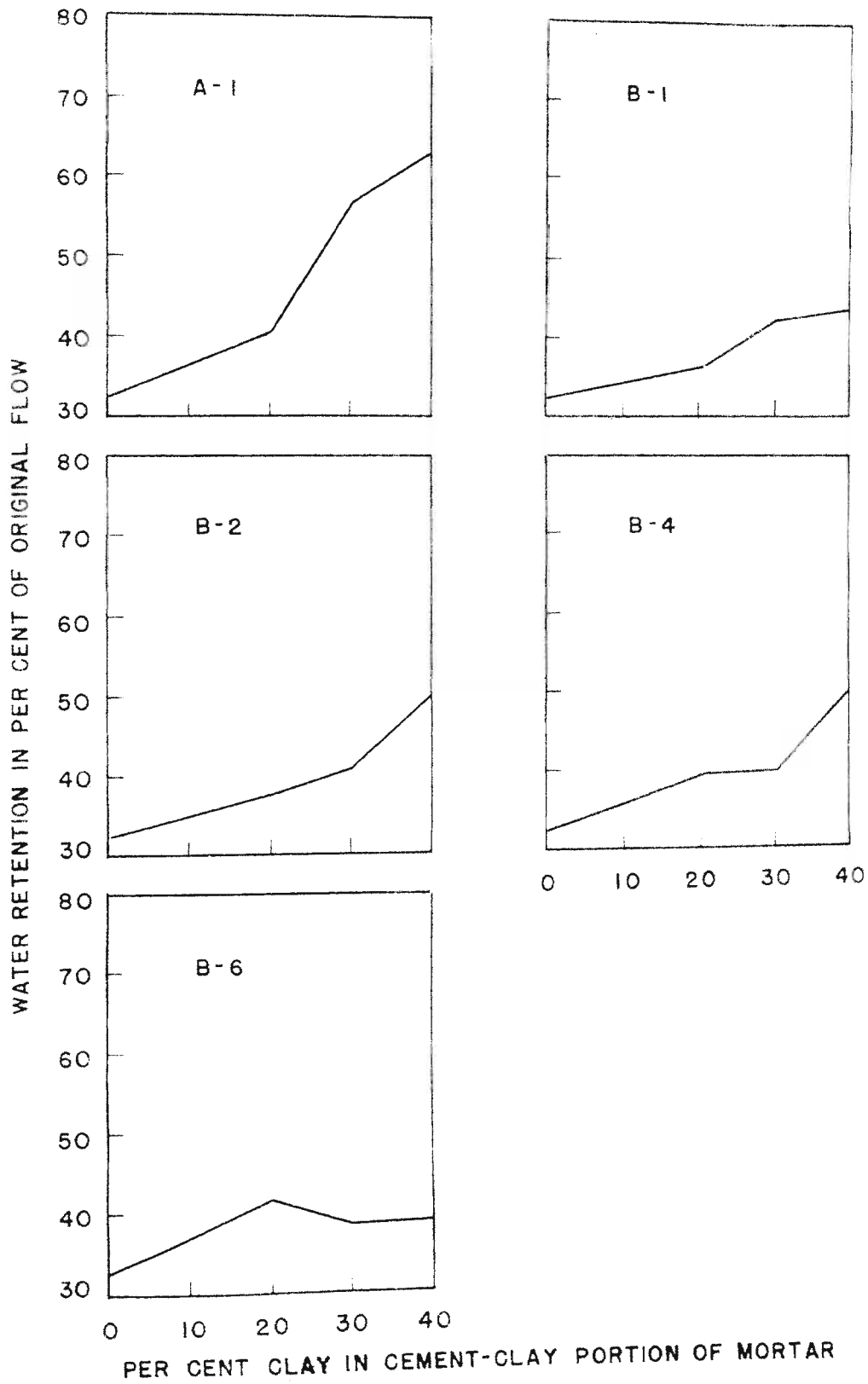


Fig. 27 Relation of water retention to the per cent of clay material in the cement-clay portion of the mortar

