University of Minnesota

Minnesota Geological Survey

George M. Schwartz, Director

Summary Report No. 5

THE POSSIBILITIES OF BLOATING CLAYS IN MINNESOTA

by

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INTRODUCTION

The object of this investigation was to determine the possibilities of Minnesota clay and shale deposits as raw materials for lightweight concrete aggregates. The project was proposed by the Natural Resources Committee of the Minnesota Association of Professional Engineers. Dr. George M. Schwartz, director of the Minnesota Geological Survey, assigned the exploration and testing work to the author, who during the summers of 1948 and 1949 carried out the investigation. The results of the sampling and testing of Minnesota clay deposits are presented and discussed.

The writer is greatly indebted to Dr. George M. Schwartz for proposing the problem, and for his many valuable suggestions. Dr. John W. Gruner gave much invaluable advice and inspiration. He pointed out new avenues of study, and supervised the experimental work. Dr. S. S. Goldich offered many valuable suggestions concerning the petrographic work on these clays. Mr. Lee C. Peck made the desired chemical analyses and advised the author on many technical problems. Mr. Henry H. Wade was very cooperative in permitting the use of the facilities of the Mines Experimental Station. Mr. H. G. Iverson of the Tuscaloosa, Alabama branch of the Bureau of Mines kindly tested and evaluated a number of samples and helped set up the testing procedures used in this project. Other samples were tested for rotary kiln firing characteristics by Dr. G. R. Pole of the Minnesota Mining and Manufacturing Company. The Minnesota Geological Survey financed the research. Mrs. Ruth Lawrence, director of the University of Minnesota Art Galleries, who tested many different Minnesota clays for other purposes generously made her results available. To all these people and organizations, the writer wishes to express his profound thanks.

