NORTH DAKOTA GEOLOGICAL SURVEY

Wilson M. Laird, State Geologist

INVESTIGATION OF LIGHTWEIGHT AGGREGATE POSSIBILITIES OF SOME NORTH DAKOTA CLAYS AND SHALES

by

Oscar E. Manz

A cooperative project
of
the North Dakota Geological Survey
and
the College of Engineering

North Dakota Geological Survey Report of Investigation No. 17

College of Engineering Departmental Bulletin No. 13

Grand Forks, North Dakota

1954

TABLE OF CONTENTS PAGE

Figures

Tables

ABSTRACT

Continuous increases in the use of concrete masonry units has imposed a problem on the suppliers of the necessary aggregates. The problem has been made even more critical through the trend toward the demand for lightweight units. In addition to the normal characteristics of concrete such as strength, durability, and water tightness, lightweight concrete has other desirable qualities such as greater insulating properties, sound absorption, and easier handling because of lightness of weight.

Up to a decade ago, the demand for lightweight aggregates was being met through the supplies of cinders from power generating stations, through the expansion of blast-furnace slag, and through localized use of natural lightweight aggregates such as volcanic cinders and pumice. These sources have been insufficient to supply the increasing demand. Within the last four years, rapid development of processes for the bloating of local clays and shales has made up part of this deficit in lightweight aggregates.

As part of a long range program of research at the University of North Dakota, preliminary tests have been run to determine the lightweight aggregate possibilities of the deposits of lower grade clays and shales throughout North Dakota which are not too well suited to the manufacture of brick, building tile, or other ceramic products. The tests performed were as follows: bloating tests in an electric furnace with subsequent determination of color, cell size and specific gravity; and differential thermal analysis to determine minerals present.

INTRODUCTION

The American Society for Testing Materials (1) a standards agency, defines lightweight aggregate for concrete as follows: "Lightweight aggregates shall consist of pumice, lava, tufa, burned clay, burned shale, suitable cinders or other strong durable particles. The unit weight of the fine aggregate (minus 3/8 inch) cannot exceed 75 lbs. per cubic foot and the weight of the coarse aggregate cannot exceed 55 lbs. per cubic foot."

Among the most important qualities that should be sought in a good lightweight aggregate are the following:

1. Lightness of weight.
2. Strength.
3. Particle shape to promote good workability.
4. Low water absorption.

5. Uniform particle size gradation.

6. Chemical inertness.

7. Low production cost.

These seven properties are discussed more fully below:

1. In order to make a concrete that will effect a worthwhile saving in weight, the lightweight aggregate should weigh approximately half of the weight of the conventional sand and gravel. Ideally, the weight should range between 40 to 60 lbs. per cubic foot.

2. Strength is of course essential and the individual particles of aggregate should be strong enough to develop the maximum efficiency of the cement. For a concrete of a given strength, a stronger aggregate will require less cement than a weaker one, thus resulting in savings in both cost and weight.

3. Absence of sharp edges and corners is preferred, for the ideal aggregate should have a well-rounded preferably spherical surface.

4. Low water absorption is desired because, when made into concrete, an aggregate with a tendency to absorb water tends to dehydrate the cement with a resulting deleterious effect upon the setting of the concrete. Pre-soaking can compensate, however, to some extent for any shortcomings the aggregate may have with respect to water absorption.

5. Size gradation is a most important requirement necessary to insure good workability, as the aggregate must be composed of a range of sizes including a sufficient quantity of fines. For good structural concrete, approximately 50% by volume should be fines, 3/8 inch and finer.

6. Chemical inertness of the aggregate is a necessity. Compounds that tend to react with the cement and effect its setting should not be present.

7. The cost of the aggregate is the ultimate factor that determines its acceptability. The initial cost per cubic yard over sand, gravel and

crushed rock aggregate must be offset by savings in weight, which reduces transportation costs and the steel requirements and permits use of lighter forms or by the attainment of better thermal and acoustical insulation properties.

Cole and Zetterstrom (2) have given the following summary of the development of lightweight aggregate and concrete:

"The country - wide search for lightweight aggregates was necessitated toward the end of the nineteenth century by the change in building design from thick, heavy, load-bearing walls to a framework of structural steel beams and columns with thin walls, followed shortly by reinforced concrete and structural concrete as materials for the erection of the load supporting frame. Ordinary concrete, with sand and gravel aggregate, weighs about 150 lbs. to the cubic foot. A lighter concrete was needed to relieve the dead load, make possible the construction of higher buildings, and the addition of stories to existing buildings.

The search for lighter-weight building materials for use in partitions, floors, and exterior walls led to extensive research in their thermal and sound insulating properties. Eventually, the lightweight aggregate concretes, comprising one group of lightweight construction materials, were developed. They were introduced first in the construction of commercial buildings and later into residential construction. Now they have a great variety of uses, the more important ones including lightweight concrete masonry units, tilt-up wall panels, precast roof and floor slabs; and lightweight structural concrete multiple-story buildings, barges, piers, and super structures of bridges, bridge decks, and jet plane runways and aprons. The lightweight structural concrete is utilized in precast, prestressed, and monolithic concrete construction. Another type of lightweight aggregate, called ultra-lightweight aggregate is used extensively for loose insulating fill, industrial insulations, plant culture plaster and stucco aggregate.

The use of lightweight concrete products has not developed to a great extent in North Dakota, and at present, its major use has been limited to the production of structural blocks. It is probable that its greatest potential for use in North Dakota is in the form of precast panels or in tilt-up panel construction.

The opening of a plant in 1953 at Mandan, North Dakota to produce lightweight aggregate from the shale of the Cannonball formation and the scheduled opening of a second plant at Noonan, North Dakota in 1955 to produce aggregate from clay occurring with lignite beds are important factors in the rapid growth of aggregate plants utilizing clay, shale or slate in this country. The sources of cinders, furnace slag and pumice have been inadequate for the ever increasing demand for lightweight aggregate. Cinder supply is actually diminishing through the change over on the part of many steam power plants from lump to powdered coal; processed slag aggregates are limited by the supply of blast furnace slag available; and natural lightweight aggregates are more or less limited in use to the western part of the United States because that is where the natural deposits are and freight rates limit the economical shipping range.

CERAMIC INDUSTRY IN NORTH DAKOTA

According to Clapp (5), the ceramic industry in North Dakota began in the 1870's with the production of common brick at Fargo. The industry spread with the development of settlements, first in the Red River Valley, and then to central and western North Dakota. By 1905, there were 18 plants in operation in the state, all producing common brick. Four plants using the yellow silt of the Lake Agassiz deposits were located near

Grand Forks, the chief center of the industry. Other plants using the yellow silt were located at Fargo, Grafton, Minto, Drayton, and Abercrombie. Only one plant, situation at Omemee, utilized the yellow silt of the Lake Souris deposits. Plants located at Hillsboro, Williston, Minot, and Mandan were using alluvial clays to manufacture common brick. Plants located in the lignite area near the towns of Burlington, Kenmare, Wilton, Richardton and Dickinson made use of clay occurring in the Fort Union formation. There were plants at Rolla and Bismarck which made brick from glacial till, while at Mayo the only plant in the state using Cretaceous shale, was in operation. Most of these plants were of a temporary character and are now only historic relics.

Light-burning Tertiary clays of the Golden Valley formation were used in the manufacture of face brick, building tile, and fire brick, with plants located at Dickinson and Hebron. Operations ceased at Dickinson in the late 30's, but the plant at Hebron is today the oldest and largest plant in operatioh in the state, and in 1952 it used 20,000 tons of clay to produce almost 10 million brick and tile.

The only other brick plant in operation in the state, at Hettinger, uses about 1500 tons of clay each year in the manufacture of brick and tile.

The University of North Dakota has maintained a ceramic department since 1910 for the purpose of teaching pottery handicraft. Only North Dakota clay is used and pieces of pottery produced in the department are sold to the public.

A small pottery at Wahpeton has been manufacturing ceramic figures, wall plaques, and other art objects since 1940, using clay from Mandan.