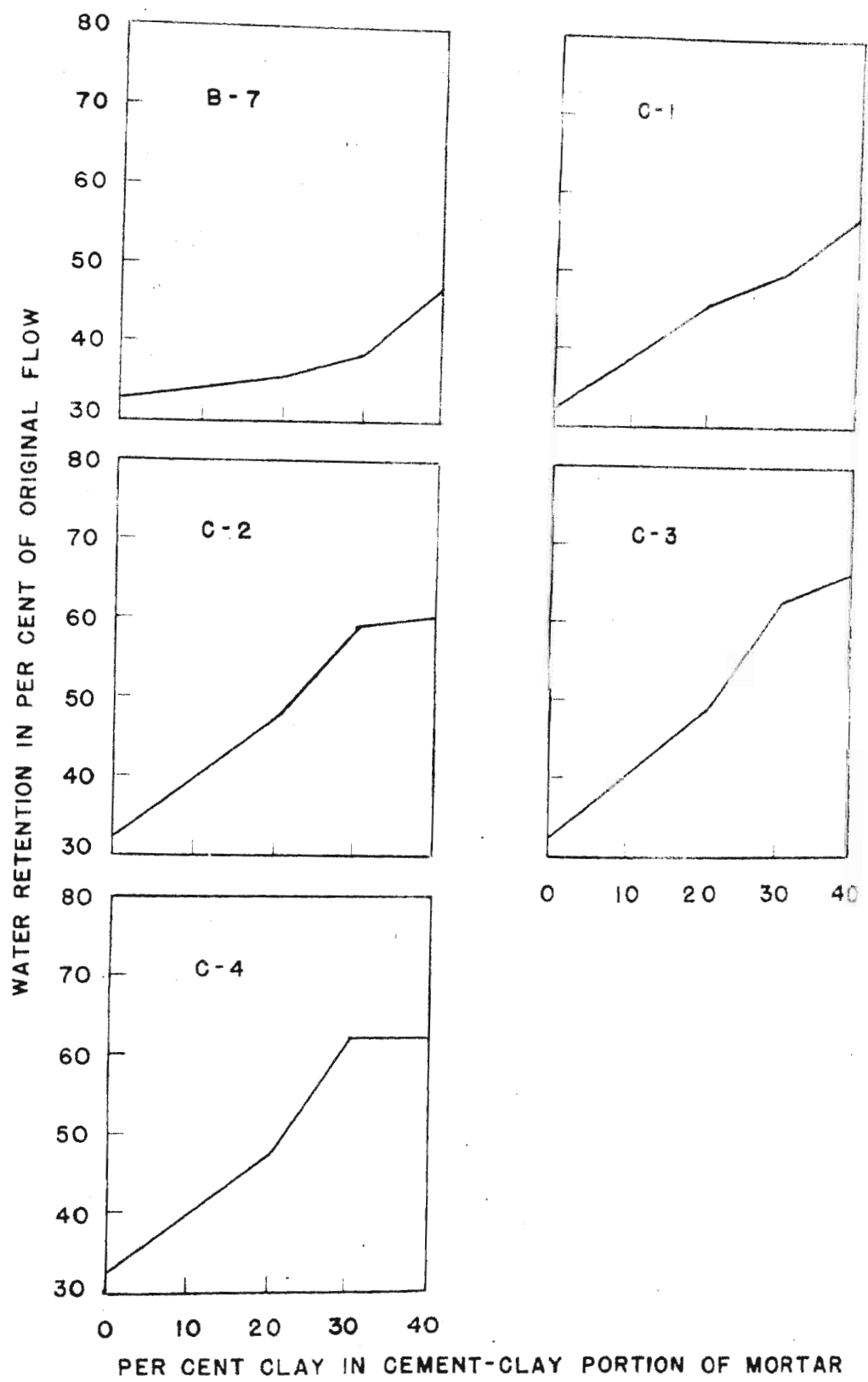


Fig. 28 Relation of water retention to the per cent of clay material in the cement-clay portion of the mortar.