TESTING OF MINNESOTA CLAY DEPOSITS

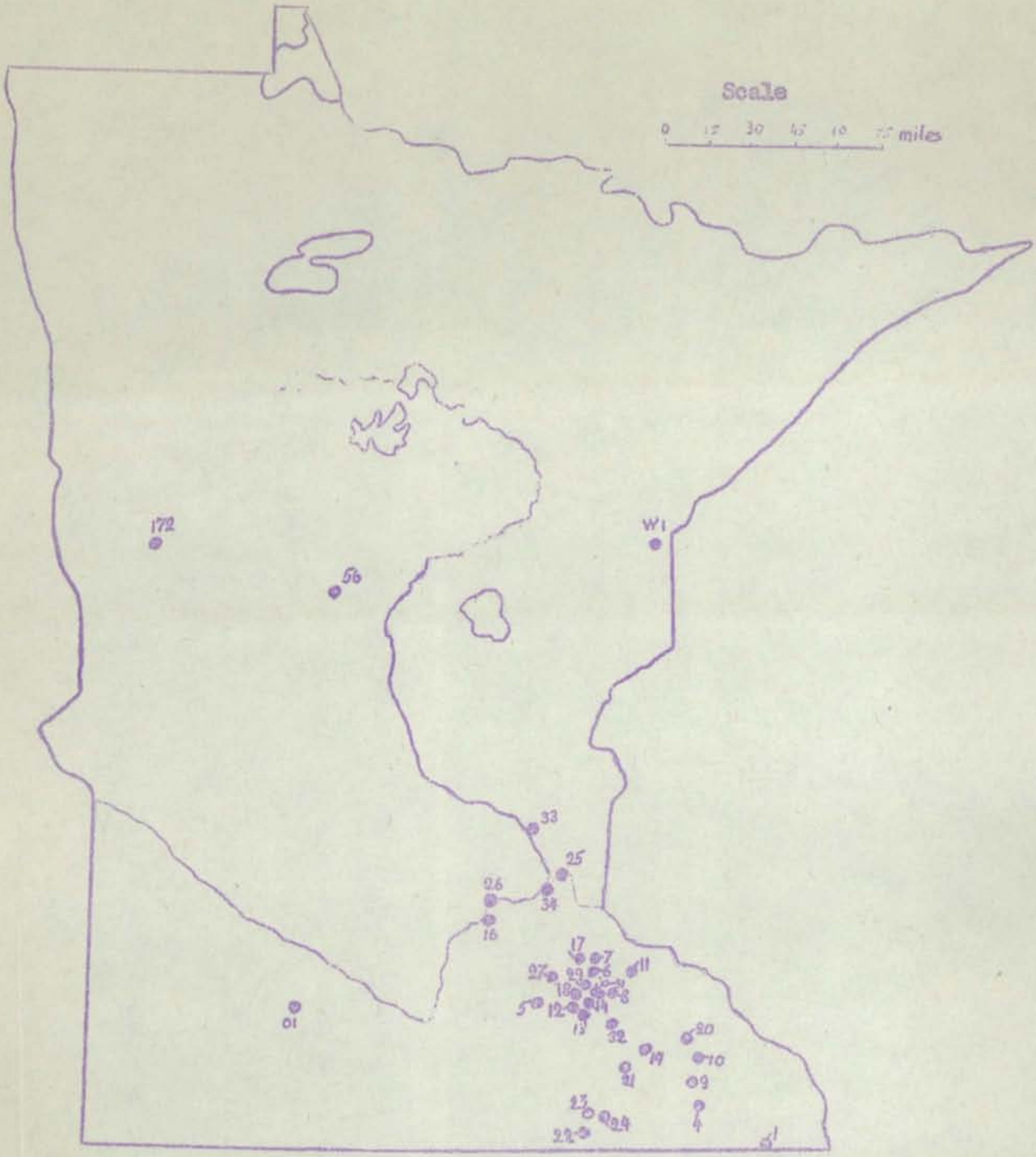
Sampling Procedures

The field work for this project consisted of sampling the principal clay and shale deposits of Minnesota (Grout 1919), and any new exposures that could be found. Because earlier reports indicated that many deposits of the Decorah shale bloated (Ruth Lawrence, verbal communication), the author did the most collecting from this formation. Also, the thickness and texture of this shale and its proximity to markets or rail transportation would make it the most feasible raw material for lightweight aggregate. Other clays and some loess deposits were collected when encountered, but nearly two-thirds of the samples were Decorah shale. The samples were taken by means of channeling, using the chisel end of a brick hammer which made a 1-1/2 inch wide slot down the face of an exposure. About five pounds were collected for every five feet five inch vertical section. Limestone beds and pebbles were also included in the sample. The locations of the deposits have been plotted on Plate I.

Laboratory Procedures

Air dried samples were sent through a small jaw crusher. A quarter of the sample, cut out with a Jones splitter, was then put consecutively through a rolls crusher and a pulverizer until it would pass through a sixty mesh screen. Because

MAP OF MINNESOTA SHOWING LOCATIONS OF CLAYS SAMPLED



(For types of clays and exact locations, see Minnesota geologic map.)

any commercial use of this material would require the screening out of limestone layers and pebbles, this material was hand picked from the samples before they were crushed. To one hundred grams of the powdered clay, water was slowly added from a burette until the desired plasticity was reached for hand packing into molds. The number of cubic centimeters of water used per 100 grams of clay is spoken of as the "water of plasticity" and is expressed as a percentage. This value, of course, is not an exact physical constant of the clay, but a factor which has an element of human error. It is useful, however, as a qualitative estimation of the "workability" of the clay in a pug mill or in an extrusion process should the material require pelletizing. The clay was then packed into a small mold and cast into briquettes 1-1/2" x 1" x 1/4" in size. The sample number and other information was enscribed in the moist clay before it was air dried.

The clays were fired in an electric furnace built by the author. The furnace was heated by six 3/4" x 27" "Globars" operating at 220 volts and using a current of about 38 amperes. These heat an alundum muffle of 5" x 7" x 14" around which is a shell of 7-1/2" of porous fire brick insulation. Such a furnace will reach 2300°F in about six hours and can be used for temperatures up to 2600°F. A platinum and platinum-13% rhodium thermocouple was used for all temperature measurements. The furnace accommodated eighteen briquettes, and thus six briquettes of each sample were fired at a time. At the start the furnace was turned up to its maximum heating spead and the temperature carefully watched. At 1800°F one briquette was taken out, at 1900°F another, and so forth until at 2300°F the last briquette was removed.

After cooling, the briquettes were examined to see whether bloating had taken place and at what temperature it had started. Overbloating, shrinkage, color and texture were also noted and recorded. The dimensions were carefully measured on the 1900° briquette and on an unfired, air dried briquette, and their volumes computed from these measurements. The ratio of the volume of the air dried briquette to that of the mold represents the volume shrinkage in air drying. The ratio of the volume of the 1900°F briquette to that of the air dried briquette represents the volume loss or gain in firing to common brick temperatures. From each sample which bloated the briquette was chosen which had the best texture in addition to a satisfactory degree of expansion. It was carefully measured so that a close approximation of its volume could be calculated.

Another property determined from the fired briquettes was the temperature of vitrification of the clays. This is defined by Grout (1919) as the temperature at which "....enough of the clay has fused to fill nearly all the pores between the unfused grains, but unfused material is still abundant and the fused portion is so viscous that the ware keeps its shape. Shrinkage is usually high." With the above definition in mind, the author estimated the vitrification temperature by examining the sequence of fired briquettes. Tables I and II contain all the pertinent firing data from the samples tested.

ECONOMIC EVALUATION OF DEPOSITS TESTED

The estimation of the economic value of the deposits of bloating clays which follows must be considered as purely preliminary report. It is meant to be a guide for further investigation and testing of favorable deposits. Auger sampling or even drilling with firing tests on the cuttings would be necessary before

Table I

Firing Data on Clays that Bloated

No.	Type	Location	Plast. % H_2O	Bloating Range Start	Bloating Range Over	Vol. Chg. Best Bloat Air Dried	Color 2000°
$R4_4$	Decorah lower 5'	*Fillmore 26 104N11W cut on US 52	40	2200°	-	1.6	Brick-red
$R5_1$	Decorah top 5'	Rice 6 109N20W old clay pit	41	2000	2200°	1.9	Brown
$R8_3$	Decorah middle 5'	Goodhue 21 110N16W old Gunderson pit	38	2000	2300	2.9	Brick-red
R9	Decorah 6' cut	Fillmore: Cut, Rd 74 on hill in Chatfield	47	2000	2300	2.4	Brown-red
R10	Decorah 8' cut	Olmsted 16 106N11W Cut on US 14	42	2300	-	1.2	Lgt. Brown
R16	Glacial 6' clay	Scott Co. in Jordan N end on US 169	33	2150	2250	1.6	Orange
R20	Loess 7' cut	Goodhue 13 107N12W bank of creek	23	2300	-	1.5	Brownish-orange
R21 1-3	Glacial 12½' cut	Olmsted 6 105N15W Stream bank Co.Rd.W	24	2200	2300	1.2	Orange
R24	Cretac. 6' cut	Mower 28 103N17W 1 mi. N of US 16	25	2300	-	1.4	Drk. Orange
R25 1-3	Decorah lower 35'	Ramsey 7 28N23W Twin City Brick Co.	44	2050	2250	2.5	Brown