In the spring of 1953, a lightweight aggregate plant began operations at Mandan, producing aggregate from shale of the Cannonball formation by means of a rotary kiln. A similar plant is being constructed at Noonan, and will use clay which occurs below a lignite seam. A comparison of the operation of two rotary kilns in North Dakota, one at Mandan using natural gas for fuel and the other at Noonan using dried and powdered lignite, will be of interest to the ceramic industry.

BLOATING OF CLAYS AND SHALES

All clays and shales do not bloat or expand upon heating to elevated temperatures. Some are too refractory and others melt or slag over a very narrow temperature range, forming a dense glassy mass. Many non-bloating clays and shales can be converted to bloaters by adding proper admixtures and intimately mixing these with the raw materials before firing.

The bloating property of a material does not depend on the basic type of clay minerals present. Clays and shales usually used for making bloated products are similar in many respects to those from which brick, tile, sewer pipe, and other structural clay products are manufactured. These are the common or low-grade clays with fusibility ranging from 1850°F to about 2400°F., which expand satisfactorily on heating.

Extraneous materials, usually referred to as impurities, in a clay or shale are responsible for the bloating phenomenon. Bloating results from the generation and expansion of gas or vapor within the mass itself when the material has been heated to incipient fusion and is in a semiplastic state. The source of the gas or vapor is not always obvious. Conley, Wilson and Klinefelter (3) indicated that carbonaceous materials, various iron compounds, limestone, dolomite, and gypsum are potential sources of gases for bloating, within the temperature range of commercial bloating.

Clays become more refractory as the content of aluminum silicate increases and also with the increase of quartz or silica above certain limits. Metallic oxides, alkaline, earths (lime and magnesia), and alkalies (potassium and sodium) act as fluxes to lower the temperature of fusion when added to clays.

The origin of clays and shales has been discussed by the author (4) in a previous report on some of the clays and shales of North Dakota. A brief summary of the geological occurrence of North Dakota clays and shales is also included in the previous report by the author (4A).

The commercial bloating of clays and shales is performed either by burning them in rotary kilns or by sintering them on grates. The rotary kiln method which is used by most producers of expanded shale is the older of the two. In this process, the raw material is crushed, and then introduced at the upper end of a revolving inclined circular kiln similar to the type used in the manufacture of portland cement. In passing through the kiln, the material reaches a temperature of from 1800° to 2200°F. At this temperature range the shale begins to become plastic and internal gases cause it to expand. The desirable extent of bloating is attained by the time the shale reaches the discharge end of the kiln. The bloated material leaves the kiln to cool and is then crushed and graded.

The sintering process consists of placing a shale, clay or slate charge on a grate, which may be either of the circular or travelling horizontal type. Heat is introduced within a burning chamber either from the top or bottom. Usually, in the sintering process, a small amount of powdered coal or other fuel is added to the charge to accelerate burning, and the draft chambers over which the charge passes keep the temperatures at the fusion point.

Clays or shales with narrow bloating temperature ranges may cause trouble in rotary kilns because it is difficult to control the temperatures, and as a result agglomeration or adhesion to the kiln lining may occur. In the sintering process there is a closer control of the temperature, so any material that causes trouble in a rotary kiln would be suited for the sintering process. However, an inferior aggregate may occur in the sintering process if there is incomplete combustion of the fuel which is mixed with the clay or shale.

EXPERIMENTAL PROCEDURE

Field Methods

The samples tested at the University were collected over a period of two years with the cooperation of the College of Engineering at the University of North Dakota, the North Dakota Geological Survey, the United States Geological Survey, and the U. S. Army Corps of Engineers. The samples were designated with the prefix 52, 53, or 54 to denote the year they were gathered.

Hvorslev (6) describes rotary drilling rigs similar to the one used by the U. S. Geological Survey in their program of determining the ground water supplies of North Dakota. The Corps of Engineers drilled several holes at the site of the Grand Forks Flood Control Levee and obtained continuous, undisturbed samples of the Lake Agassiz clay unit by using a 5" fixed piston sampler. The functioning of this type of sampler and a summary of its advantages is given by Taylor (7).

Laboratory Methods

The evaluation of a clay, shale or slate as suitable material for the production of lightweight aggregate requires careful investigation of the many variables affecting the bloating of clays. If bloating of a clay or shale is feasible, then it is necessary to determine the maximum allowable expansion to give desired density and required strength of the aggregate. There is no substitute for testing concrete mixes in the evaluation of lightweight aggregate materials.

Preliminary testing in a stationary furnace requires only a small quantity of clay or shale, and facilitates the elimination of samples that are too refractory or have a short vitrification range, as well as those that are not uniform in composition. Usually an electric muffle furnace is used to provide accurate temperature control and to facilitate the testing of several samples simultaneously. Conley, Wilson, Klinefelter and others (3A) found that most clays bloated best if placed in the furnace for 5 to 15 minutes after the expected bloating temperature in the furnace had been reached.

Matthews (8) ran numerous tests in both a stationary furnace and a small test rotary kiln, and he states that if a stationary furnace produces a well bloated, dull appearing aggregate, indications are favorable for a test in a rotary kiln. He also states that the gas-fired test kiln, used for his work, produced good aggregate with a retention time of 15 minutes. When given a retention time of 30 minutes, as is common in commercial kiln, no bloating occurred. Matthews test kiln measured 5 feet long with an inside diameter of 5". Commercial kilns vary in inside diameter from 6 to 8 feet, and in length from 60 to 125 feet.

Although most commercial clay and shale aggregate plants use rotary kilns, the sintering process allows closer temperature control. If variations of the pitch and speed of a test kiln, as well as fuels and temperatures, do not produce a suitable aggregate, a small sintering machine may prove feasible.

When aggregate tests are performed with a small rotary kiln, there is usually enough bloated material to enable the determination of the bulk density, the crushing strength, and the volume expansion. The American Society for Testing Materials (10) has specifications for the determination of other properties: organic impurites, soundness, abrasion, and fineness modulus.

Bloated clay or shale aggregate, when used in concrete mixes, must meet certain specifications based on the following properties: compressive strength, unit weight, shrinkage, absorption, freezing and thawing, sound and heat insulation.

The investigation of several clays and shales included in this report was done as a preliminary survey of the ceramic lightweight aggregate possibilities in North Dakota. Laboratory tests, performed with limited facilities, have been supplemented with information obtained from aggregate plants in operation in North and South Dakota.

Equipment available at the University made it possible to adopt a procedure similar to that used by Cole and Zetterstrom (2B) for their Investigation of Lightweight Aggregates of North and South Dakota.

On the following pages is an outline of the laboratory procedure followed while testing the clays and shales included in this report.

1. Representative portions of the samples were sized to minus 3 mesh plus 4 mesh so as to provide 200 to 300 grams of material approximating rotary kiln feed. To prevent popping of the clay or shale, when placed into the furnace at temperatures above 1800°F, the test portions were preheated to 1000°F. Tests by Conley and Klinefelter (3B) have shown that this treatment does not lessen the bloating qualities of the clay or shale.

2. For the bloating tests in the Harper Electric Globar Furnace, insulation brick were fitted into the opening provided for the door, and a 3 inch square opening was left for a removable firebrick plug to be

inserted. Small trays made of 20 gauge stainless steel, 1-3/4 by $2\frac{1}{4}$ by $\frac{1}{2}$ inch, were used for bloating tests of small portions of the samples, containing 25 to 35 pieces of the preheated, minus 3 plus 4 mesh material. A metal holder was constructed to place the trays into the furnace through the 3 inch hole provided in the insulation brick door. When the furnace had reached the desired temperature for the first bloating test, usually 1850°F, 2 trays were placed on the metal holder and pushed into the furnace. The samples were retained in the furnace for 5 minutes with the temperature held constant. Additional preheated samples were used for each trial as the temperature was increased by increments of 50°F. Since the range of most light weight aggregate rotary kilns is from 1800 to 2200°F, a maximum of 2250° was used in the bloating tests for this report. After cooling, those samples that had not fused, were easily removed from the trays.

