NRRI/GMIN-TR-88/05

PRELIMINARY ASSESSMENT OF THE INDUSTRIAL CLAYS AT WRENSHALL, MINNESOTA

By

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January 1988
Technical Report
NRRI/GMIN-TR-88/05

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Recommended Citation

Heine, J., and Hauck, S.A., 1988, Preliminary Assessment of the Industrial Clays at Wrenshall, Minnesota: University of Minnesota Duluth, Natural Resources Research Institute, Technical Report NRRI/GMIN-TR-88/05, 19 p.

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INTRODUCTION

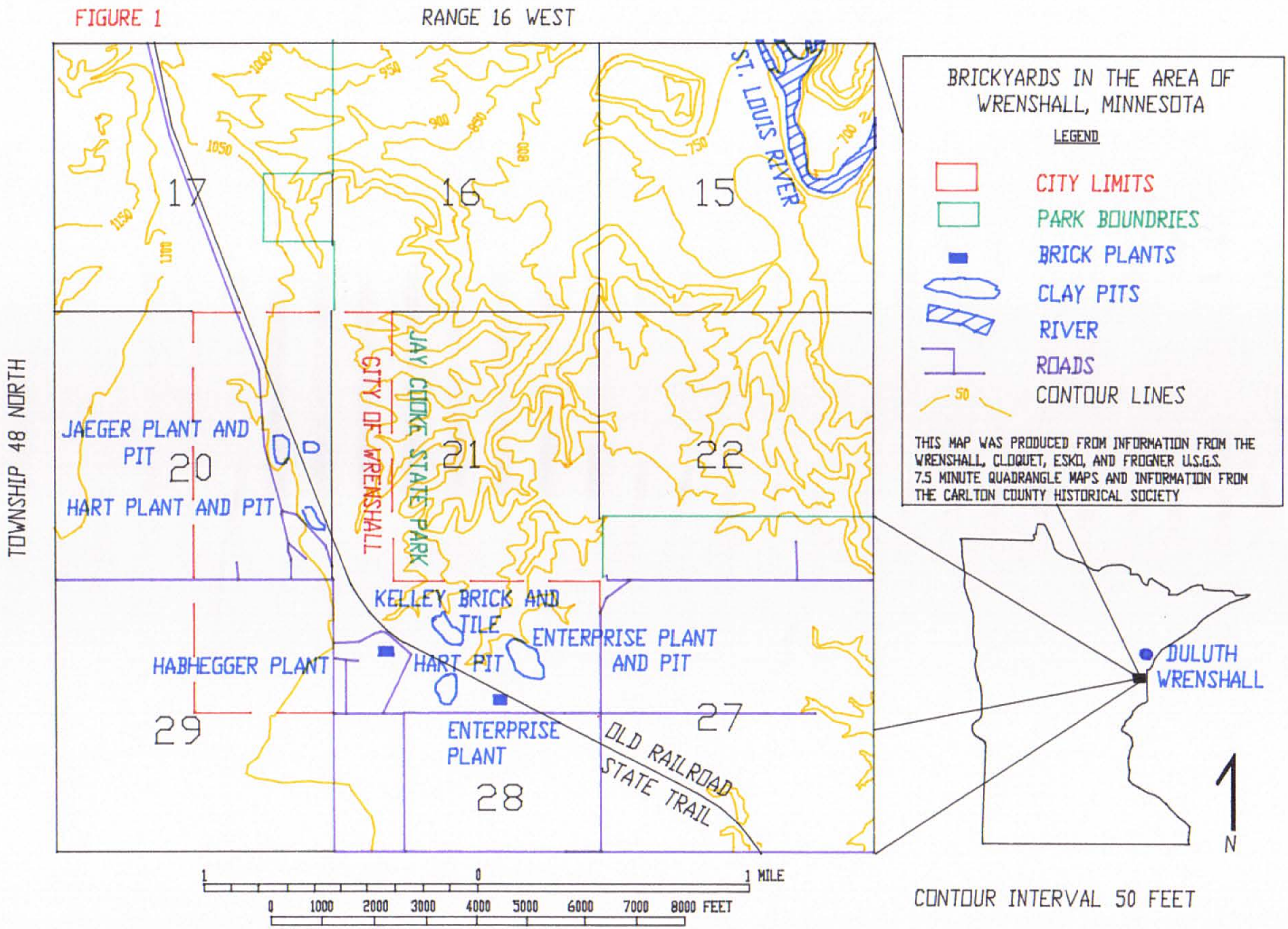
The Wrenshall, Minnesota area has a long history of clay production and it appears there are still abundant clay resources remaining in the area. Wrenshall is located eight miles southeast of Cloquet, Minnesota, and 20 miles southwest of Duluth, Minnesota. The production period in the Wrenshall area was 1882-1953, and the products produced were primarily brick and clay tiles. Because of the productive history and the potential resource available, the Natural Resources Research Institute (NRRI) of UMD became involved in a study to determine the feasibility of using Wrenshall clays in the manufacturing of clay products. This study includes the determination of the physical and chemical properties of Wrenshall clays, the development of a clay products inventory, and a products-property match analysis.