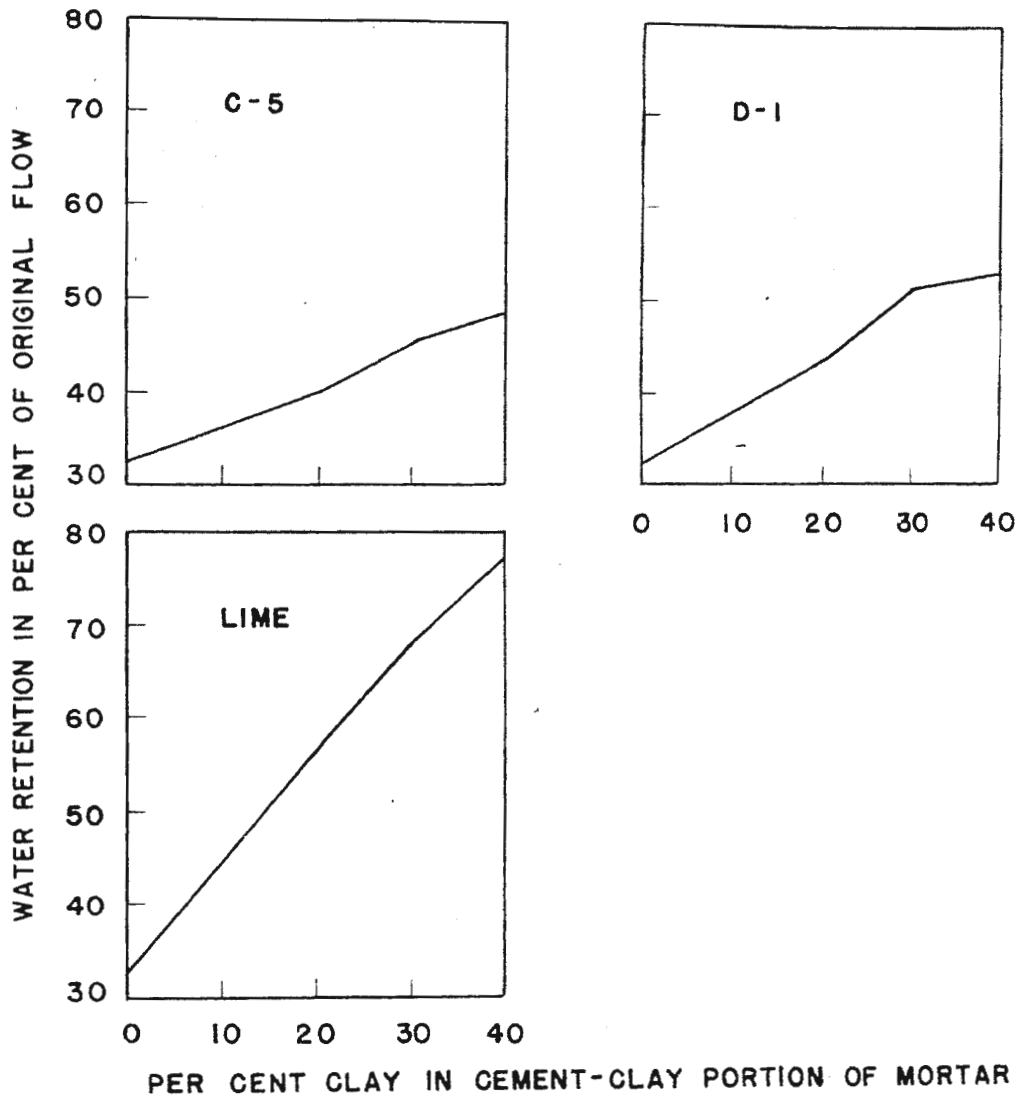


Fig. 29 Relation of water retention to the per cent clay material in the cement-clay portion of the mortar.