3. The color, cell size, and specific gravity of the fired samples were determined. For color, the standard Munsell chart was used. Specific gravity was determined by floats tests in the following solutions of known specific gravity: pure carbon tetrachloride (specific gravity 1.6); mixture containing 60% carbon tetrachloride and 40% benzene (specific gravity 1.3); water (specific gravity 1.0); and ethyl alcohol (specific gravity 0.8). The unfired clay and shale samples have approximately 1.8 - 1.9 specific gravity. Any samples that barely floated in the 1.6 mix had bloated slightly. The 1.3 mix was used to determine the lower end of the bloating range, and the specific gravity indicated by slight sticking together of the particles was considered the upper limit. The appearance of the fired materials was noted and recorded as angular or rounded, and dull or fused. The bloated particles were fractured and the average

size cell of several pieces determined. Cells averaging 1 millimeter in diameter were designated as "large"; those averaging about ½ millimeter were termed "medium"; and those termed "small" were practically invisible to the naked eye.

4. Small portions of several of the clay and shale samples (dried to room temperature) were ground to pass a 100 mesh sieve. A 3-4 gram portion was used for a differential thermal analysis which aids in indentifying clay minerals and other non-clay minerals by comparison of characteristic curves resulting from endothermic and exothermic reactions which occur during heating of the minerals.

5. Although the presence of carbonates would be detected by the differential thermal analysis, some of the samples were tested with hydrochloric acid to detect any effervescence caused by carbonates.

DISCUSSION OF CLAYS STUDIED

Fortunately, the samples of clay and shale can be grouped into three main divisions with the members of each respective group occurring in the same geological formation. These groups will be discussed in detail on the following pages.

General Discussion

Most of the clays and shales included in this report are plastic and fine grained, and range in color from greenish gray to medium gray. The bloating tests performed on most of the samples produced suitable aggregate within the commercial temperature range of rotary kilns. The bloated materials are mostly dull, angular to rounded, and a light brown color.

Group 1

Samples: 53-38, 54-27, 54-28, 54-29, 54-30, 54-50, 54-51, 54-52, 54-53, 54-54.

The field samples of Group 1 consist of Lake Agassiz deposits with the following characteristics:

1. Medium gray to dark greenish gray, sticky, plastic clay of the Lake Agassiz "clay unit".

2. Yellow, sandy clay of the "silt unit".

Referring to Table 3, page 20, it is noted that all the members of Group 1 contain montmorillonite as the basic clay mineral. The samples of the "silt unit" contain carbonate but no organic material, whereas, the "clay unit" samples contain organic material and produce only slight indications of the presence of carbonates. Samples of the "silt unit" produce effervescence when tested with hydrochloric acid, whereas, samples of the "clay unit" produce only a slight effervescence. Differential thermal analysis curves for Group 1 are shown in Fig. 2.

The samples of the "clay unit" are fine grained and non-stratified. Undisturbed samples obtained from the U. S. Army Corps of Engineers indicate that the clay unit has very high liquid limit and natural water content. Dried test cores from the Corps of Engineers produced excessive shrinkage and warping, with conchoidal fracture. Considerable pressure was required to break down lumps of the dried material with a pestle.

The samples tested were from Fargo, Neche, Grand Forks, and intermediate points, and indicate that the thickness of the silt unit ranges from 20 to 35 feet, and the clay unit from 40 to 75 feet. From Fargo to Neche is a distance of about 150 miles, and includes the major portion of the Red River Valley. Matthews (8A) tested plastic clay of the Lake Agassiz deposits which were sampled at Winnipeg, Manitoba. Near the edge of the Red River Valley, on the bank of Kelly's Slough, samples 54-30, of medium gray, plastic clay occurs less than 5 feet below the surface. Rominger and Ruttledge (9) state that at Crookston, Minnesota, there is approximately 50 feet of plastic clay occurring below 20 feet of silt.

Table 1 pages 17-18, gives the bloating ranges and cell sizes for the bloated pieces for Group 1. All the samples of Group 1 bloated within the temperature range 1,850° to 2,100°F, and most of the samples had a bloating range of 100° to 150°F. The following samples had bloating ranges of 50°F or less: 53-38; the silt portions of samples 54-27 and 54-28; the 110 to 140 foot portion of sample 54-28; and the 55 to 99 foot portion of sample 54-53. The cell size of the bloated pieces ranged from small, at the lower end of the bloating range, to large at the upper end.

As shown in Table 2, page 19, the specific gravity at the upper limit of the bloating range for most of the samples was 0.8. In most instances, those bloated pieces with specific gravities of 0.8 or less, were quite easily crushed between the fingers.

Table 1

Bloating Range, Outward Appearance and Cell Size

Group 1 (Lake Agassiz Deposits)

Sample No.	Outward Appearance	Bloating Range (°F)	Cell Size
53-38	Grayish orange to pale brown, dull.	2100	Medium
-A- 54-27 (10-15 ft.) (silt unit)	Dark yellowish brown, dull slightly rounded.	2100	Med. to large
-B- 54-27 (15-20 ft.) (Silt unit)	Light brown to pale brown, dull, slightly rounded.	2,000-2,100	Small to med.
-C- 54-27 (20-25 ft.)	Light brown to pale yellowish brown, dull, angular to slightly rounded.	1,950-2,050	Small to large
-D- 54-27 (25-40 ft.)	Light brown to pale brown, dull, angular to rounded.	1,900-2,050	Small to large
-E- 54-27 (40-50 ft.)	Light brown to grayish orange, dull, angular to rounded.	1,900-2,000	Small to large
54-27 (Blend of 10-50 ft.)	Mostly light brown, some grayish orange, dull, angular to rounded.	1,900-2,050	Small to large
54-28 (0-30 ft.) (silt unit)	Light brown, dull, slightly rounded.	2,050-2,100	Small to large
-A- 54-28 (30-40 ft.)	Light brown to grayish orange, dull, slightly angular to rounded.	1,900-2,000	Small to med.
-B- 54-28 (40-50 ft.)	Light brown, dull, rounded.	1,900-2,000	Small to med.
-C- 54-28 (60-70 ft.)	Light brown, dull, angular.	1,900-2,000	Small to large
54-28 (110-120 ft)	Light brown to grayish orange, dull, angular to slightly rounded.	1,950-2,000	Small to med.
54-28 (130-140 ft)	Light brown to grayish orange, dull, slightly rounded to rounded.	1,950-2,000	Small
54-28 (150-160 ft)	Light brown to grayish orange, dull, angular.	2,000-2,050	Small to med.

Bloating Range, Outward Appearance, Cell Size

Group 1 (Lake Agassiz Deposits)

Sample	Outward Appearance	Bloating Range (°F)	Cell Size
54-29		approx. 2,075	
54-30	Moderate reddish orange to moderate yellowish brown, dull, rounded.	1,850-2,000	Small to large
54-50 (P3) (30-33 ft)	Light brown to moderate reddish orange, dull, rounded.	1,850-2,000	Small to large
54-50 (P7) (40-43 ft)	Light brown, dull, rounded.	1,900-2,000	Small to large
54-51 (44 ft)	Light brown to grayish orange, dull, angular to rounded.	1,900-2,050	Small to large
54-52	Light brown to grayish orange, dull, slightly rounded.	2,000-2,100	Small to large
54-53 (20-55 ft)	Light brown, dull, rounded.	1,900-2,050	Small to large
54-53 (55-90 ft)	Light brown, dull, few pieces rounded.	2,000-2,050	Small to large
54-54 (35-55 Ft)	Light brown to grayish orange to pale brown, dull, slightly rounded.	1,950-2,100	Small to large
54-54 (55-75 ft)	Pale yellowish brown to grayish orange, dull, slightly rounded.	1,950-2,050	Small to large

* Light brown refers to Munsell color No. 5YR6/4

Table 2

Approximate Specific Gravity Values

Group 1 (Lake Agassiz Deposits)