The objective of NRRI's study is to aid the development of a viable clay products industry in the Wrenshall area. This report describes some initial results, provides a preliminary assessment of Wrenshall clays and outlines a plan for further study.

HISTORY OF CLAY PRODUCTION IN WRENSHALL, MINNESOTA

In 1882, Fred Habhegger opened the first brickyard in the area near Clear Creek, northeast of the town of Wrenshall. He moved his operations west in 1888, opening the Habhegger Brick Company in Wrenshall. Following this beginning, other brickyards were opened in Wrenshall; i.e., the Hart, Jaegger, two Enterprise plants, Rusfelt, and Kelly Brick and Tile Company (Figure 1). During the period 1888-1953, 600 million bricks were produced from Wrenshall clays. The Habhegger plant produced 4 million bricks per year when operating. Peak production at the Enterprise plants was 6.5 million bricks per year. The Wrenshall Brick Company (formerly the Rusfelt Brickyards) produced 3.5 million

FIGURE 1


RANGE 16 WEST
TOWNSHIP 48 NORTH
BRICKYARDS IN THE AREA OF WRENSHALL, MINNESOTA
LEGEND
CITY LIMITS
PARK BOUNDRIES
BRICK PLANTS
CLAY PITS
RIVER
ROADS
CONTOUR LINES
THIS MAP WAS PRODUCED FROM INFORMATION FROM THE WRENSHALL, CLOQUET, ESKO, AND FROGNER U.S.G.S. 7.5 MINUTE QUADRANGLE MAPS AND INFORMATION FROM THE CARLTON COUNTY HISTORICAL SOCIETY
ST. LOUIS RIVER
CITY OF WRENSHALL
JAY COOKE STATE PARK
JAEGER PLANT AND PIT
HART PLANT AND PIT
KELLEY BRICK AND TILE
HART PIT
ENTERPRISE PLANT AND PIT
HABHEGGER PLANT
ENTERPRISE PLANT
OLD RAILROAD STATE TRAIL
17
16
15
20
21
22
29
28
27
DULUTH
WRENSHALL
N
1 MILE
0 1000 2000 3000 4000 5000 6000 7000 8000 FEET
CONTOUR INTERVAL 50 FEET

bricks in 1953 before closing. Presently an abandoned refinery is located on the site of the Wrenshall Brick Company. The bricks that were originally produced from Wrenshall clays were underfired backing brick, which were salmon to buff in color. Closing of the brick plants was due to a number of reasons which included economics, technology and shift in market demands.

INITIAL STUDY

A preliminary investigation was conducted to determine the potential of Wrenshall clays for making state-of-the-art brick and other clay products. The investigation included preliminary geology and the determination of extrudability, drying shrinkage, firing shrinkage, fired porosity, fired color and firing range.

There are two types of clay in the Wrenshall area, a lower grey clay and an overlying red clay, with some interlayering of the two clays. Both clays are glacial lake deposits from the glacial lakes Nemadji and Duluth. The source of the grey clay is to the northwest, while the source of the red clay is to the northeast.