Table I--Continued

No.	Type	Location	Plast. % H_2O	Bloating Range Start	Bloating Range Over	Vol. Chg. Best Bloat Air Dried	Color 2000°
$R27_1$	Decorah lower 6'	Rice 25, 30 111N19,20W 4.4 mi. S. Northfield	32	2200	2300	1.3	Orange
R29	Decorah 5' cut	Goodhue 30 111N17W SW corner, rd. cut	35	2150	2300	1.7	Brick-red
$R30_3$	Decorah lower 5'	Goodhue 21 110N17W $NE\frac{1}{2}$, creek bank	43	2150	2250	1.6	Brown
R32	Loess 15' cut	Dodge 9, 10 108N16W $N\frac{1}{2}$ section line	25	2200	-	1.5	Brownish-orange
R33	Glacial	Anoka 15 31N34W old pits near NPRR	26	2150	2300	1.6	Brown
R34 1-2	Decorah 12' cut	Dakota 34 28N23W near Mendota Bridge	41	2000	2300	1.5	Brown
G56	Glacial 6' cut	Todd 7 133N32W NPRR $1\frac{1}{2}$ mi. E of town	25	2200	2300	1.3	Brown

* Fillmore County Section 26 T104N R11W

Table II

Firing Data on All Clays

Sample No.	Clay Type	Location	% Water of Plast.	Ratio Volume Changes Air Dried / Mold	Ratio Volume Changes 1900° / Air Dried	Color 1800°	Color Change 2000°	Vitrification
R1	Decorah mid. 5'	Houston Sec.19 T101N R7W RR underpass US14	35%	.76	.93	Orange	Lighter	-
R2	Decorah top 5½'	Same as R1	39	.70	.90	Orange	Lighter	2300°F
R3	Decorah Lower 6'	Same as R1	30	.77	.95	Orange	Lighter	-
$R4_1$	Decorah top 5½'	Fillmore Sec.26 T104N R11W cut on US 52	46	.68	.92	Orange	Lighter	2300
$R4_2$	Decorah	Same $R4_1$	42	.63	.98	Orange	Lighter	2250
$R4_3$	Decorah	Same as $R4_1$	38	.71	.98	Orange	Lighter	2225
$R4_4$*	Decorah lower 5½'	Same as $R4_1$	32	.75	.92	Orange	Darker	2100
$R5_1$*	Decorah top 5'	Rice Sec.6 T109N R20W old Lieb Quarry	41	.67	.82	Orange	Darker	2100
$R5_2$	Decorah lower 5'	Same as $R5_1$	31	.74	.99	Buff	Lighter	2300
$R6_1$	Decorah lower 5½'	Goodhue Sec.8 T110N R17W Rd.cut US52	34	.81	.88	Red	Darker	2300
$R6_2$	Decorah top 5½'	Same as $R6_1$	33	.76	.94	Buff	Lighter	2250
$R7_1$	Decorah lower 5½'	Goodhue Sec.20 T112N R17W NE¼ 20 Co.Rd.cut	23	.81	.91	Buff	Lighter	2300
$R7_2$	Decorah top 5½'	Same as $R7_1$	33	.75	.97	Buff	Lighter	2300
$R8_1$	Decorah top 5½'	Goodhue Sec. 21 T110N R16W Gunderson Pit	27	.77	.99	Buff	Lighter	2200
$R8_2$	Decorah	Same as $R8_1$	33	.76	.91	Orange	Lighter	-
$R8_3$ *	Decorah	Same as $R8_1$	38	.62	.75	Red	Darker	2100
$R8_4$	Decorah	Same as $R8_1$	30	.77	.94	Orange	Lighter	2150
$R8_5$	Decorah lower 5½'	Same as $R8_1$	33	.74	.98	Orange	Lighter	2200
R9*	Decorah 6' cut	Fillmore St.Rd.74 hill in Chatfield	47	.65	.77	Orange	Darker	2100

Table II (Cont.)