Sample No.	1,850°F	1,900°F	1,950°F	2,000°F	2,050°F	2,100°F	2,150°F
53-38				1.6	1.3-1.6	0.8-1.6	0.8(1)
54-27 (10-15)A			1.6	1.6	1.3-1.6	1.0-1.3	
54-27 (15-20)B			1.3-1.6	1.0-1.6	0.8-1.0	0.8-1.0	
54-27 (20-25)C			1.0-1.3	1.0-1.3	0.8-1.0	0.8 (1)	
54-27 (25-40)D		1.0-1.6	1.0-1.3	0.8-1.3	0.8		
54-27 (40-50)E		1.0-1.6	0.8-1.0	0.8	0.8 (1)		
54-27 (Blend)		0.8-1.6	0.8-1.3	0.8-1.3	0.8-1.3	0.8 (1)	
54-28 (0-30)		1.6	1.6	1.3-1.6	0.8-1.6	0.8-1.0	
54-28 (30-40)A		1.0-1.3	0.8-1.3	0.8	0.8 (1)		
54-28 (40-50)B		1.0-1.3	0.8-1.3	0.8	0.8 (1)		
54-28 (60-70)C		1.0-1.3	0.8-1.3	0.8	0.8 (1)		
54-28 (110-120)		1.3-1.6	0.8-1.3	0.8-1.0	0.8 (1)		
54-28 (130-140)		1.3-1.6	1.0-1.6	0.8-1.3	0.8 (1)		
54-28 (150-160)		1.6	1.3-1.6	0.8-1.6	0.8-1.6	0.8-1.3 (1)	
54-29					1.3-1.6	0.8-1.0 (1)	
54-30	0.8-1.3	0.8	0.8	0.8	0.8 (1)		
54-50 (P3)	1.0	0.8-1.0	0.8	0.8	0.8 (1)		
54-50 (P7)		1.0-1.3	0.8-1.0	0.8	0.8 (1)		
54-51		1.0-1.6	0.8-1.6	0.8-1.3	0.8-1.3	0.8-1.0 (1)	
54-52 (30-70)		1.6	1.3-1.6	1.3-1.6	0.8-1.6	0.8-1.6	
54-53 (20-55)		1.0-1.6	1.0-1.6	0.8-1.3	0.8-1.0	0.8 (1)	
54-53 (55-90)		1.3-1.6	1.3-1.6	0.8-1.6	0.8-1.3	0.8-1.0 (1)	
54-54 (35-55)		1.3-1.6	0.8-1.6	0.8-1.6	0.8-1.6	0.8-1.6	
54-54 (55-75)		1.3-1.6	1.0-1.6	0.8-1.6	0.8-1.6	0.8-1.0 (1)	

(1) Denotes temperatures above the bloating range.

Table 3

Results of Differential Thermal Analyses and Hyrochloric Acid Tests

(Lake Agassiz Deposits)
Group 1

Sample No.	Type of Reaction	Temperature Range (^{o}F)	Minerals Present	Acid Test
54-27 A	Endothermic	100- 500	Montmorillonite	Effervescence
	Endothermic	1400-1600	Carbonate	
54-27 B	Endothermic	100- 500	Montmorillonite	Effervescence
	Endothermic	900-1400	Montmorillonite	
	Endothermic	1400-1550	Carbonate	
54-27 C	Endothermic	100- 700	Montmorillonite	Some effervescence
	Endothermic	900-1200	Montmorillonite	
	Endothermic	1450-1550	Carbonate	
54-27 D	Endothermic	100- 500	Montmorillonite	Slight effervescence
	Exothermic	500- 700	Organic	
	Endothermic	900-1200	Montmorillonite	
	Endothermic	1400-1550	Carbonate (slight)	
54-27 E	Endothermic	100- 500	Montmorillonite	Slight effervescence
	Exothermic	500- 900	Organic	
	Endothermic	900-1200	Montmorillonite	
	Endothermic	1400-1450	Carbonate (slight)	
54-28 A	Endothermic	100- 500	Montmorillonite	Slight effervescence
	Exothermic	500- 900	Organic	
	Endothermic	1400-1500	Carbonate (slight)	
54-28 B	Endothermic	100- 500	Montmorillonite	Slight effervescence
	Exothermic	500- 900	Organic	
	Endothermic	1400-1500	Carbonate (slight)	
54-28 C	Endothermic	100- 500	Montmorillonite	Slight effervescence
	Exothermic	500- 900	Organic	
	Endothermic	900-1400	Montmorillonite	
54-30	Endothermic	100- 600	Montmorillonite	
	Exothermic	600-1000	Organic (slight)	
	Endothermic	1000-1400	Montmorillonite	
54-50 (P3)	Endothermic	100- 500	Montmorillonite	Slight effervescence
	Exothermic	500- 900	Organic (slight)	
	Endothermic	900-1600	Montmorillonite	
54-50 (P7)	Endothermic	100- 500	Montmorillonite	Very slight effervescence
	Exothermic	600- 900	Organic	
	Endothermic	900-1600	Montmorillonite	
54-51	Endothermic	100- 600	Montmorillonite	Effervescence
	Exothermic	600- 900	Organic	
	Endothermic	1500-1600	Carbonate (Sharp)	

Group 2

Samples: 53-25, 53-28, 53-29, 53-31, 54-1, 54-2, 54-3, 54-18, 54-24, 54-33, 54-38.

The field samples of this group occur in the Tongue River member and have the following characteristics:

1. All greenish gray in color except sample 54-18 which is brown.
2. All plastic except sample 54-38 which is sandy.
3. Occurring in consolidated lumps.

As shown in Fig. 3, and in Table 6, page 24, the following samples contain montmorillonite: 53-28, 53-29, 54-1, 54-24, 54-38. Sample 54-18 contains considerable organic material.

Referring to Table 4, page 22, it is observed that all the members of Group 2 bloated with the exception of samples 54-2, 54-3, & 54-38. The bloating ranges occurred between 1,950 and 2,200°F and varied from 50 to 100°F, except for sample 54-33 which had a range less than 50°F. There was little variation in cell size for the different samples between the lower and upper limits of the bloating ranges. Most of the cells were small, with the exception of the medium or large cells which occurred at the upper end of the bloating ranges of the following samples: 53-25, 54-1, 54-18, and 54-24.

As noticed for Group 1, and as shown in Table 5, page 23, the specific gravity at the upper limit of the bloating range for most of the samples is 0.8. The bloated pieces with specific gravities of 0.8 or less are also quite easily crushed between the fingers.

Table 4

Bloating Range, Outward Appearance, Cell Size

Tongue River Member

Group 2

Sample No.	Outward Appearance	Bloating Range (°F)	Cell Size
53-25	Grayish orange to moderate yellowish brown, dull, rounded.	2,000-2,100	Small to medium.
53-28	Pale yellowish orange, dull, rounded.	2,050-2,100	Small.
53-29	Pale brown to moderate brown, dull, rounded.	2,050-2,150	Small.
53-31	Dark, yellowish brown to pale brown, dull, rounded.	2,150-2,200	Small.
54-1	Light brown to pale brown, dull, angular to rounded.	1,950-2,050	Small to medium.
54-2	Very pale orange, dull, angular.	------------	
54-3	White, dull, angular.	-------------	
54-18	Light brown, dull, rounded.	1,950-2,050	Small to large.
54-24	Light brown to dark yellowish brown, dull, slightly rounded to rounded.	2,050-2,150	Small to medium.
54-33	Grayish brown to very dusky red, some glassy, slightly rounded.	2,150	Small.
54-38	Light brown, dull, angular	--------------	

* Light brown refers to Munsell color No. 5YR6/4.

Table 5

Approximate Specific Gravity Values

Tongue River Member

Group 2

Sample No.	1,950°F	2,000°F	2,050°F	2,100°F	2,150°F	2,200°F	2,250°F
53-25		0.8-1.0	0.8	0.8	0.8 (1)		
53-28	1.3-1.6	1.3-1.6	1.0-1.3	1.0	0.8 (1)		
53-29		1.3-1.6	1.0-1.3	0.8-0.9	0.7-0.9	0.7 (1)	
53-31		1.6	1.6	1.6	1.0-1.6	1.0-1.3	0.8-0.9(1)
54-1	1.3	1.0	0.8	0.8 (1)			
54-2		1.6	1.6	1.6	1.6		
54-3		1.6	1.6	1.6	1.6		
54-18	1.2	1.0	0.8	0.8 (1)			
54-24	1.3-1.6	1.3-1.6	1.0-1.6	1.0-1.3	0.8-1.0		
54-33			1.6	1.6	1.0-1.3		
54-38		1.6	1.3-1.6	0.8-1.6	0.8 (1)		

(1) Denotes temperatures above the bloating range.