Clay samples for testing were taken from two former areas of production. The grey clay was taken from the former Kelly Brick and Tile pit in the NE 1/4, NW 1/4 of SEC 28, T48N R16W. The red clay sample was taken from the Enterprise pit in the NE 1/4, SEC 28, T48N, R16W.

Samples were collected from small test pits within the larger clay pits. The red clay sample was collected from a slump in the Enterprise Brickyard pit, which could be traced back to the pit wall. The walls in both of the pits are badly slumped and they are partially filled with water. The clay samples were taken from a range of approximately one to five feet below the weathered surface. Care was taken to collect an unweathered sample of each type of clay in order to study each clay's physical characteristics. These clays were tested separately and in blends of red to grey of 67/33 and 60/40. Testing of the clay samples was completed by the Department of Ceramic Engineering, University of Missouri-Rolla. Test bricks were extruded from the material and the characteristics previously described were determined. The data from testing is included in Appendix I of this report. Descriptions of the test procedures are included with the data (Appendix I).

CONCLUSIONS

The clays show good working and firing characteristics, but have a yellow-brown color when fired to 2050°F. The clays have good plastic properties and extrude well over a range of moisture contents. Drying shrinkage is good for the clays, the grey being 8 percent and the tests on red indicating a low range value of 5.5 percent. Firing shrinkages at 2050°F are in the acceptable range of 4-6 percent. At 2050°F the grey clay has a firing porosity of 16 percent, while the red has a value of 25 percent. The clays start densification at about 1950°F. At 2050°F, the clays are in the well-matured range. The low values of the grey clay, 9-12 percent, would make it suitable for extreme freeze-thaw applications. Firing ranges appear to be narrow, but more detailed firing is necessary to further delimit the ranges. The 2050°F fired color is a yellowing-brown, which may be due to bleaching of iron oxide by calcium carbonate. The 1850°F fired color is a cream color. Table 1 illustrates the technical color values.

Table 1

Munsell Color Description for Unfired Clay, Clay Fired at 1850°F and 2050°F.

	SAMPLE RED	67%RED/33%GREY	60%RED/40%GREY	GREY
UNFIRED CLAY				
MUNSELL COLOR	5 YR 7/2	7.5 YR 7/2	7.5 YR 7/2	2.5 YR 8.5/2
COLOR	RED GREY	RED GREY	RED GREY	GREY
1850°F				
MUNSELL COLOR	10 YR 8/4	7.5 YR 8.5/4	7.5 YR 8/4	7.5 YR 7.5/4
COLOR	GREY RED	YELLOW RED	YELLOW RED	YELLOW RED
2050°F				
MUNSELL COLOR	10 YR 8/5	10 YR 8/6	10 YR 7/6	10 YR 6.5/6
COLOR	YELLOW BROWN	YELLOW BROWN	YELLOW BROWN	YELLOW BROWN

Explanation of Terms in Table 1

The Munsell Color System is a method of describing the color of material based on three variables: hue, value, and chroma. The identification is written as:

HUE SYMBOL	VALUE/CHROMA
10 YR	9/2

HUE SYMBOL is a description of color based on five different major hues and five combinations of these hues. The major hues are red (R), yellow (Y), green (G), blue (B), and purple (P). The hue symbol for the red clay changes from 5 YR to 10 YR as the clay is fired, indicating a change in color to more yellow hues.

VALUE/ indicates the lightness or darkness of the color, where 0/ is absolute white and 10/ is absolute black.

/CHROMA indicates the degree of departure of a given hue from a neutral grey of the same value, giving a measure of the intensity of the hue. The chroma number increases as the intensity of the color increases.

Munsell Book of Color
Matte Finish Collection
Munsell Color Company, Inc.
Baltimore, Maryland (1976)

The color of the fired clay makes the material less suitable for use in the manufacturing of brick. As the clay is fired to higher temperatures, the hue shifts from red-yellow to a more yellow hue. The red clays become darker (have a higher value) as they are fired, while the grey clays become slightly lighter. The color intensity, chroma, increases with firing in both the red and grey clays. Plate 1 shows the raw clays and fired test bricks at temperatures of 1850°F and 2050°F. The photograph in Plate 2 shows pages from the Munsell Book of Color which is used to determine the color description of the materials found in Table 1. Because there is a strong preference for red and/or buff colors in the brick industry today, the Wrenshall brick colors are not colors which are popular at this time. However, there are additives and glazes which can be used to alter this characteristic.

