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THE PURIFICATION OF MISSOURI FIRE CLAYS

By

JAMES OFFUTT

A

THESIS

Submitted To The Faculty Of The
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI
in partial fulfillment of the work required for the
Degree Of
BACHELOR OF SCIENCE IN CERAMIC ENGINEERING

Rolla, Missouri

1932

INTRODUCTION

At the present time there appears to be an unlimited supply of high grade fire clay available for most any purpose. But some time in the future there will rise the problem of purifying the low grade clay to such an extent that it will be used in high grade fire clay products.

Whatever purification method may be employed it must be applicable on a commercial scale and the cost of purification must be very low.

Until the present time little if any work along this line has been reported. Apparently no one has ever attempted and serious efforts to purify low grade fire clay. Therefore, the field is too broad to cover in a short time. It must be developed step by step.

It is the object of this research to pursue several possible and plausible methods of purification and to determine the approximate merits of each in such a manner that latter research men may know which fields show the most promise. With this object in mind we proceed.

REVIEW OF PREVIOUS WORK

So far as could be determined from a careful survey of the Journals of the American Ceramic Society and other literature, very little work has ever been done along the line of purification of fire clays. It is apparent that the majority of manufacturers consider the present supply of fire clay to be almost inexhaustible. Some of the methods used in the purification of other clays could not be used at all with fire clay.

However, Mr A.V. Bleininger included fire clay with a number of other clays in an attempt to add to the good qualities of the clay by electrical separation. His report was written for the Transactions of the American Ceramic Society. This report was printed in the 15th volume of the Transactions issued in 1913.

An abstract of the article as far as it pertains to fire clay follows:

A very thin slip was made of the material being studied. It is a well known fact that all of the particles are supposed to carry charges (electrical). When electrodes are placed in the slip the particles will seek one or the other of the electrodes.

The fire clay was ground through 40 mesh and made into a thin slip. The fire clay used was Number 3 Ohio Fire Clay. About 0.1% caustic soda was added to the slip to act as an electrolyte.

The tests of the final clay obtained gave very good res-

ults. The tests made a very good appearance with a very uniform color including no iron spots. The shrinkage was reduced appreciably.

This test apparently has no primary part in the purification of fire clays. The process simply replaces the filter press to a certain extent. The operation is very simple and this is a decided advantage. The writer mentions that there was no hope at that time of ever being able to settle ferric iron out of the slip. A follow up of the method was to be made, according to the writer, but apparently nothing was ever done to advance the work since no further reports could be found.

This method of purification may work very well for some clays but the results obtained with fire clay were not of such a nature to warrant electrical separation on a commercial scale.

RAW MATERIALS

Cone 31 has been set by leading Ceramists of the country as the dividing line between low and high grade fire clays. It is apparent that any clay which had a pyrometric cone equivalent of more than 31 would demand a higher sale price.

Any cheap method of transfixing a lower grade clay to such an extent that its P.C.E. value would be cone 31 or higher would be profitable.

In open pit mining there is always much clay of a lower quality than can be used for number one fire brick. In most open pit plants there is also a huge dump of very low grade clay filled with many impurities. Both of these conditions prevail at the A.P. Green Fire Brick Plant at Mexico, Missouri.

Samples of low grade material from the pit and a representative sample of the dump clay were used in this research.

DISCUSSION OF PROCEDURE

Several possible methods of purifying the fire clay were pursued in this research. A brief outline of each method is given below:

Original samples

A uniform sample of the original material was crushed in the rolls through 20 mesh and moulded stiff mud into standard test bars. Test cones were moulded from the material.

Disintegration

A representative sample of the original materials was pass through a squirrel cage disintegrator. The sample from the dump was divided into two portions one passed through 40 mesh and the other through a 20 mesh screen. All of the pit sample was passed through the 20 mesh screen. Standard test bars and test cones were moulded stiff mud from the materials.

Washing

A good quantity of the material was placed in a blunger and allowed to slake until nearly all of the material was in the form of a slip. The slip was then divided into four proportions ~~\$\$\$~~ ^{which were} screed respectively in the slip state through 40, 80, 100, and 150 mesh screens. The slip was allowed to dry sufficiently to mould stiff mud into test bars and test cones.

Weathering

Samples were placed in perforated pans, covered with thin cloths and set on the roof of a building to be submitted to the weather. At the end of one and two months respectively a sample of the material was crushed through 20 mesh moulded stiff mud into test bars and test cones.

TESTING THE BARS

The bars were fired bone dry in an electric drier and fired to cone 19 in a test furnace by following the firing schedule given elsewhere in this report.

Green shrinkage, fired shrinkage, green modulus, fired modulus, absorption, and P.C.E. values were determined for each group. The P.C.E. tests were run in a Fulton-Coursen test furnace.