Table 6

Results of Differential Thermal Analyses

Tongue River Member

Group 2

Sample No.	Type of Reaction	Temperature Range (°F)	Minerals Present
53-28	Endothermic	100- 300	Montmorillonite
	Exothermic	300- 900	Organic (slight)
	Endothermic	1000-1500	Montmorillonite
53-29	Endothermic	100- 400	Montmorillonite
	Endothermic	800-1200	Montmorillonite
54-1	Endothermic	100- 300	Montmorillonite
	Endothermic	1000-1400	Montmorillonite
54-18	Exothermic	400- 900	Organic
54-24	Endothermic	100- 300	Montmorillonite
	Endothermic	900-1400	Montmorillonite
54-38	Endothermic	100- 500	Montmorillonite
	Endothermic	1200-1550	Carbonate (slight)

Table 7

Bloating Range, Outward Appearance, Cell Size

Cannonball Formation

Group 3

Sample No.	Outward Appearance	Bloating Range (°F)	Cell Size
52-17	Pale brown to grayish orange, dull, rounded.	2,050	Small to large.
52-18	Pale brown, dull, slightly rounded.	2,100	Small.
53-9	Pale brown to dark, yellowish brown, slightly glossy, rounded.	2,200	Medium.
53-14	Dark, yellowish brown, dull, round to slightly stuck.	2,050-2,100	Medium.
53-15	Moderate brown, dull, rounded, slight sticking.	2,050-2,100	Medium to large.
53-17	Dull, angular.	-----------	
53-18	Pale brown, dull, slightly rounded.	Approx, 2,125	Small.
53-19	Pale brown to grayish brown, dull, rounded.	2,050-2,100	Medium to large.

Table 8

Approximate Specific Gravity Values

Cannonball Formation

Group 3

Sample No.	2,000°F	2,050°F	2,100°F	2,150°F	2,200°F	2,250°F
52-17		0.8-1.6	0.8 (1)			
52-18		1.6	1.3-1.6	0.8-1.0		
53-9	1.6	1.6	1.6	1.6	1.3-1.6	1.0-1.1 (1)
53-14	1.6	1.0-1.3	0.8-1.3	0.8 (1)		
53-15	1.3-1.6	1.0-1.3		0.8 (1)		
53-17		1.6	1.6	1.3-1.6		
53-18		1.6	1.3-1.6	1.0-1.3 (1)		
53-19	1.5	0.8-1.6	0.8	0.8 (1)		

(1) Denotes temperatures above bloating range.

Table 9

Results of Differential Thermal Analyses

Cannonball Formation

Group 3

Sample No.	Type of Reaction	Temperature Range (^{o}F)	Minerals Present
53-9	Endothermic	100- 500	Montmorillonite
	Exothermic	850- 950	Pyrite or Marcasite
53-14	Endothermic	100- 600	Montmorillonite
	Endothermic	1450-1600	Carbonate
53-19	Endothermic	100- 600	Montmorillonite
	Endothermic	900-1300	Montmorillonite
	Endothermic	1450-1550	Carbonate
53-15	Endothermic	100- 600	Montmorillonite
	Endothermic	1400-1550	Carbonate

Miscellaneous Samples

Samples: 52-1, 52-2, 52-3, 52-6, 54-12, 54-16, 54-19.

Sample 52-1, as received from the field, is a grayish blue shale; sample 52-2 is a light cream colored, sandy clay; sample 52-3 is light brown, bentonitic clay with yellow staining and is badly slaked; sample 52-6 is a yellow brown and rather silty clay; sample 54-12 is dark gray, very hard shale; sample 54-17 is medium gray, very hard shale; sample 54-19 is dark gray shale.

The following samples bloated within the temperature range 1,950 to 2,150°F: 52-1, 52-3, and 54-19. Samples 52-2, 52-6, 54-12 and 54-16 did not bloat within the commercial bloating temperature range. Sample 54-19 has a bloating range of 150°F, whereas samples 52-1, and 52-3 have a range of 50°F. The cells of the bloated pieces of sample 52-1 are small to large; those for sample 52-3 are small; and those for sample 54-19 are mostly small except for the occurrence of medium cells at the upper limit of the bloating range. See Table 10, page 30.

For the samples that bloated, it is noted from Table 11, page 31 that the specific gravity of the pieces at the upper limit of the bloating range is approximately 0.8.

As shown in Fig. 3 and in Table 12, page 32, the following samples contain montmorillonite: 54-12, 54-16, and 54-19. Sample 52-1 contains a considerable amount of organic material.

Table 10

Bloating Range, Outward Appearance, Cell Size

Miscellaneous Samples

Sample No.	Outward Appearance	Bloating Range (^{o}F)	Cell Size
52-1	Pale brown, slight gloss, slightly stuck, angular.	2,100-2,150	Small to large.
52-2	Moderate brown, dull, angular.	------------	
52-3	Moderate brown, dull, angular.	2,050-2,100	Small.
52-6		------------	Small.
54-12	Angular, dull.	------------	Small.
54-16	Moderate reddish brown, angular, dull.	------------	Small.
54-19	Light brown, dull, rounded.	1,950-2,100	Small to medium.

Table 11

Approximate Specific Gravity Values

Miscellaneous Samples

Sample No.	1,950°F	2,000°F	2,050°F	2,100°F	2,150°F	2,200°F	2,250°F
52-1		1.6	1.3-1.6	1.0-1.6	0.8-1.3	0.8 (1)	
52-2		1.6	1.6	1.6	1.6		
52-3		1.3-1.6	1.1-1.4	1.0-1.1	0.9-1.0 (1)		
52-6		1.6	1.6	1.6	1.3-1.6		
54-12		1.6	1.6	1.6	1.6		
54-17			1.6	1.6	1.6		
54-19	1.2-1.3	1.2-1.3	1.0	0.8	0.8 (1)		

(1) Denotes temperatures above the bloating range.

Table 12

Results of Differential Thermal Analyses

Miscellaneous Samples

Sample No.	Type of Reaction	Temperature Range (°F)	Minerals Present
52-1	Exothermic	300-1000	Organic
54-12	Endothermic	100- 600	Montmorillonite
	Endothermic	1200-1650	Carbonate
54-16	Endothermic	100- 400	Montmorillonite
54-19	Endothermic	100- 500	Montmorillonite
	Exothermic	500- 900	Organic
	Endothermic	900-1400	Montmorillonite

CONCLUSIONS

1. Judging from the results of this investigation, there are extensive and widespread deposits of clay and shale in the state which are suitable for the production of lightweight aggregate. Although this investigation was only preliminary, the establishment of two lightweight aggregate plants in the state since 1953 is conclusive evidence of the suitability of some of the clay and shale deposits for production of lightweight aggregate.

2. The gray colored clay samples of Group 1, although they are from widely separated portions of the Red River Valley, produced excellent aggregate, as shown by the color, bloating range, cell size, and specific gravity. Due to the 100 to 150°F bloating ranges occurring within the temperature range of 1,850 to 2,100°F, indications are favorable for rotary kiln tests where agglomeration is undesirable and the cost of fuel is a major item. The gray clay in the Red River Valley lies under 20 to 35 feet of yellow, silty clay which has been used in former years for common brick and building tile. Referring to Table 1, page 17, it is noted that a blend of 10 feet of silt and 30 feet of the gray clay produced suitable aggregate. To reduce the cost of removal of overburden it would be feasible to mix a portion of the yellow silt with the clay below. The high natural water content and resulting sticky nature of the gray clay would present a problem in the production of lightweight aggregate.

3. The clays of Group 2 produced aggregate comparable to that from Group 1 clays, but the shorter bloating ranges of Group 2 clays of from 50 to 100°F, and occurring within the temperature range of 1,950 to 2,200°F would indicate that closer control would have to be exercised in rotary kiln tests. The utilization of clay occurring with lignite beds and

with excessive overburden would be uneconomical unless the removal of overburden can be charged to lignite operations. The use of powdered lignite as fuel will further reduce the cost of producing lightweight aggregate from such deposits.