RECOMMENDATIONS

The following recommendations for further work in the Wrenshall area are suggested to better delineate the clay resources and to alter the clay material to improve its marketing characteristics. Work which should be done in the area includes:

1. Better definition of the extent of the clay resources; e.g., auger drilling on a wide spacing.
2. A more complete sampling and testing program should be done in the area of Wrenshall, Minnesota, to better define the properties of the clays; e.g., rheology, mineralogy, etc.
3. Chemical analysis of the clays should be run to determine the overall chemical content and hopefully better understand the cause for the problems with the fired color.
4. More complete firing test work should be done to better define the firing ranges of the clays.
5. Manganese dioxide, taconite concentrate, and other coloring additives should be added to the clays in order to alter the fired color of the material.

Plate 1. Raw clays and fired test bricks shown above are examples which depict the shift in the color of the clays as firing temperatures increase.

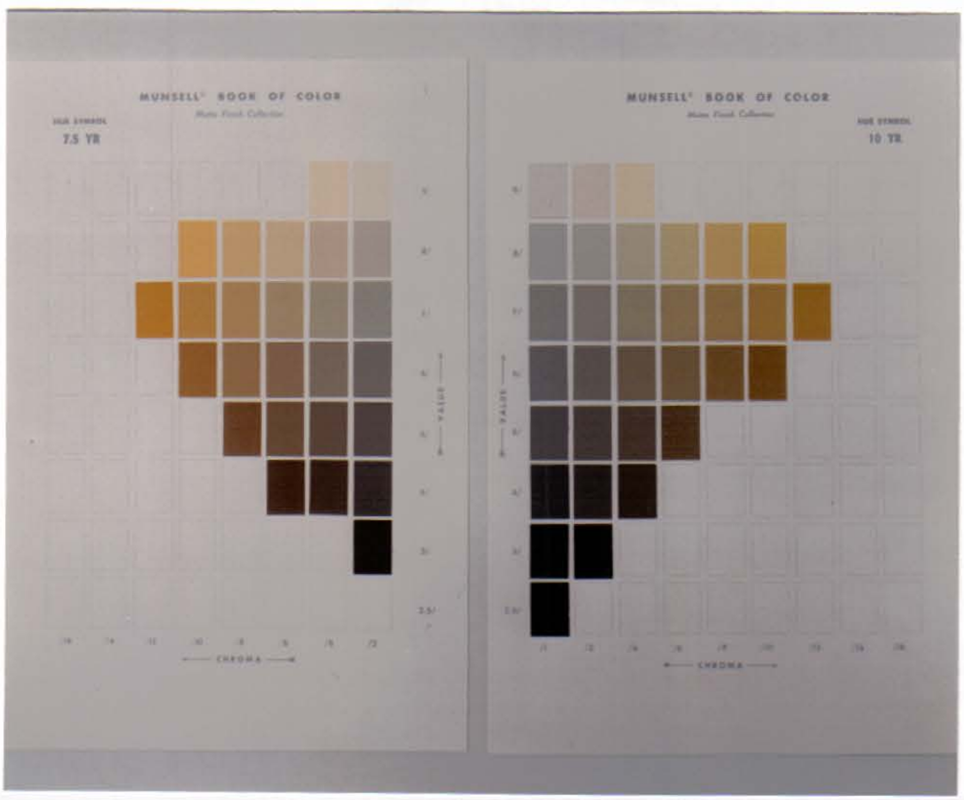


Plate 2. These pages from the Munsell Book of Color show examples of the standards from which the data in Table 1 are derived.