A comparison of the water retention values to the amount of clay material of colloidal size fails to show any definite relationship of increased water retention to clays having high amounts of colloidal material. This may be attributed to additional factors of adsorbed salts, type of clay mineral, and the type of particle surface, which also have an influence on the water retention values.

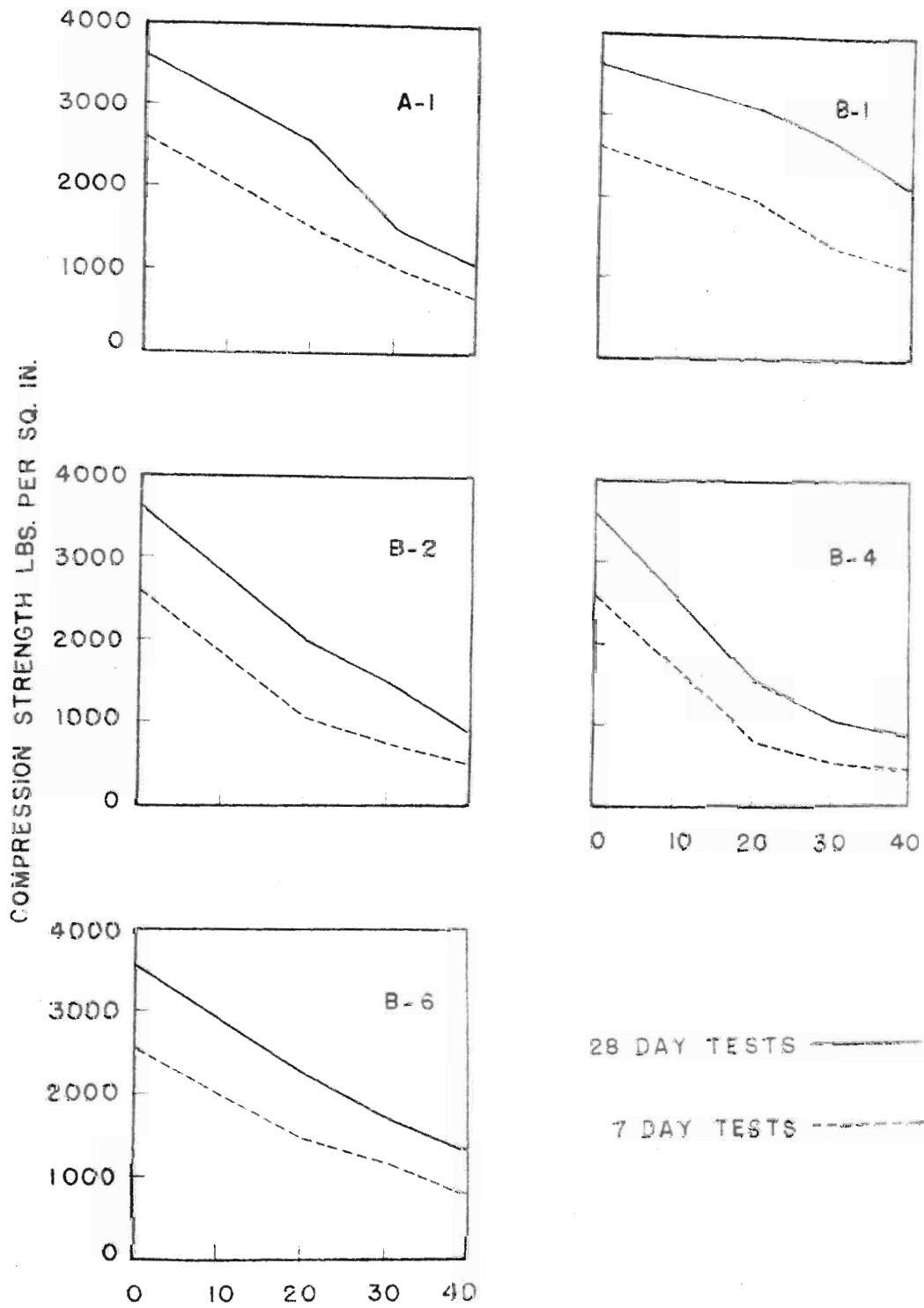
The mortar, containing shale sample C-3 in 40 per cent replacement of the cement, was the only one which meets the minimum water retention requirement of 65 per cent. Mortars having values between 60 and 65 per cent at 40 per cent replacement of the cement by the clay include those using samples A-1 and C-4 as plasticizers. These two shales could probably be used as plasticizers in mortars if an ungraded sand were used and the structural units saturated with water before the wall is laid up.