4. The bloating ranges of 50°F or less for the Group 3 shales indicate that very careful control would be required for rotary kiln tests. A lightweight aggregate plant has been producing aggregate successfully since 1953 from the same deposit where samples 52-17, 52-18 and 53-9 were obtained.

5. The following properties of the bloated pieces of sample 54-19 have good correlation with the properties of this same material in a commercial plant: the light brown color, the dull rounded pieces, and the bloating range of 150°F. Sample 52-1 would probably produce severe agglomeration in a rotary kiln; sample 52-3 will require close control. The addition of admixtures to samples 52-2, 52-6, 54-12 and 54-17 might cause them to bloat.

Recommendations For Further Research

1. Since most of the clays and shales in this report produced well bloated, dull-appearing aggregate, it is suggested that larger scale tests be performed in a rotary test kiln. This would eliminate those clays that would give trouble from agglomeration and would provide enough material to do the following tests:

    A. Bulk density.

    B. Crushing strength.

    C. Volume expansion.

    D. Organic impurities; soundness, abrasion; fineness modulus.

2. If the aggregate meets standard specifications, then the following tedts with concrete mixes should be done.

    A. Compressive strength.

    B. Unit weight.

    C. Shrinkage.

    D. Absorption.

    E. Freezing and thawing.

    F. Sound and Heat Insulation.

3. A careful study should be made of the many uses to which lightweight aggregate can be adapted and should be a part of the evaluation of clay or shale as suitable material for lightweight aggregate.

Appendix 1

LOCATION AND DESCRIPTION OF SAMPLES TESTED AT THE UNIVERSITY

Sample No. 52-1

North Roosevelt Park area.

Section 27, T. 148 N., R. 99 W., McKenzie County.

Outcrop of blue, bentonitic shale, 0.7 miles north of the entrance to the park, adjacent to Highway #85 and on the east side of the road. There is 20 feet of the blue shale exposed, with 5 feet of carbonaceous shale above.

Samples No. 52-2 and 52-3

Dickinson area.

Section 23, T. 138 N., R. 98 W., Stark County.

A small outcrop in the Little Bad Lands near Dickinson. The outcrop is 15 feet high with 12 feet of light cream colored sandy clay at the bottom and with 3 feet of light brown, bentonitic clay at the top. Sample 52-2 is the cream colored clay and 52-3 is the brown clay.

Sample 52-6

Hettinger area.

NW$\frac{1}{4}$ Section 1, T. 129 N., R. 96 W., Adams County.

Hettinger Brick Plant, 1 mile north of Hettinger. From deposit used for manufacture of common brick. Clay sampled is yellow brown.

Samples No. 52-17 and 52-18

Mandan area.

NE$\frac{1}{4}$ Section 32, T. 139 N., R. 81 W., Morton County.

Marine shale of the Cannonball formation exposed in a cut west of Mandan and south of the No. 10 Highway and N. P. Railroad. The Molite, Inc. Lightweight Aggregate Plant is located at the base of the cut. Sample 52-17 is from a seam of 10 feet of dark, greenish gray to black shale and 52-18 is from a 10 foot seam which is above the seam containing 52-17 and is separated from it by 10 feet of poorer shale.

Sample No. 53-9

Mandan area.

NE$\frac{1}{4}$ Section 32, T. 139 N., R. 81 W., Morton County.

Marine shale of the Cannonball formation used by the Molite, Inc. Plant. From the same cut where samples 52-17 and 52-18 were taken, but it is a representative sample of 20 to 25 feet of the cut.

Sample 53-14

Mandan area.

SW$\frac{1}{4}$ Section 29, T. 139 N., R. 81 W., Morton County.

North side of the Heart River, directly across from the Molite, Inc. Plant. Exposure of 10 feet of olive gray shale of the Cannonball formation, with yellow streaks. Representative sample taken of 6 feet of the seam. There is 3 to 25 feet of overburden.

Sample 53-15

Mandan area.

SW$\frac{1}{4}$ Section 34, T. 139 N., R. 81 W., Morton County.

Marine shale of the Cannonball formation, south of Mandan near the old Fort Lincoln Highway and SW of a bridge. Outcrop of 6 feet of olive gray shale with yellow streaks, exposed where water has washed slumped material away and formed a narrow channel.

Samples No. 53-17, 53-18, and 53-19

Mandan area.

NE$\frac{1}{4}$ Section 27, T. 139 N., R. 81 W., Morton County.

Marine shale of the Cannonball formation on north side of Mandan in a very prominent cut. Sample 53-17 is from the east side of the cut in a 20 foot seam of olive gray, sandy shale, with yellow streaks. Sample 53-18 is below 53-17 in a 10 foot seam of medium olive gray shale. Sample 53-19 is on the south side of the cut in the same seam as 53-18.

Sample No. 53-25

Williston area.

SW$\frac{1}{4}$ Section 11, T. 154 N., R. 100 W., Williams County.

A 2 foot seam of very plastic, greenish gray shale between 2 lignite seams.

Located on the old Lovejoy Mine property.

Sample No. 53-28

Beulah area.

SW$\frac{1}{4}$ Section 9, T. 143 N., R. 87 W. Oliver County.

Exposure of 5 feet of gray, plastic clay with up to 20 feet of overburden.

Located 1$\frac{1}{2}$ miles south east of farm of Adolf Raszler.

Sample 53-29.

Richardton area.

NW$\frac{1}{4}$ Section 32, T. 138 N., R 92 W., Stark County.

Exposure on west side of No. 8 Highway, 10.5 miles south of No. 10 Highway.

A spring has washed away the slumped material to expose a 6 foot seam of greenish gray, plastic clay, which underlies a foot of lignite and 8 feet of overburden.

Sample 53-31

Buelah area.

NE$\frac{1}{4}$ Section 11, T. 143 N., R. 88 W., Mercer County.

A 4 foot seam of greenish gray, plastic clay occurring above a lignite seam, in a cut on the road allowance on the west side of No. 49 Highway, 2.5 miles south of No. 7 Highway.

Sample No. 53-38

Emerado area.

NE$\frac{1}{4}$ Section 14, T. 152 N., R. 52 W., Grand Forks County.

A plastic, medium gray clay mixed with limonite and having 2 feet of overburden.

Taken from the bank of Kelly's Slough, south of the N. P. Railroad and about 20 yards west of a road running south from school No. 22.

Sample No. 54-1

Marmarth area.

SE$\frac{1}{4}$ Section 18, T. 132 N., R. 104 W., Bowman County.

Outcrop in butte, 10 miles south east of bridge over Little Missouri at Marmarth. Sample from a 3 foot seam of greenish gray, plastic shale with 20 to 30 feet of sandy overburden. Located on the south side of the butte, 20 yards from the center line of Highway No. 12.

Sample No. 54-2

Dickinson area.

NE$\frac{1}{4}$ Section 12, T. 136 N., R. 97 W., Hettinger County.

Exposed in cut bank on west side of Highway No. 22, 20 miles south of a bridge at Dickinson on Highway No. 22. Sample is from a 6 foot seam of greenish gray, plastic clay, with 10 feet of overburden.

Sample No. 54-3

Dickinson area.

SE$\frac{1}{4}$ Section 28, T. 139 N., R. 96 W., Stark County.

An exposure of 10 feet of greenish gray, plastic clay, located 4 miles south of a bridge on Highway No. 22 over the Heart River at Dickinson. Exposed in a cut bank on the west side of the Highway, 10 yards from the center line.

Sample No. 54-12

Concrete area.

NE$\frac{1}{4}$ Section 25, T. 161 N., R. 57 W., Cavalier County.

A 30 foot seam of dark gray, very hard shale of the Niobrara formation, exposed where a small stream has washed away slumped material. A cement plant was formerly located adjacent to this exposure.

Sample No. 54-16

Milton area.

NE$\frac{1}{4}$ Section 4, T. 159 N., R. 57 W., Cavalier County.

Shale of the Pierre formation, exposed in a ravine 200 yards south of No. 29 Highway, and 6.7 miles west of No. 32 Highway. There is 15 feet of Pierre shale exposed, with 6 feet of overburden.