Immediate Testing Plan

The NRRI Minerals Division plans to continue testing the Wrenshall clays in order to better define the properties of the material for use in the construction and ceramic industries. Testing will also be completed to try to alter some of the characteristics which make the clay less useful for these purposes. Work which is being done at this time includes:

1. Chemical analysis of the clays run by Chemex Labs, Inc. for determination of the chemical composition of the clay to help understand the changes which occur in the material as it is fired.

2. X-ray analysis and SEM (scanning electron microscope) to determine the mineralogy and crystallography of the material.

3. Particle size analysis to determine the size range of the material.

4. Test bricks are being made to test the use of manganese dioxide and taconite concentrate to change the color of the fired material.

5. The test bricks will be fired in both oxidizing and reducing atmospheres to study the effect of the atmosphere on the color of the fired material. Pyrometric cones will be made in the same way to determine the proper firing temperature of each mixture of the clays and coloring agent in each atmosphere.

APPENDIX 1

TEST DATA FROM DEPARTMENT OF CERAMIC ENGINEERING
UNIVERSITY OF MISSOURI-ROLLA

UNIVERSITY OF MISSOURI-ROLLA

School of Mines and Metallurgy

Department of Ceramic Engineering

120 Fulton Hall
Rolla, Missouri 65401-0249
Telephone (314) 341-4401

October 1, 1986

Dr. John Heine
University of Minnesota
Natural Resources Research Inst.
3151 Miller Trunk Hwy
Duluth, MN 55811

Dear John:

The tabular data and a discussion is appended. As I mentioned on the phone the clays were unique in their firing behavior, especially in regard to their color and color is critical in most applications today. You might consider showing the specimens fired at 2050°F to architects to see what their reactions might be, however, I can't imagine trying to build a variety of face brick colors on these yellow-firing clays. They could be stained to a series of grays using manganese oxide and there is always the possibility of applying the various antiquing treatments and coatings popular today. But, in general, strong preference is for a base clay(s) that fires either to red shades or to buff shades. These are essential if you are going to produce architectural brick for hospitals, shopping centers, etc.

Sincerely yours,

Robert E. Moore

Robert E. Moore
Department Chairman

REM/lp
Enclosures

Report on the Testing of Clays
(Red Clay, 67/33 R/G, 60/40 R/G, Grey Clay)

I. Description of Testing

Two end number clays, described as Red Clay and Grey Clay were submitted for test to see whether they were suitable for use in the manufacture of structural clay products, especially for brick. As these clays had previously been used in blends, two intermediate formulations were also evaluated. Properties of interest in this study included:

1. extendability
2. drying shrinkage
3. firing shrinkage
4. fired porosity (absorption)
5. fired color
6. firing range

II. Test Procedures

The clays were thoroughly blended in a muller mixer, tempered with water and extruded using a laboratory model extruder into 1" x 1" cross-section bars. The wet bars were cut, weighed and marked for shrinkage and then dried at 110°C overnight. Drying shrinkage and water content were determined and 4 bars each from the 4 formulas were fired to 1850, 1950, 2050 and 2150°F, respectively, in an electric globar furnace. The fired specimens were measured for firing shrinkage and their fired color noted. they were then weighed in the saturated state to determine fired porosity. The shrinkage and porosity data were used in the assessment of firing range.

III. Discussion of Results

1. Extrudability

The red and the grey clays and the blends all extruded well over a range of moisture contents suggesting the clays have a good clay mineral content, i.e., they have good plastic properties. The grey clay requires more H_2O - is more plastic than the red.

2. Drying Shrinkage

These data show that shrinkage for the grey clay is appreciably higher than the red, 8 percent compared to 5.5%. This reflects the higher clay mineral content in the grey clay and the correspondingly higher non-plastics in the red clay. A drying shrinkage of only 5.5% is quite low.

3. Firing Shrinkage

The firing shrinkage as a function of temperature measures the rate at which the body matures. The data show no appreciable shrinkage until the 2050°F firing when shrinkages are in an acceptable 4-6% range. It was not possible to get shrinkage data at 2150°F due to warpage (high glass content).