In comparison with the lime mortars, even the use of shale sample C-3 does not give too favorable results. With the shale a water retention of 65 per cent is gained at nearly 40 per cent replacement, whereas the lime mortar gave minimum water retention requirement at about 28 per cent replacement of the cement by lime.

#### COMPRESSION STRENGTH OF MORTARS

The compression strength of the mortars in which the cement has been replaced with 20, 30, and 40 per cent of clay is shown graphically in figures 30, 31 and 32. As can be noted from the curves, increased replacement of the cement by clay reduces the compression strength of the mortars sometimes as much as 30-35 per cent, with additional 10 per cent replacement.

In general, the mortars utilizing clays with larger amounts of



PER CENT CLAY MATERIAL IN CEMENT-CLAY PORTION OF MORTAR

Fig. 30 Compressive strength of mortars containing shale (A-1 & B-6), loess (B-1), and clay (B-2 & B-4), at 7 and 28 days.

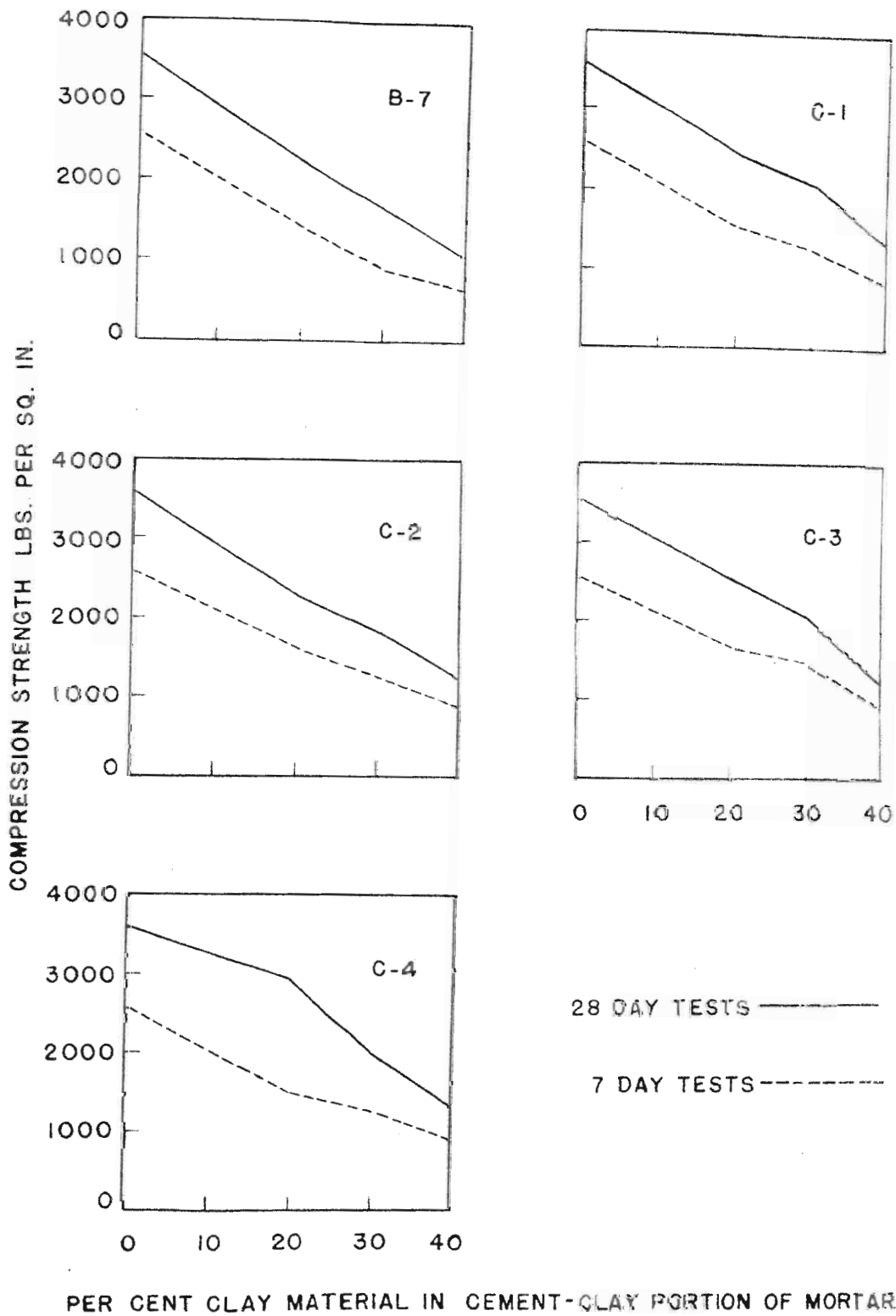


Fig. 31 Compressive strength of mortars containing shale at 7 and 28 days.