Sample No.	Clay Type	Location	% Water of Plast.	Ratio Volume Changes: Air Dried / Mold	Ratio Volume Changes: 1900° / Air Dried	Color 1800°	Color Change 2000°	Vitrification
$R10_1$*	Decorah top 4'	Olmsted Sec.16 T106NR11W W of Dover on US14	42	.77	.81	Orange	Darker	2200
$R10_2$*	Decorah Lower 4'	Same as $R10_1$	42	.63	.91	Orange	Darker	2250
$R11_1$	Cretac. top 5'5"	Goodhue Sec.14 T111NR15W Goodhue Clay pits	21	.81	.98	Peach	Same	-
$R11_2$	Cretac. lower 5½'	Same as $R11_1$	22	.82	.98	Peach	Same	-
$R12_1$	Decorah top 5'5"	Goodhue Sec.8 T109NR18W N side US 60; creek	35	.76	.91	Buff	Lighter	2300
$R12_2$	Decorah mid.5'5"	Same as $R12_1$	32	.76	.98	Buff	Lighter	2300
$R12_3$	Decorah lower 5½'	Same as $R12_1$	23	.83	1.01	Tan	Lighter	2300
R13	Decorah 6' bank	Goodhue Sec.10 T109NR18W Bank of Pearl Creek	24	.84	1.00	Cream	Lighter	2300
$R14_1$	Decorah top 5½'	Goodhue Sec.35 T110NR18W Bank Zumbro River	24	.81	1.05	Tan	Lighter	2300
$R14_2$	Decorah	Same as $R14_1$	23	.86	.99	Tan	Lighter	2300
$R14_3$	Decorah	Same as $R14_1$	27	.81	1.02	Tan	Lighter	2250
$R14_4$	Decorah	Same as $R14_1$	33	.83	.91	Buff	Lighter	-
$R14_5$	Decorah lower 5½'	Same as $R14_1$	28	.82	.92	Orange	Lighter	2300
R16*	Glacial 6' clay	Scott Co. In Jordan N end on US 169	33	.73	.96	Orange	Same	2150
R17	Decorah 7' cut	Goodhue Sec.13 T112N R18W Cut on Co.Rd. 19	28	.87	.97	R-buff	Lighter	2200
R18	Decorah 5½' cut	Goodhue Sec.15 T110N R18W Creek cut, State 56	33	.73	.98	Buff	Lighter	2300
R19	Decorah 5½' cut	Olmsted Sec.32 T107N R14W Hill on U.S. 14	25	.85	1.02	Peach	Lighter	2300
R20*	Loess 7' cut	Goodhue Sec.13 T107N R12W Bank of Creek	23	.83	1.00	Orange	Darker	2250
$R21_1$*	Glacial lower 2½'	Olmsted Sec. 6 T105N R15W stream bank Co.Rd. W	25	.80	1.05	Orange	Darker	2150

Table II (Cont.)

Sample No.	Clay Type	Location	% Water of Plast.	Ratio Volume Changes Air Dried / Mold	Ratio Volume Changes 1900° / Air Dried	Color 1800°	Color Change 2000°	Vitrification
R 21_2*	Glacial mid. 5'	Same as $R21_1$	24	.82	1.04	Orange	Drkr.	2200
R 21_3*	Glacial top 5'	Same as $R21_1$	21	.80	1.00	Orange	Drkr.	2200
R22	Cretac. 6' cut	Mower Sec.35 T102NR18W exav. on US 218	34	.76	.87	Orange	Drkr.	–
R23	Cretac. 5' clay	Mower Sec.30 T103NR17W Stream bank on Co. Rd.	21	.84	1.02	Orange	Drkr.	–
R24*	Cretac. 6' cut	Mower Sec.28 T103NR17W 1 mi. N. of US 16	25	.75	1.04	Orange	Drkr.	2300
$R25_1$*	Decorah lower 10'	Ramsy Sec.7 T28N R23W Twin City Brick Co.	35	.72	.77	Tan	Drkr.	2100
$R25_2$*	Decorah next 12'	Same as $R25_1$	48	.66	.79	Tan	Drkr.	2100
$R25_3$	Decorah next 12'	Same as $R25_1$	50	.68	.78	Tan	Drkr.	2100
$R25_4$	Decorah next 12'	Same as $R25_1$	27	.81	.98	Lt. Tan	Lgtr.	2350
$R25_5$	Decorah top 15'	Same as $R25_1$	35	.75	.96	Lt. Tan	Lgtr.	2200
R26	Glacial 7' cut	Carver Co. pits 1 mi. NW of Chaska, Minn.	21	.93	.98	Cream	Lgtr.	2200
$R27_1$*	Decorah lower 6'	Rice Sec. 25, 30 T111N R19,20W 4.4 mi. S. Northfield	32	.78	.94	Orange	Drkr.	2200
$R27_2$	Decorah top $5\frac{1}{2}$'	Same as $R27_1$	24	.86	.99	Lt. Tan	Lgtr.	2300
R28	Loess 12' cut	Goodhue Co. Rd. (9) $\frac{1}{4}$ mi. W. of Sogn	24	.99	.97	Buff	Same	2200
R29*	Decorah 5' cut	Goodhue Sec.30 T111N R17W SW Corner, Rd. cut	35	.82	.76	Red	Drkr.	2150
$R30_1$	Decorah top $5\frac{1}{2}$'	Goodhue Sec.21 T110N R17W NE$\frac{1}{4}$, creek bank	35	.76	.84	Orange	Drkr.	2200
$R30_2$	Decorah mid. $5\frac{1}{2}$'	Same as $R30_1$	42	.69	.94	Red	Lgtr.	2300
$R30_3$	Decorah lower 5'	Same as $R30_1$	43	.70	.82	Red	Drkr.	2200

Table II (Cont.)