LEGEND

ODOR--Original sample from dump.

OPCR--Original sample from pit.

CPW1--Pit sample weathered about one month.

ODW1--Dump sample weathered about one month.

OPW2--Pit sample weathered two months.

ODW2--Dump sample weathered two months.

CPSL40--Pit sample slaked and wet screened thru 40 mesh.

OPSL80-- " " " " " " " 80 "

OPSL100-- " " " " " " " 100 "

OPSL150-- " " " " " " " 150 "

ODSL40--Dump " " " " " " " 40 "

ODSL80-- " " " " " " " 80 "

ODSL100-- " " " " " " " 100 "

ODSL150-- " " " " " " " 150 "

OPDIS40--Pit sample disintegrated and screened thru 40 mesh,

ODDIS20--Dump sample disintegrated and screened thru 20 mesh.

ODDIS40-- " " " " " " " 40 "

SUMMARIZED DATA

Bars	Drying Shr.	Fired Shr.	Green Mod. of Rupt.	Fired Mod. of Rupt.	Abs.	P.C.E.	Comments
ODOR	8.4	4.2	24	2355	.23	29	Light tannish yellow-iron spots.
OPOR	4.9	4.0	0	1760	5.3	30	Yellowish brown Iron speckled
OPW1	5.3	4.6	40	1410	7.1	31	Yellowish tan Few bad iron spots
ODW1	9.6	3.8	26	1810	.68	Less than 30	Dark brown Few iron spots
OPW2	4.4	4.1	44	1260	5.9	31	Light yellow tan Iron spots
ODW2	-.9	6.2	228	2430	.81	31	Uniform dark brown
OPSL40	9.6	8.5	167	2440	.62	30-31	Chocolate brown
OPSL80	9.7	9.0	162	2550	0	30-31	Uniform greenish brown
OPSL100	9.4	9.2	190	2320	0	Less than 30	Uniform dark green Black cored
OPSL150	9.2	9.0	180	1840	0	"	" " "
ODSL40	13.3		226			Less than 30	All ODSL bars were overfired, bloated cracked, swollen, and dis-colored. ODSL100 was laminated somewhat.
ODSL80	13.1		560			"	
ODSL100	14.0		364			"	
ODSL150	13.9		610			"	
OPDIS40	5.8	5.5	0	1220	9	31-32	Brown tan Few iron spots
ODDIS20	6.5	5.5	104	2920	1.2	31-32	Light tan
ODDIS40	8.1	5.9	95	3420	0	31-32	Ligth speckled tan

LEGEND OF SYMBOLS USED IN DATA

- G.L.--Green length of bar given in inches.
- D.L.--Dry length of bar given in inches.
- F.L.~~3~~²Fired length of bar given in inches.
- D.S.--Dry shrinkage of bar given as a percentage of the dry length.
- F.S.--Fired shrinkage of bar given as a percentage of the dry length.
- B.--Breadth of break in the modulus of rupture test.
- D.~~3~~²Depth of break in the modulus of rupture test.
- L.--Length of span in the modulus of rupture test.
(Note: D, B, and L are in inches.)
- G.M.R.--Green modulus of rupture in pounds per square inch.
- F.M.R.--Fired modulus of rupture in pounds per square inch.
- D.W.--Dry weight of bar in absorption test.
- W.W.--Wet weight of bar in absorption test.
- Abs.--Absorption in percentage with reference to the dry weight.
- P.C.E.--Pyrometric cone equivalent.

Data Sheets

Bar - G.L.- D.L.- F.L.- D.S.-F.S.-B.- D.- Lo- G.M.R.-F.M.R.-D.W.-W.W.-ABS.

ODOR

1	5	4.60	4.48	8.7	2.6	.86	.90	200		2160	75	75	0
2	5	4.62	4.36	8.2	5.6	.92	.87	210		2300	72	72.5	.69
3	5	4.62	4.42	8.2	4.3	.90	.88	210		2260			
4	5					.95	.92	2407	230				
5	5					.90	.85	235		2700	75.5	75.5	0
Average	5			8.4	4.2				230	2355			.23

OPOR

1	5												
2	5												
3	5	4.75	4.56	5.2	4.0	.94	.85	160		1760	94	99	5.3
4	5												
5	5	4.78		4.6					0				
Average	5			4.9	4.0				0	1760			5.3

OPW1

1	5	4.76	4.56	5.1	4.2	.95	.88	110		1010	82	88	7.3
2	5	4.72	4.48	5.9	5.1	.93	.85	135		1500	84	90	7.1
3	5	4.77		4.8									
4	5					.98	.92	4.5	40				
5	5					.93	.89	170		1730	86.5	92.5	6.9
Average				5.3	4.6				40	1410			7.1