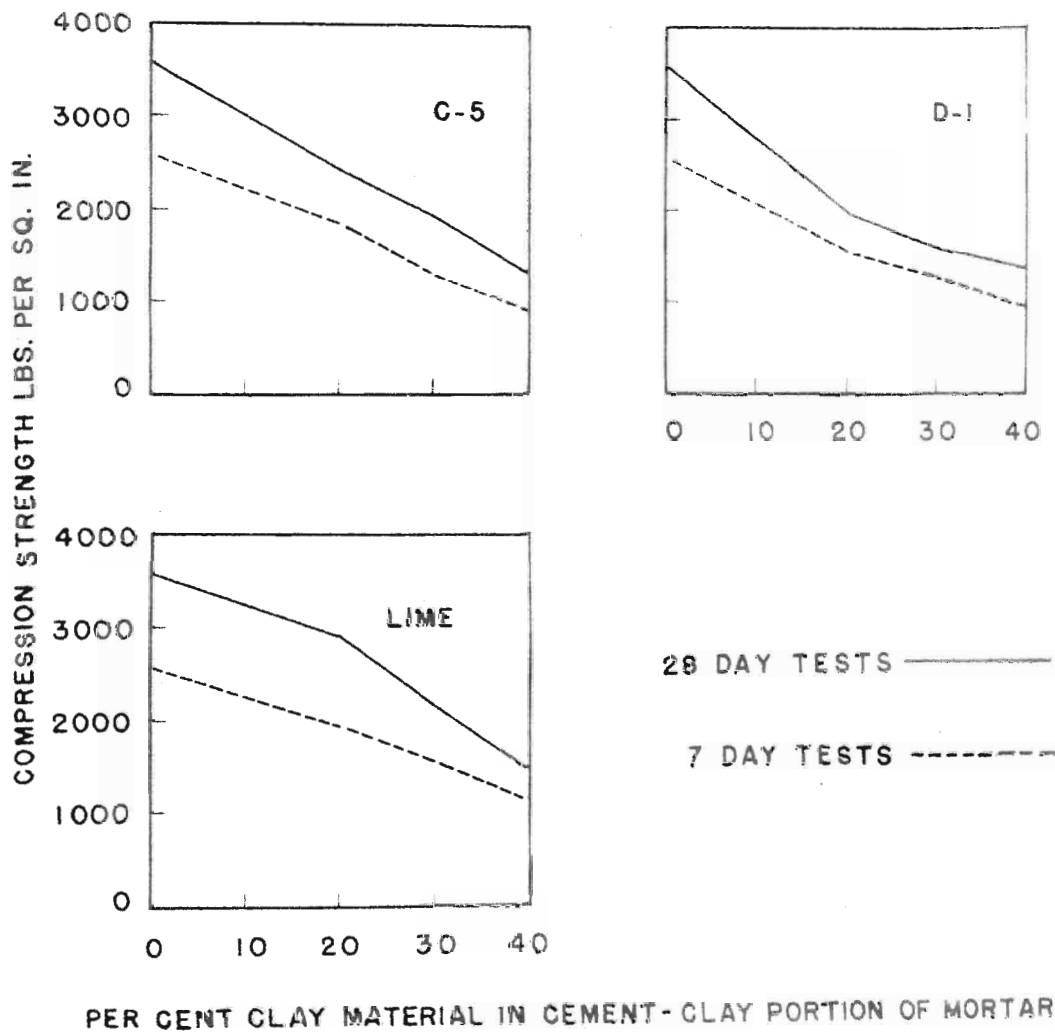


Fig. 32 Compressive strength of mortars containing shale (C-5 & D-1) and lime at 7 and 28 days.

colloidal size material present, such as the Porters Creek clays, and the Cherryville and Chester shales, gave consistently lower compression values at the end of 7 and 28 days. The colloidal clay material in the mortar probably surrounds the cement particles and suppresses the crystal growth, giving more centers of crystallization, but less interlocking growth, and therefore less strength.

The loess, sample B-1, gave mortars with replacement up to 40 per cent of equal strength to lime mortars at the end of 7 days, and superior compression strengths at 28 days. This may be explained by the fact that the loess, which contains above 50 per cent free silica, and a small proportion of clay material, serves mainly to increase the sand content of the mortar, rather than to introduce clay material plasticizer into the mix.

A. S. T. M. specifications<sup>(56)</sup> call for a minimum compressive strength of 350 psi at the age of 7 days, and 600 psi at 28 days. All of the clays and shales tested meet these strength specifications, and the majority of the samples gave compression values more than double the requirements. The loess mortars gave equal, or greater, strength than the lime mortars, and many of the Pennsylvanian shales in mortars approached the lime mortars in strength.

The workability of the mortars containing Pennsylvanian shales was equal to, or greater than, the mortars using lime as a plasticizer. The Porters Creek clays gave mortars which were too sticky, and the loess mortar had only poor to fair workability. The presence of organic material in the clays and shales gave no detrimental effect to the setting of the mortar.

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(56) *ibid.*