Sample No.	Clay Type	Location	% Water of Plast.	Ratio Volume Changes: Air Dried / Mold	Ratio Volume Changes: 1900° / Air Dried	Color 1800°	Color Change 2000°	Vitrification
R31	Loess 7' cut	Goodhue Sec.21 T110N R17W Road cut SE¼ of S.	22	1.00	1.01	Buff	Same	2200
R32*	Loess 15' cut	Dodge Sec.9,10 T108N R16W N½ section line	25	.91	1.00	Orange	Darker	2200
R33*	Glacial	Anoka Sec.15 T31N R34W old pits near NPRR	26	.83	.96	Orange	Darker	2100
$R34_1$*	Decorah lower 6'	Dakota Sec.34 T28N R23W near Mendota Bridge	37	.75	.81	Orange	Darker	2150
$R34_2$*	Decorah top 6'	Same as $R34_1$	42	.76	.71	Orange	Darker	2200
G56*	Glacial 6' cut	Todd Sec.7 T133N R32W NPRR 1½ mi E town	25	.83	.95	Orange	Darker	2100
W2	Glacial 10' level	Carlton Sec.28 T48N R16W Wrenshall pit	25	.85	.97	Buff	Lighter	2150
W3	Glacial 15' level	Same as W2	26	.86	.95	Tan	Lighter	2200
0 1	Cretac. lower 5'	Brown Sec.30 T109N R34W Oohs pit ½ mi S US14	27	.82	.98	Cream	Darker	2300
0 2	Cretac. Mid. clay	Same as 0 1	26	.94	.95	Lt. Tan	Darker	-
0 4	Cretac. top 5'	Same as 0 1	29	.74	.95	Tan	Darker	-
K 1	Glacial top 6'	Carlton Sec.20 T48N R16W Kelly pit; St.Rd. 1	25	.86	1.02	Peach	Lighter	2200
K 2	Glacial 30' level	Same as K 1	28	.91	1.00	Buff	Lighter	2200
BH2	Glacial top 6'	Carlton Sec.26 T47N R17W Blackhoof pit	40	-	-	Orange	Darker	2100

* Bloating Clays

** Explanation of Main Headings

1. Plast. % H_2O: Water of Plasticity or % added to give clay good workability.
2. Volume changes: Ratios of Volumes
   a. of air dried briquette to that of the mold.
   b. of briquette fired to 1900° F to that of the air dried briquette.

property should be leased. Table I lists all the clays which bloated, regardless of the quality of the bloating or the potential value of the deposit. The following pages consider in detail the relative merits of these deposits, and the author recommends additional work on those he considers most favorable.

Any detailed study of these deposits should first determine whether the bloated clay has the desirable properties for a lightweight aggregate. Conley, Wilson and Klinefelter (1948) set down the following seven attributes:

1) Lightweight: It should weigh no more than half as much as the standard aggregate it replaces. Normally this requires a product with a bulk density of less than 50 pounds per cubic foot.

2) Strength: This is essential for structural concrete, for the stronger the aggregate the less cement is required and the greater is the savings in costs.

3) Particle shape to promote good workability: Well rounded particles are desirable as they make a concrete which will more easily work into forms and around reinforcing bars.

4) Low water absorption: Pores should be closed, for if they fill with water, the cement would be dehydrated, resulting in a poor set.

5) Uniform particle size gradation: A range of sizes, including a sufficient quantity of fines, is necessary to insure good workability of the concrete.

6) Chemical inertness: Compounds that would tend to react with the cement and impair its setting should not be present.

7) Low Production cost: This is the ultimate factor that determines its acceptability. The added cost of the lightweight aggregate over that of sand and gravel must be offset by savings in weight that permit elimination of structural steel, or by attainment of better thermal and sound insulation qualities.

The engineering tests needed to establish the above qualities of an aggregate in question require equipment large enough to handle sizable amounts of material so that enough aggregate would be produced to actually mix into a concrete. Tests on the concrete would quickly show the worth of the aggregate. Research such as this is in the realm of the engineer and is beyond the scope of this investigation. References such as Foster (1940), Moyer (1942) and Sullivan, Austin and Rogers (1942) contain valuable information on engineering techniques for testing and production of lightweight aggregate.

Assuming that a deposit produces a desirable aggregate, and is under consideration, what qualities must the deposit have to make it a profitable investment? It must, of course, be not only of a size sufficient to supply the intended market, but be so situated that it could be easily mined. The overburden should be an unconsolidated material which could be easily removed. The deposit itself, should be thick enough so that sufficient tonnage can be found in a relatively small area. It is best to have most of the material above the ground water table. The whole unit should be of uniform firing quality so that selctive mining would not be necessary. The material should have a long bloating range, and should not become sticky at the firing temperature. It is best to have a well-indurated material that would remain in lumps which could survive screening and handling. Otherwise, a process of pugging and pelletizing would be necessary, increasing the costs. Lastly, for a bulk product such as this, the proximity to ready markets and/or cheap transportation is an obvious advantage.

The Decorah Shale

For the purposes of this investigation, the term "Decorah" has been applied to the rocks which are predominantly shale and which lie between the Galena and Platteville limestones. Many interbedded limestone layers occur near the upper and lower boundaries of this lithologic unit, but these gradational zones are not usable as a raw material for lightweight aggregate.

The advantages of this shale over the other types of deposits are many. It is the only usable material located in and around Minneapolis and St. Paul. Other outcrop areas (see Stauffer and Thiel, 1941) are in the southeastern part of the state near smaller centers of population such as Rochester and Northfield. Tests have shown that at one locality as much as thirty-five feet has good uniform bloating qualities. The part which does not bloat makes an excellent brick so that the whole formation could be utilized. The shale breaks out into blocky lumps that could be dried, screened and fed directly into the rotary kiln with a minimum of handling. A description and evaluation of the best Decorah deposits investigated by the author follow in order of their desirability.