4. Fired Porosity

The fired porosities somewhat follow the shrinkage in that they were unaltered by the 1850°F but they did reflect densification at 1950°F; the grey clay tightening up before the red. The 2050°F firing promoted much more vitrification in the gray clay (≈16% porosity) than the red (≈25% porosity).

The grey body could be considered mature at 2050°F. At 2150°F all four bodies were too glassy to be measured. The absorption of the 2050°F firings only were calculated. They are all in the well-matured range. The low values for the grey clay, 9-12%, would make it suitable for extreme freeze-thaw resistance applications.

5. Fired Color

The unusual yellow color of these bodies was surprising. The salmon color of the highly porous underfired clay was usual. Apparently the iron oxide is being bleached by calcium oxide to reduce the Fe coloration to a non-existent level. An oxide chemical analysis would be very helpful in analyzing the color origins.

6. Firing Range

The bodies, especially the grey end number tend to vitrify rather sharply although more detailed firing is needed to fully characterize the firing ranges. Modern tunnel type kilns can utilize clays with much narrower ranges than in the past.

IV. Summary Conclusions

The color of the fired clay appears to be a major problem for use as a brick clay. It's conceivable that stains and coatings could be used. It is also possible that a dense quarry-tile type product could be made from these clays. The narrow firing range shouldn't be a problem with modern tunnel-kiln firing. The good plasticity of the clays and clay blends is a plus as is the relatively low drying shrinkage.

Respectfully submitted,

Robert E. Moore

Robert E. Moore
Ceramic Engineering Department
University of Missouri-Rolla
October 1, 1986

POROSITY
(weight in grams)

RED CLAY
1850 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY
A1	134.5	83.4	171.1	41.7%
A3	135.0	84.6	173.0	43.0%
A4	136.0	84.4	173.5	42.1%

67/33;R/G
1850 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY
B2	128.6	77.3	158.6	36.9%
B3	134.7	81.5	166.8	37.6%
B4	127.9	77.5	159.0	38.2%

60/40;R/G
1850 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY
C2	139.0	85.0	174.1	39.4%
C3	133.8	82.5	167.8	39.9%
C4	139.4	85.3	174.3	39.2%

GREY CLAY
1850 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY
D1	134.4	79.7	162.4	33.9%
D2	138.0	82.4	167.7	34.8%
D4	148.9	90.1	181.7	35.8%

POROSITY
(weight in grams)

RED CLAY
1950 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY
A13	137.8	61.7	177.3	34.2%
A14	133.9	62.0	174.9	36.3%
A15	141.1	63.1	182.8	34.8%
A16	138.8	62.9	177.8	33.9%

67/33;R/G
1950 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY
B13	122.5	52.9	154.8	31.7%
B14	120.8	52.3	153.1	32.2%
B15	123.2	56.1	154.7	31.9%
B16	135.9	58.1	169.5	30.2%

60/40;R/G
1950 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY
C13	130.7	65.4	167.4	36.0%
C14	140.8	70.0	179.9	35.6%
C15	126.4	69.3	161.7	38.2%
C16	137.4	65.4	175.7	34.7%

GREY CLAY
1950 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY
D13	135.2	65.8	166.4	31.0%
D14	130.2	66.4	158.1	30.4%
D15	136.8	60.9	166.8	28.3%
D16	137.9	71.0	170.0	32.4%

POROSITY
(weight in grams)

RED CLAY
2050 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY	ABSORPTION %
A7	145.3	71.1	165.0	21.0%	13.7
A8	140.6	73.1	166.1	27.4%	17.7

67/33;R/G
2050 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY	ABSORPTION %
B5	131.9	56.7	155.9	24.2%	18.8
B6	123.8	57.4	148.0	26.7%	19.3

60/40;R/G
2050 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY	ABSORPTION %
C5	134.3	62.4	154.8	22.2%	15.6
C6	75.1	32.8	88.2	23.6%	17.3

GREY CLAY
2050 F

NO.	DRY WT.	SUSTN. WT.	SAT. WT.	POROSITY	ABSORPTION %
D6	139.0	62.8	151.8	14.4%	9.3
D7	138.6	58.8	151.6	14.0%	9.4
D8	136.9	69.9	153.5	19.9%	12.4