## SUMMARY

On the basis of firing behavior and other properties, the loess, lower Pleasanton shale, upper Pleasanton shale, Weston shale, Lawrence shale, and Lagonda shale could be used for the production of face brick and other structural clay products. The upper Pleasanton shale could also be used in the manufacture of paving brick and railroad ballast because of its long firing range at low porosity values.

Good or excellent bloated-shale light-weight aggregate could be prepared from the Cherryville, Chester, lower and upper Pleasanton, Weston, and Lawrence shales, while a fairly good light-weight aggregate could be produced by simply vitrifying the Porters Creek clay.

Only the Weston shale meets A.S.T.M. specifications on masonry mortar with respect to minimum water retention values. However, all the clays and shales in the investigation exceeded minimum requirements of compression strength for masonry mortars.

The correlation of physical properties of plasticity, grain size, and type of clay and accessory mineral with the ceramic properties was discussed.

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## VITA

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After attending the Kansas City Junior College, he entered the Missouri School of Mines and Metallurgy in September, 1941, and graduated in January, 1943, with the degree of Bachelor of Science in Ceramic Engineering.

On October 3, 1942, he was married to Miss Mary Elizabeth Stuck of Kansas City. A child, David Lee, was born December 9, 1943.

Immediately after graduation from the Missouri School of Mines, he entered the Army Air Forces, and was released from active duty in February, 1946, with the rank of Captain. Since that time he has pursued his studies for the degree of Master of Science in Ceramic Engineering as a student at the Missouri School of Mines and Metallurgy, Rolla, Missouri.

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