$R25_{1-3}$: This deposit, sampled in the pit of the Twin City Brick and Tile Company in St. Paul, was by far the best in all respects. Here in a pit already being worked within the city limits of St. Paul is a thickness of over thirty feet of shale (bottom three levels of the pit) with good bloating quality. A few thin limestone beds are present but are easily removable by screening. Tests show that rotary kiln firing at about 2100°F produces excellent bloated material. The tendency to become slightly sticky at temperatures above 2150°F indicates that a careful temperature control would be necessary. The proximity to markets and fuel supply, and the quality of bloating make this the best possible source of raw material for light weight aggregate for the Twin City area, and would only be limited by the not too extensive reserves, and their probable greater value for use in face brick.

$R34_{1-2}$: Located in Dakota County, Section 34 T31N R34W in the bluff behind the gas station at the east end of the Mendota bridge, this exposure represents the southern limits of the Decorah shale in the Twin City area. Its bloating quality is very similar to, if not better than, the St. Paul deposit. This suggests that the Decorah shale which lies in the area south of the city limits of St. Paul and within the bend of the Mississippi river may all be of good bloating quality. This area and outcrops along the river bluff from the village of Mendota to St. Paul should be investigated thoroughly. There is, however, a problem of zoning within this area that may interfere with utilization.

$R8_3$: This is one of the samples from the old Gunderson pit near Zumbrota, Goodhue County (SW1/4 of NE1/4 Sec. 21 T110W R16W). It is inferior to the above mentioned deposits both in location and bloating ability. Only the middle one of the five channel samples, a thickness of 5-1/2 feet, bloated and the pit is nearly fifty miles from the Twin Cities, the chief market for lightweight aggregate.

The deposit is extensive, however, and the chance that the unweathered material would fire differently warrants further investigation should none of the deposits near the Twin Cities prove to be useful.

$R27_1$: In Rice County, Sections 25 and 30 T111N R19, 20W along a county road 4.4 miles south of Northfield, there is adequate acreage of Decorah shale with little overburden. Only the lower six feet bloated, however, and the firing showed a very short range in bloating. The outcrop was quite weathered, however, so that further testing of fresher material obtained by augering or drilling is recommended.

Another area which might deserve a more detailed investigation centers around Cannon Falls and south of the Cannon River in Goodhue County. Some old sections described by Stauffer and Thiel (1941) indicate that a much greater thickness of Decorah is present in this area than was sampled by the author (R6, R7, R17). The firing tests on the Decorah sampled from deposits in the St. Paul area (R25, R34) showed that only the lower half of this lithologic unit bloated. It may be reasonable to assume, therefore, that in the Cannon Falls area there may be much shale that lies below the outcrops sampled by the author, which might have a bloating quality comparable to that in the St. Paul area. Except for deposits near the Twin Cities, which might be unavailable because of zoning restrictions, the Decorah shale near Cannon Falls would be the closest occurrence of this material to the chief market for lightweight aggregate. For this reason the author recommends a more detailed study which would disclose the worth of the lower part of the Decorah in this area.

In the opinion of the author, the other Decorah deposits listed on Table I are not worth further study. Some bloat very poorly and/or are too far from suitable markets or transportation. Others have too much overburden to be mined economically, or do not have enough reserves.

Loess

There are two main disadvantages to the using of this material rather than the Decorah shale for bloating. In the first place, it is not a consolidated sediment and would have to be pugged and pelletized before it could be fired. Also, it requires a relatively high temperature for bloating (about 2200°F) and has a short range. It does bloat, however, and with pelletizing should produce a good product. Only two loess samples bloated, and both are fairly near Rochester. If these could be handled cheaply enough they might be of value.

R20: Sampled from an outcrop in a creek bed three miles ENEof Viola in Olmsted County, Section 13 T107N R12W, this loess has an exposed thickness of over six feet and adequate acreage. It does not start to bloat until about 2200°F and has a very short range, becoming sticky and starting to slag down at 2300°F. It would require careful temperature control in firing.

R32: Located along State road 57 in the north half of Sections 9 and 10 T108N R16W in Dodge County, this exposure shows a fifteen foot thickness of uniform loess, which apparently covers a wide area. It bloats well, starting at 2200°, and begins to get sticky at 2300°F. The range is short and would require careful firing. This deposit is probably better than R20 in that it is thicker, more uniform, and would be cheaper to mine.

Glacial Lake Clays

There are many good clay deposits of this type throughout Minnesota and many of them have been or are being worked for brick and tile (see Grout, 1919 and Bradley, 1950, unpublished thesis, University of Minnesota). The author sampled and tested many of these deposits and found only three worthy of further consideration. In the first place, a very few are located near the Twin Cities, and would have to produce excellent product at a low cost tc be economically feasible. Like the loess deposits, most of these would require pugging and pelletizing before firing. Three of the best deposits are reported here, but they are not recommended for further study unless the Decorah shale is found to be not usable.