SHRINKAGE

RED CLAY
1850 F

NO.	INITIAL	DRIED	DR.SHRNK	FIRED	FR.SHRNK
A1	4.000	3.791	5.225	3.790	0.026
A2	4.000	3.786	5.350	3.806	-0.528
A3	4.000	3.789	5.275	3.799	-0.264
A4	4.000	3.782	5.450	3.796	-0.370

67/33;R/G
1850 F

NO.	INITIAL	DRIED	DR.SHRNK	FIRED	FR. SHRNK
B1	4.000	3.830	4.250	3.820	0.261
B2	4.000	3.836	4.100	3.824	0.313
B3	4.000	3.822	4.450	3.807	0.392
B4	4.000	3.815	4.625	3.806	0.236

60/40;R/G
1850 F

NO.	INITIAL	DRIED	DR.SHRNK	FIRED	FR. SHRNK
C1	4.000	3.751	6.225	3.750	0.027
C2	4.000	3.760	6.000	3.759	0.027
C3	4.000	3.759	6.025	3.749	0.266
C4	4.000	3.763	5.925	3.760	0.080

GREY CLAY
1850 F

NO.	INITIAL	DRIED	DR.SHRNK	FIRED	FR. SHRNK
D1	4.000	3.670	8.250	3.663	0.191
D2	4.000	3.683	7.925	3.685	-0.054
D3	4.000	3.675	8.125	3.679	-0.109
D4	4.000	3.671	8.225	3.677	-0.163

SHRINKAGE

RED CLAY
1950 F

NO.	INITIAL	DRIED	DR. SHRNK	FIRED	FR. SHRNK
A13	4.000	3.785	5.375	3.782	0.079
A14	4.000	3.769	5.775	3.768	0.027
A15	4.000	3.787	5.325	3.781	0.158
A16	4.000	3.797	5.075	3.770	0.711

67/33;R/G
1950 F

NO.	INITIAL	DRIED	DR. SHRNK	FIRED	FR. SHRNK
B13	4.000	3.840	4.000	3.817	0.599
B14	4.000	3.828	4.300	3.806	0.575
B15	4.000	3.827	4.325	3.808	0.496
B16	4.000	3.825	4.375	3.797	0.732

60/40;R/G
1950 F

NO.	INITIAL	DRIED	DR. SHRNK	FIRED	FR. SHRNK
C13	4.000	3.755	6.125	3.734	0.559
C14	4.000	3.768	5.800	3.755	0.345
C15	4.000	3.757	6.075	3.735	0.586
C16	4.000	3.745	6.375	3.733	0.320

GREY CLAY
1950 F

NO.	INITIAL	DRIED	DR. SHRNK	FIRED	FR. SHRNK
D13	4.000	3.677	8.075	3.661	0.435
D14	4.000	3.666	8.350	3.638	0.764
D15	4.000	3.662	8.450	3.654	0.218
D16	4.000	3.679	8.025	3.674	0.136

SHRINKAGE

RED CLAY
2050 F

NO.	INITIAL	DRIED	DR.SHRNK	FIRED	FR. SHRNK
A5	4.000	3.775	5.625	3.605	4.503
A6	4.000	3.780	5.500	3.497	7.487
A7	4.000	3.785	5.375	3.580	5.416
A8	4.000	3.774	5.650	3.606	4.452

67/33;R/G
2050 F

NO.	INITIAL	DRIED	DR.SHRNK	FIRED	FR. SHRNK
B5	4.000	3.837	4.075	3.697	3.649
B6	4.000	3.842	3.950	3.699	3.722
B7	4.000	3.849	3.775	3.696	3.975
B8	4.000	3.847	3.825	3.602	6.369

60/40;R/G
2050 F

NO.	INITIAL	DRIED	DR. SHRNK	FIRED	FR. SHRNK
C5	4.000	3.763	5.925	3.562	5.341
C6	4.000	3.756	6.100	3.596	4.260
C7	4.000	3.755	6.125	3.474	7.483
C8	4.000	3.752	6.200	3.453	7.969