Sample No. 54-18

Bowman area.

Section 2, T. 131 N., R. 102 W., Bowman County.

A chocolate brown, very plastic clay from a mound which is 60 feet in diameter and 15-18 feet high, with nearly perpendicular sides. The mound rests on a ridge which is about $\frac{1}{2}$ mile long and has a 10 foot seam of clay similar to that in the mound. There is practically no overburden on the mound.

Sample No. 54-19

Rapid City, South Dakota area.

Dark gray shale from the Lower Virgin Creek member of the Pierre shale formation, located in the pit of the Light Aggregates, Inc., 8 miles east of Rapid City, S.D. on U.S. Highway 14 and 1$\frac{1}{2}$ miles south. The 20 foot exposure is near the top of a knoll with 3 to 15 feet of overburden.

Sample No. 54-24

Noonan area.

Section 10, T. 162 N., R. 95 W., Divide County.

Greenish gray, plastic clay from Baukol Noonan, Inc. mine S.E. of Noonan, N.D. The sample is from a 8 foot layer occurring below a 7 foot seam of lignite and 50 feet of overburden. An area 100 feet wide and $\frac{1}{4}$ mile long has been stripped and the clay occurring in this area will be used to produce aggregate in the plant being constructed by Baukol Noonan, Inc.

Sample No. 54-27 (A-E)

Grand Forks area.

SE$\frac{1}{4}$ Section 5, T. 151 N., R. 50 W., Grand Forks County.

A State Water Conservation Commission drilling rig was used to obtain samples of the Lake Agassiz deposits at the campus of the University of North Dakota. An 8" hole was drilled to a depth of 50 feet, about 100 yards east of the east wall of the stadium. The description and depth of the samples taken, is as follows:

10-15 feet (A) - yellow, sandy clay with iron stains.

15-20 feet (B) - yellow, sandy clay, iron stains.

20-25 feet (C) - yellow, sandy, dark greenish gray, plastic mixed.

25-40 feet (D) - dark greenish gray, sticky plastic clay.

40-50 feet (E) - dark greenish gray, sticky plastic clay.

Sample No. 54-28 (A-C)

Neche area.

NW$\frac{1}{4}$ Section 31, T. 164 N., R. 53 W., Pembina County.

The USGS drilling crew took the following samples from a test hole in the Red River Valley:

0-30 feet - yellow, sandy clay.

30-40 feet (A) - dark greenish gray, sticky, plastic clay.

40-50 feet (B) - " " " " " "

50-60 feet (C) - " " " " " "

110-120 feet - " " " " " "

130-140 feet - " " " " " "

150-160 feet - " " " " " "

Sample No. 54-29 and 54-30

Emerado area.

SW$\frac{1}{4}$ Section 13, T. 152 N., R. 52 W., Grand Forks County.

Lake Agassiz deposit on south bank of Kelly's slough, $\frac{1}{4}$ mile south of the N.P. Railroad crossing, and 100 feet east of the center line of the road. A gully has been cut by runoff and provided an excellent spot for digging. There is one foot of overburden below which is one foot of laminated clay consisting of yellow sandy clay, medium gray clay and iron oxide staining. Below the laminated clay is sticky, medium gray clay which extends to an unknown depth below the one foot that was dug. Sample 54-29 is the laminated clay and 54-30 is the medium gray below.

Sample No. 54-33

Noonan area.

Section 8, T. 162 N., R. 95 W., Divide County.

Very plastic, greenish gray clay from the west end of the Hought-Tylor Pit of the Baukol Noonan, Inc. mine. Sample taken from an 8 foot layer occurring below the lignite and located 2 miles west of the location of sample 54-24.

Sample No. 54-38

Noonan area.

Section 10, T. 162 N., R. 95 W., Divide County.

Greenish gray, sandy clay from Baukol Noonan mine.

Sample No. 54-50 (P3 and P7)

Grand Forks area.

NW$\frac{1}{4}$ Section 11, T. 151 N., R. 50 W., Grand Forks County.

Dark greenish gray clay of the Lake Agassiz deposits, taken from cores supplied by the U.S. Army Corps of Engineers during the test drilling at the site of the Grand Forks Flood Control Levee in 1954. Sample No. 54-50 was obtained from a hole drilled on the west bank of the Red River.

The portion P3 is from Elev. 780.6 to 778.1, and the portion P7 is from Elev. 768.1 to 770.6.

Sample No. 54-51

Grand Forks area.

SW$\frac{1}{4}$ Section 11, T. 151 N., R. 50 W., Grand Forks County.

Dark greenish gray, plastic clay of the Lake Agassiz deposits, obtained at Elev. 780 by the U. S. Army Corps of Engineers, in 1954, at the site of the Grand Forks Flood Control Levee.

Sample No. 54-52

Portland area.

SE$\frac{1}{4}$ Section 32, T. 147 N., R. 51 W., Traill County.

Dark greenish gray, plastic clay of the Lake Agassiz deposits, occurring as a 40 foot layer below 30 feet of silt and soil. The portion tested is representative of the 40 foot layer, as obtained from the USGS samples which are filed at the University of North Dakota.

Sample No. 54-53

Hunter area.

NW$\frac{1}{4}$ Section 24, T. 143 N., R. 52 W., Cass County.

Dark greenish gray, plastic clay of the Lake Agassiz deposits, occurring as a 40 foot layer below 35 feet of silt and soil. The portion tested is representative of the 40 foot layer, as obtained from USGS samples which are filed at the University of North Dakota. Equal portions of the samples were blended into two composite samples, one representing the 35 to 55 foot interval, and the other the 55 to 75 foot interval.

Sample No. 54-54

Fargo area.

$NW\frac{1}{4}$ Section 1, T. 139 N., R. 49 W., Cass County.

Greenish gray clay of the Lake Agassiz deposits, occurring as a 75 foot layer below 18' of silt and soil. The portion tested is representative of the 75' layer, as obtained from the USGS samples filed at the University of N.D. Equal portions of the samples were blended into 2 composite samples; one representing the 20 to 55' interval, and the other, the 55 to 90' interval.

APPENDIX 2

LOCATION OF DESCRIPTION OF SAMPLES TESTED BY BUREAU OF MINES

FOR REPORT OF INVESTIGATIONS 5065

SAMPLE NO.	GENERAL AREA	LOCATION AND MATERIAL
3	Williston	T. 154 N., R. 100 W., Section 3. Dark gray bituminous shale, underlying the Bruegger coal bed in the Fort Union formation.
5.	Minot	T. 152 N., R. 82 W., Section 24. Blue-gray clay, overlying coal at Quality Lignite Co., mine, 24 miles southeast of Minot. Fort Union formation.
6	Garrison	T. 148 N., R. 83 W. Section 18. Gray clay overlying coal. Fort Union formation.
20	Belfield	T. 141 N., R. 99 W., Section 32, $SW\frac{1}{4}$ $SW\frac{1}{4}$. Gray clay in Fort Union formation.
22	Marmarth	T. 133 N., R. 105 W., Section 29 $SW\frac{1}{4}$. Gray bentonitic shale at base of a butte in the Hell Creek formation.
37	Zap	T. 144 N., R. 89 W., Section 24. Clinker from Dakota Collieries at Zap.

Cole and Zetterstrom (2A) found that the above clays and shales were suitable materials for the production of lightweight aggregate.

APPENDIX 3

CORRELATION OF DIFFERENTIAL THERMAL CHARACTERISTICS AND CERAMIC PROPERTIES

This material is briefed from the following publication:

Grim, R.E., & Rowland, R. A., Differential Thermal Analysis of Clays and Shales, A Control and Prospecting Method: Journal of the American Ceramic Society, Volume 27, March 1, 1944, pp. 73-74.

The thermal curves of the various types of clay illustrate certain general relationships between clay composition, as reflected in the curves, and ceramic properties that may be summarized as follows:

(a) An endothermic reaction below about 200°C (392°F) usually indicates the presence of montmorillonite or illite. A clay material containing these components is apt to have high plasticity and high shrinkage and will probably be nonrefractory and will burn red. In general, the larger this reaction, the higher are the plasticity and shrinkage.