ODW1

1	5	4.57	4.40	9.6	3.7	.94	.85	195		2120	65	65.5	.72
2	5	4.56	4.40	9.6	3.5	.94	.85	130		1500	68.5	69	.73
3	5	4.57	4.38	9.6	4.2	.90	.86	205		2300	70.5	80.5	0
4	5					.94	.90	26.3	260				
5	5					.92	.86	120		1320	73	73.5	.68
Average				9.6	3.8				260	1810			.53

Data Sheets

Bar - G.L.- B.L.- F.L.- D.S.-F.S.-B.-D.-Lo.- G.M.R.-F.M.R.-D.W.-W.W.-ABS.

OPW2

1	5	4.79	4.63	4.4	3.4	1.02	.96	105	840	100	105	5	
2	5	4.79	4.56	4.4	\$\$\$	4.4	1.00	1.00	170	1220	93	99.5	6.9
3	5	4.79	4.58	4.4	4.4	1.04	1.00	180	1430	91	96	5.5	
4	5					1.12	1.04	741	44				
5	5					1.02	.98	205	1560				
Average			4.4	4.1					44	1260		6.5	

ODW2

1	5	5.05	4.74	-.9	6.1	1.0	.99	280	2160			
2	5	5.05	4.75	-.9	6.1	1.0	.95	370	3100	105	105.5	.47
3	5	5.05	4.72	-.19	6.4	1.06	1.03	34.3	228			
4	5	5.05	4.72	-.9	6.4	1.00	.98	260	2020	93	94	1.08
5	5					1.00	.97	310	2440	123.5	124.5	.89
Average				-.9	6.2				228	2430		.81

OPSL40

1	5	4.55	4.13	9.9	7.1	.87	.80	270	3640	94	95	1.05
2	5	4.59	4.15	9.0	9.6	.94	.80	120	1510	76.5	77	.64
3	5	4.55	4.15	9.9	8.8	.92	.80	220	2800	72	72	0
4	5					1.04	.96	21.2	167			
5	5					.90	.88	170	1820	65.5	66	.77
Average				9.6	8.5				167	2440		.62

OPSL80

1 ^D	5	4.54	4.18	10.1	7.9	.89	.81	185	2380	86	86	0
2	5	4.55	4.12	9.9	9.4	.91	.84	165	1930	70	70	0
3	5	4.58	4.18	9.2	8.7	.95	.84	300	3350	96	96	0
4	5					1.03	.95	20	162			
5												
Average				9.7	9.0				162	2550		0

Data Sheets

Bar - G.L.- B.L.- F.L.- D.S.-F.S.- B.-D.-Lo. $\frac{1}{2}$ B.M.R.-F.M.R.-D.W.-W.W.-ABS.

OPSL100

1	5	4.57	4.15	9.4	9.2	.93	.82	250		3000	86	86	0
2	5	4.58	4.18	8.7	9.4	.95	.82	140		1650	72	72	0
3	5	4.58	4.14	9.4	9.6								
4	5					1.06	.98	25.6	190				
5													
Average				9.4	9.2				190	2320			0

OPSL150

1	5	4.58	4.18	9.2	8.7	.92	.83	230		2720	95	95	0
2	5	4.59	4.15	9.3	9.6	.94	.85	105		1160	95	95	0
3	5	4.57	4.18	9.2	8.7	.94	.89	190		1920	81.5	81.5	0
4	5					1.06	.95	23	180				
5	5					.95	.86	150		1580	73	73	0
Average				9.2	9.0				180	1840			0

ODSL40

1	5	4.42		13.1									
2	5	4.40		13.6									
3	5	4.41		13.4									
4	5					.92	.87	20.9	226				
5													
Average				13.3					226				

ODSL80

1	5	4.42		13.1									
2	5	4.42		13.1									
3	5	4.42		13.1									
4						.92	.87	52.4	560				
5													
Average				13.1					560				

DataSheets

<u>Bar</u>	<u>G.L.</u>	<u>D.L.</u>	<u>F.L.</u>	<u>D.S.</u>	<u>F.S.</u>	<u>B.</u>	<u>D.</u>	<u>Lo.</u>	<u>G.M.R.</u>	<u>F.M.R.</u>	<u>D.W.</u>	<u>W.W.</u>	<u>Abs.</u>
ODDIS40													
1	5	4.62	4.36	8.2	5.6	.88	.84	250		3020	58	58	0
2	5	4.62	4.34	8.2	6.0	.90	.83	300		3630	60.5	60.5	0
3	5	4.64	4.35	7.8	6.3	.90	.84	305		3600			
4	5					.95	.91	10	95				
5													
Average				8.1	5.9				95	3420			0

Data Sheets

Bar - C.L.- D.L.- F.L.- D.S.-F.S.- B.-D.-Lo.-G.M.R.-F.M.R.-D.W.-W.W.-ABS.