GREY CLAY
2050 F

NO.	INITIAL	DRIED	DR. SHRNK	FIRED	FR. SHRNK
D5	4.000	3.673	8.175	3.490	4.982
D6	4.000	3.684	7.900	3.426	7.003
D7	4.000	3.674	8.150	3.419	6.941
D8	4.000	3.682	7.950	3.486	5.323

SHRINKAGE

RED CLAY
2150 F

NO.	INITIAL	DRIED	DR. SHRNK
A9	4.000	3.792	5.200
A10	4.000	3.777	5.575
A11	4.000	3.770	5.750
A12	4.000	3.790	5.250

67/33;R/G
2150 F

NO.	INITIAL	DRIED	DR. SHRNK
B9	4.000	3.847	3.825
B10	4.000	3.828	4.300
B11	4.000	3.813	4.675
B12	4.000	3.832	4.200

60/40;R/G
2150 F

NO.	INITIAL	DRIED	DR. SHRNK
C9	4.000	3.756	6.100
C10	4.000	3.761	5.975
C11	4.000	3.758	6.050
C12	4.000	3.741	6.475

GREY CLAY
2150 F

NO.	INITIAL	DRIED	DR. SHRNK
D9	4.000	3.672	8.200
D10	4.000	3.664	8.400
D11	4.000	3.685	7.875
D12	4.000	3.680	8.000


RED 2050°F
GREY 2050°F
RED 1850°F
GREY 1850°F
RED CLAY
GREY CLAY


RED 2050°F
GREY 2050°F
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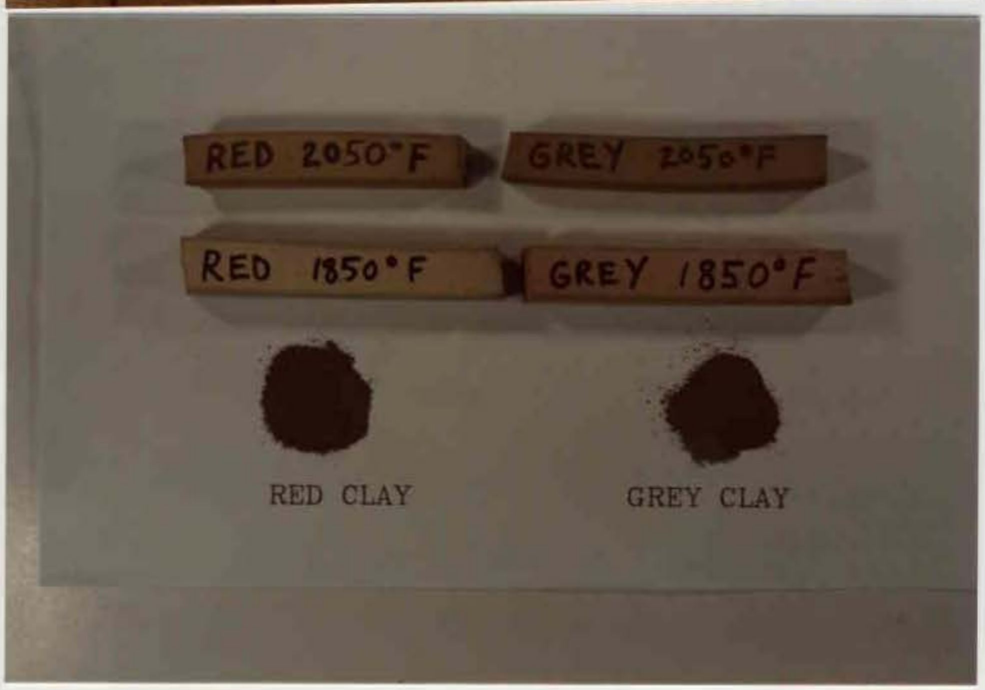
RED CLAY
GREY CLAY


RED 2050°F
GREY 2050°F
RED 1850°F
GREY 1850°F
RED CLAY
GREY CLAY


RED 2050°F
GREY 2050°F
RED 1850°F
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