R33: This sample is from an old pit near the Northern Pacific Railroad tracks where they cross the triple width highway between New Brighton and Anoka. It is located in Anoka County, Section 15 T21N R34W. This sample was given first choice, not because it produced the best bloated product, but because of its favorable location with respect to the Twin Cities. It started bloating at 2150°F, expanded suddenly, and became sticky and overbloated at 2300°F. The range is short, but it warrants further investigation.

G56: This deposit of glacial lake clay is located in Todd County 1-1/2 miles east of Staples along the Northern Pacific Railroad in Section 7 T133N R32W. It bloats suddenly at 2200°F but quickly becomes soft and sticky giving it a firing range that is very short for commercial uses. This reaction, in addition to its location, makes it an unlikely source for lightweight aggregate.

$R21_{1-3}$: These samples were taken from a stream bank in Section 6 T105N R15W, 1-1/2 miles west of Rockdell in Olmsted County. They represent a twelve foot thickness of Keewatin Drift Clay. There seems to be a large acreage. This clay bloats quite well starting at 2200°F, but has a rather short range. It contains pebbles and cobbles which would have to be removed before the clay could be utilized. This factor, in addition to its isolated location, makes it a poor prospect.

Cretaceous Clay

Of all tested only one sample (R24) of Cretaceous clay bloated. The bloat was poor, however, and at a high temperature. The deposit is thin and located far from the Twin Cities. Further investigation of these deposits are not recommended unless a good market developes in Austin and Albert Lea.

PETROGRAPHIC STUDIES

Because of the mineralogic complexities of the loess and glacial lake deposits, the petrographic studies were confined to the Decorah shale. None of the Cretaceous material bloated. For this reason it was also excluded from further studies. The many samples of Decorah are very similar in appearance, yet when fired they react quite differently. Their colors when fired, temperatures of vitrification, and their abilities to bloat seem to bear no relationship to the appearance of the unfired shale. Indeed, there are many cases where in a series of channel samples from one thick outcrop, one sample bloated, while that adjacent to it did not. Two such pairs, $R8_2$-$R8_3$ and $R25_3$-$R25_4$, were submitted for chemical analyses, and detailed petrographic work was done on these in order to distinguish any differences between them, for it seemed that here was a good opportunity to closely compare two similar samples which react quite differently. The work was carried on along three major lines: (1) chemical analyses; (2) particle size analyses; and (3) mineralogic determinations. The chemical analyses of six samples, four of Decorah shale and two of glacial lake clays, were made by the Rock Analysis Laboratory of the University of Minnesota. The results of these analyses may be found in Table III.

Particle size fractions were separated from a number of samples of Decorah shale. It was hoped that concentrations of the accessory minerals would be found in these fractions, since accurate mineral identifications by means of X-ray diffraction patterns are not possible for most minerals unless they occur in amounts over about 10%. In order to obtain size fractions for material which is very fine grained two procedures were employed. The first method used was the pipette technique described by Pettijohn and Krumbein (1939), and the second was by hydraulic elutriation. The elutriator used was designed by Strathmore R. B. Cooke (1937) of the School of Mines, University of Minnesota. Both methods are based on Stoke's law of settling velocities of particles in a viscous medium.

The only practical method of determining the mineral composition of the fine fractions separated from these clays is by means of X-rays. Since calcite is abundant and gives a complex X-ray pattern, it was desirable to remove it first. HCl could not be used as it would attack many of the clay minerals as well. A suggestion by Grim (1934) led to a very satisfactory method of removing the calcite. Carbon dioxide was bubbled through a water suspension of the sample until a manometer attached to the container showed a gas pressure equivalent to about thirty-six inches of water above the sample. About twelve hours was usually sufficient time for the carbonic acid to dissolve all the calcite from the small amount of clay needed for X-ray studies. A small piece of iron was included with the sample, and by agitating this periodically with a magnet the sample was stirred sufficiently to hasten the dissolving process. Washing was done by centrifuging and decanting.

Tables IV and IVa show the distribution of minerals in the various fractions of the Decorah shale as determined by X-ray studies. It can be seen from these tables that nearly all the accessory minerals, except orthoclase, occur in the coarser fractions of the clay. The feldspar, which is authigenic in origin, is present in fractions as small as 1/420 mm. Gruner and Thiel (1937) studied the occurrence of authigenic feldspar in the Decorah shale and found two concentrations of orthoclase in the pipette fractions. One was in the 1/8 - 1/16 mm. size range, and the other in the range 1/256 - 1/512 mm.

Pyrite was detected in fractions 1/220 mm and larger. The specific gravity of pyrite is about 5.0, however, but size ranges for the elutriation fractions and the pipette fractions were calculated for particles whose specific gravity is 2.65 (that of quartz). Therefore, the pyrite will be found in fractions much larger than its grain diameter warrants, and no doubt some of it occurs in grains no larger than 1/420 mm. in diameter.