(b) Endothermic reactions between about 300°C (572°F) and 550°C (1022°F) usually indicate a hydroxide of alumina or ferric iron oxide. If the component is a hydroxide of alumina, the clay will be very refractory and will have low shrinkage.

(c) A broad exothermic reaction between about 200°C (392°F) and 600°C (1112°F) is the result of organic material. Clays yielding such thermal reactions will frequently be very plastic and will require careful burning to insure complete oxidation of the carbon without ruining the ware.

(d) A sharp exothermic reaction between 400°C (752°F) and 500°C (932°F) indicates pyrite or marcasite.

(e) A sharp intense endothermic reaction at about 600°C (1112°F) and a sharp exothermic reaction at about 975°C (1787°F) indicates the presence of kaolinite. A clay with this component is apt to be refractory and light firing and to have low plasticity and a relatively long vitrification range.

(f) A clay with a slight endothermic reaction at about 500°C (932°F) or 700°C (1292°F), followed by another endothermic reaction at about 900°C (1652°F), and then a final slight exothermic reaction is composed of illite or montmorillonite. A clay containing either of these clay minerals is not refractory or light burning and is apt to have a short vitrification range. If the component is montmorillonite, it will also have high plasticity and shrinkage.

(g) A small endothermic break at 575°C (1067°F) shows the presence of considerable free silica (quartz) which will reduce the plasticity and shrinkage of the clay.

(h) A sharp intense endothermic reaction at about 850°C (1562°F) indicates the presence of carbonate and therefore a clay requiring careful preparation and firing technique.

It must be emphasized that experience and caution are necessary in interpreting the composition and properties from the differential thermal curve of a clay. In studying a large number of clays, some curves will be encountered that cannot be evaluated satisfactorily without additional analytical data from optical, X-ray, or chemical analyses.

BIBLIOGRAPHY

1. American Society for Testing Materials Specifications for Light Weight Aggregates for Concrete, Part 3, p. 902, 1952.

2. Cole, W. A., & Zetterstrom, J. D., Investigation of Lightweight Aggregates of North and South Dakota: U. S. Bureau of Mines Report of Investigations #5065, pp. 1-2, 1954.

2A <u>op</u>. <u>cit</u>. p. 28.

2B <u>op</u>. <u>cit</u>. pp. 20-22.

3. Conley, John E., Klinefelter, T. A., and others, Production of Lightweight Concrete Aggregates from Clays, Shales, Slate and Other materials: U.S. Bureau of Mines Report of Investigations #4401, p. 16, 1948.

3A <u>op</u>. <u>cit</u>. p. 12.

3B <u>op</u>. <u>cit</u>. p. 60.

4. Manz, Oscar, E., Investigation of some North Dakota Clays and Shales, North Dakota Geological Survey Report of Investigations #13, pp. 3-5, 1953.

4A <u>op cit</u>. pp. 5-6.

5. Clapp, C. H., Methods of Brick Manufacture and Description of North Dakota Industry: N.D. Geol. Survey Fourth Biennial Report, Part V pp. 298-300, 310-312, 1905.

6. Hvorslev, Juul, M., Subsurface Exploration and Sampling of Soils for Civil Engineering Purposes: Waterways Experiment Station, Vicksburg, Miss., pp. 57-65, 1949.

7. Taylor, D. W., Fundamentals of Soil Mechanics, John Wiley and Sons, Inc. New York, N. Y., pp. 92-96, 1948.

8. Matthews, J. G., Preliminary Report on Coated Lightweight Concrete Aggregate from Canadian Clays and Shales, Part II, Manitoba and Saskatchewan: Canada Dept. of Mines and Technical Surveys, Ottawa, Ontario, pp. 4-5, 1952.

8A <u>op</u>. <u>cit</u>. p. 22.

9. Rominger, J. F., and Ruttledge, P. C., Use of Soil Mechanics Data in Correlation and Interpretation of Lake Agassiz Sediments: Journal of Geology, vol. 60, pp. 160-180, 1952.

10. Bauer, Edward E. Plain Concrete, McGraw Hill Book Company, Inc. New York, N. Y., pp. 83-88, 1949.

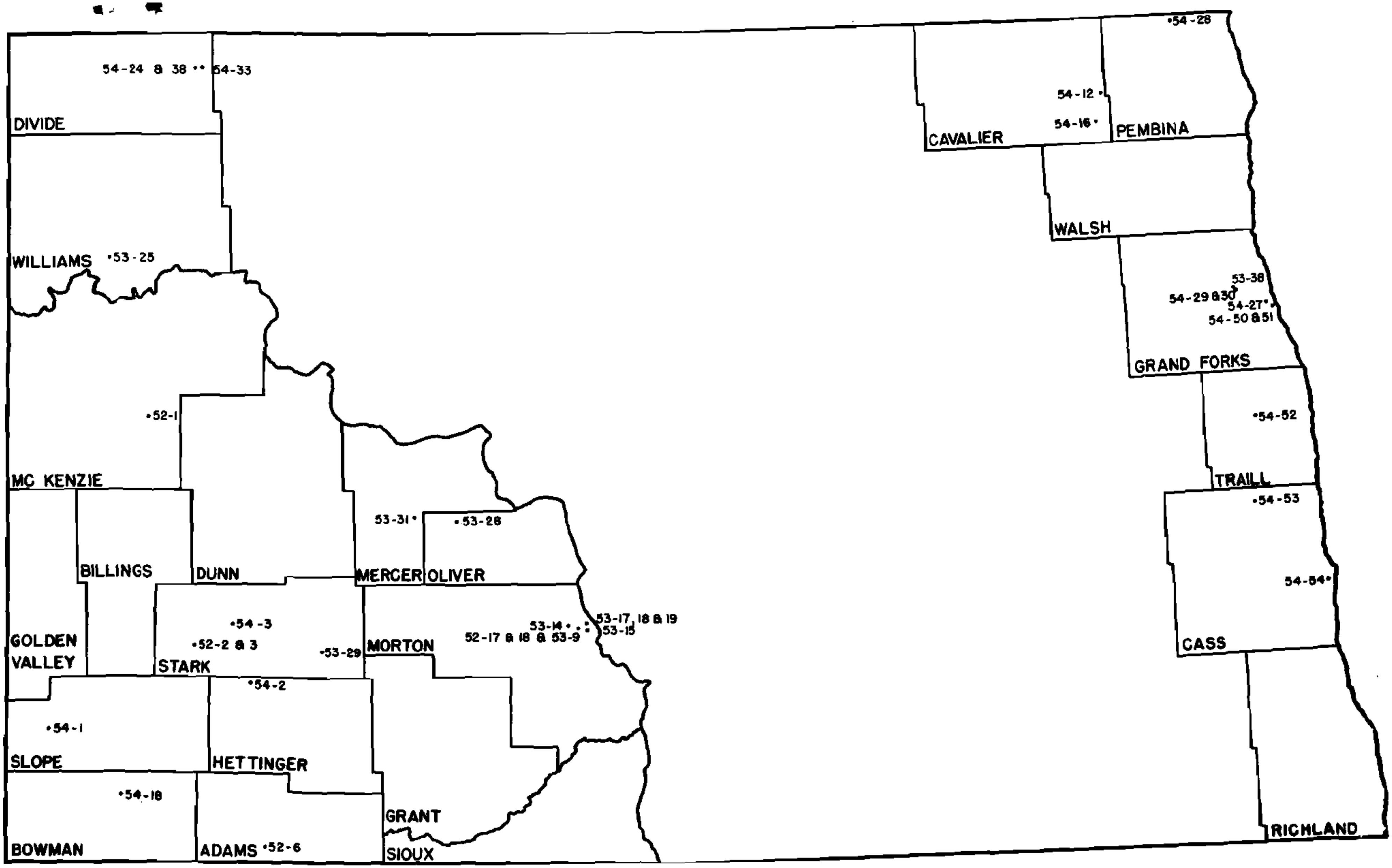

FIGURE I

SAMPLE LOCATION MAP

FIGURE 2 - DIFFERENTIAL THERMAL CURVES