The above discussion has indicated the minerals that are present in this shale and in what size fractions they occur. No difference in mineral content was found between the two pairs of adjacent channel samples ($R8_2$ - $R8_3$; $R25_3$ - $R25_4$). Since in each pair one bloated while the adjacent sample did not, it is readily seen that in these samples the cause of bloating is not due to the

Table III

Chemical Analyses

Sample Numbers	$R8_2$	*$R8_3$	*$R25_3$	$R25_4$	BH2	W1
SiO_2	40.35	48.94	48.50	43.10	50.84	51.95
Al_2O_3	16.62	20.33	20.78	15.16	13.43	10.54
Fe_2O_3	4.85	4.97	3.75	3.59	4.85	2.26
FeO	.55	.71	1.40	1.22	.93	1.28
MgO	1.95	2.53	2.83	2.84	4.68	4.84
CaO	12.19	2.89	2.26	10.68	7.30	10.29
Na_2O	.12	.20	.12	.12	1.03	1.37
K_2O	6.63	7.64	7.69	6.66	2.50	1.86
H_2O+	4.18	5.16	4.97	3.37	3.71	2.40
H_2O-	2.59	3.33	4.06	2.50	2.70	1.28
CO_2	8.96	2.25	2.05	8.95	6.86	10.87
TiO_2	.59	.78	.79	.60	.66	.49
P_2O_5	.32	.31	.20	.40	.13	.13
SO_3	-	-	.07	.14	-	-
S	.04	.02	.81	.94	.02	.16
MnO	.10	.04	.03	.07	.09	.08
BaO	.01	.01	.01	.00	.03	.05
less O for S	.02	.01	.41	.47	.01	.08
Total	100.03	100.10	99.91	99.87	99.75	99.79

Eileen K. Oslund, Analyst

* Bloating Clays

$R8_2$, $R8_3$, $R25_3$, $R25_4$ are samples of Decorah shale.

BH2 and W1 are Glacial lake clays.

(For locations and horizons, see Table II)

Table IV

Minerals in Different Pipette
Fractions of Decorah Shale

Particle Size in mm.	Sample Numbers: $R8_2$	$*R8_3$	$*R9$	$*R25_1$	$*R25_3$	$R25_4$
$>\frac{1}{32}$	illite calcite quartz limonite	illite calcite quartz limonite	illite quartz orthoclase calcite apatite	illite pyrite calcite quartz	illite pyrite orthoclase calcite quartz	illite pyrite orthoclase calcite quartz
$<\frac{1}{32}$	illite orthoclase	illite orthoclase	-	illite orthoclase quartz	illite orthoclase	illite orthoclase
$<\frac{1}{128}$	illite orthoclase	illite	-	illite orthoclase quartz	illite orthoclase	illite orthoclase
$<\frac{1}{1024}$	illite	illite	-	illite	illite	illite

(See Table II for locations)

* Bloating Clay

Table IV$_a$

Minerals in Different Elutriation
Fractions of Sample R25$_3$ (Decorah)

	Fraction Numbers					
Particle Size in microns	(1)	(2)	(3)	(4)	(5)	(6)
	2.4	2.4 - 3.3	3.3 - 4.5	4.5 - 6.5	6.5 - 10.0	10.0 - 14.0
	illite	orthoclase & little illite	orthoclase	orthoclase & little pyrite	pyrite orthoclase	pyrite & little orthoclase

* All mineral determinations in fractions less than 1/32 mm. were made by means of X-rays.

** All calcite was removed from samples before X-ray pictures were made.

Sizes calculated on basis of a specific gravity of 2.65.

presence or absence of some one or more "necessary" minerals. To complete the petrographic description of the Decorah shale, the percentage of modal minerals was determined (See Table V). This was done by assigning the proper proportions of the oxides from the chemical analyses to the minerals known to be present in the shale. For minerals with nearly exact chemical composition, this would be an easy matter, but in the case of the Decorah shale, only an approximation can be achieved for two reasons. In the first place, Grim, Bray and Bradley (1937) gave the oxide formula of illite as approximately:

$$2K_2O \cdot 3MO \cdot 8R_2O_3 \cdot 24SiO_2 \cdot 12H_2O$$

where the divalent ion "M" could be either Mg^{++} or Fe^{++}, and the trivalent ion "R" could be either Fe^{+++} or Al^{+++}. From the chemical analyses of this mineral, the proportion, Fe^{++}: Mg^{++} was approximately 1/2 : 3-1/2, and the proportion Fe^{+++}: Al^{+++} was about 1 : 7. It was necessary, however, to vary these ion proportions in order to account completely for the oxides in the analyses. In the second place, the petrographic studies could not have revealed all of the minor mineral constituents of the shale. To account for certain "surpluses" in the analyses, it was necessary to assume the presence of two additional minerals. For the excess MgO in 25_3 and 25_4, it was assumed that dolomite was present which had not been distinguished from the calcite. A sulphate was "needed" to explain the SO_3 reported in the analyses. Gypsum was the logical choice. TiO_2 probably occurs in leucoxene and P_2O_5 either in apatite or in limonite. The author made no attempt to account for the Na_2O. It is estimated that the figures reported on Table V are accurate within 0.2% for the minor constituents and within 1-2% for the major ones.

Table V

Mineral Proportions in the Decorah Shale
Calculated from the Chemical Analyses

Sample Numbers	$R8_2$	*$R8_3$	*$R25_3$	$R25_4$
illite	56.3	73.7	67.6	52.9
orthoclase	16.0	13.4	20.6	20.1
calcite	21.2	5.4	3.4	17.5
pyrite	.1	.1	1.6	1.8
limonite	3.7	2.9	3.0	-
quartz	2.7	4.5	2.4	4.2
**dolomite			1.3	3.3
**gypsum			.1	.3
Total	100.0%	100.0%	100.0%	99.9%

* Bloating Clay

** Normative minerals not detected by petrographic study

(See Table II for locations)

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