ODSL100

1	5	4.40		13.6									
2	5	4.38		14.2									
3	5	4.38		14.2									
4	5					.90	.83	30	364				
5													
Average				14.0					364				

ODSL150

1	5	4.39		13.8									
2	5	4.39		13.8									
3	5	4.37		14.3									
4	5					.89	.83	50	610				
5													
Average				14.1					610				

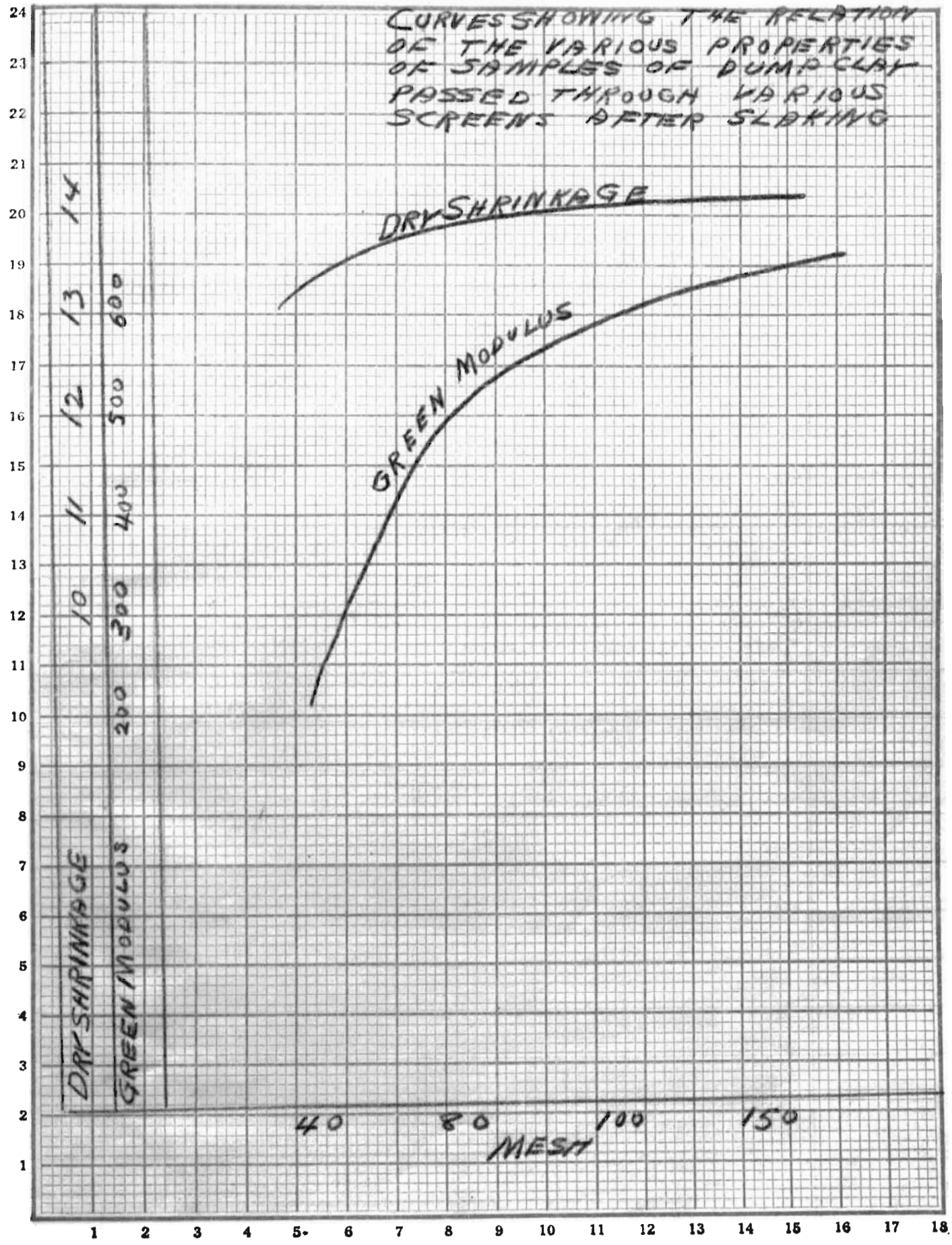
OPDIS40

1	5	4.66	4.42	7.3	5.2	.86	.86	110		1300	89	97	9
2	5	4.78	4.50	4.6	5.9	.88	.86	100		1140	78	85	9
3	5	4.74		5.5									
4	5												
5													
Average				5.8	5.5				0	1220			9

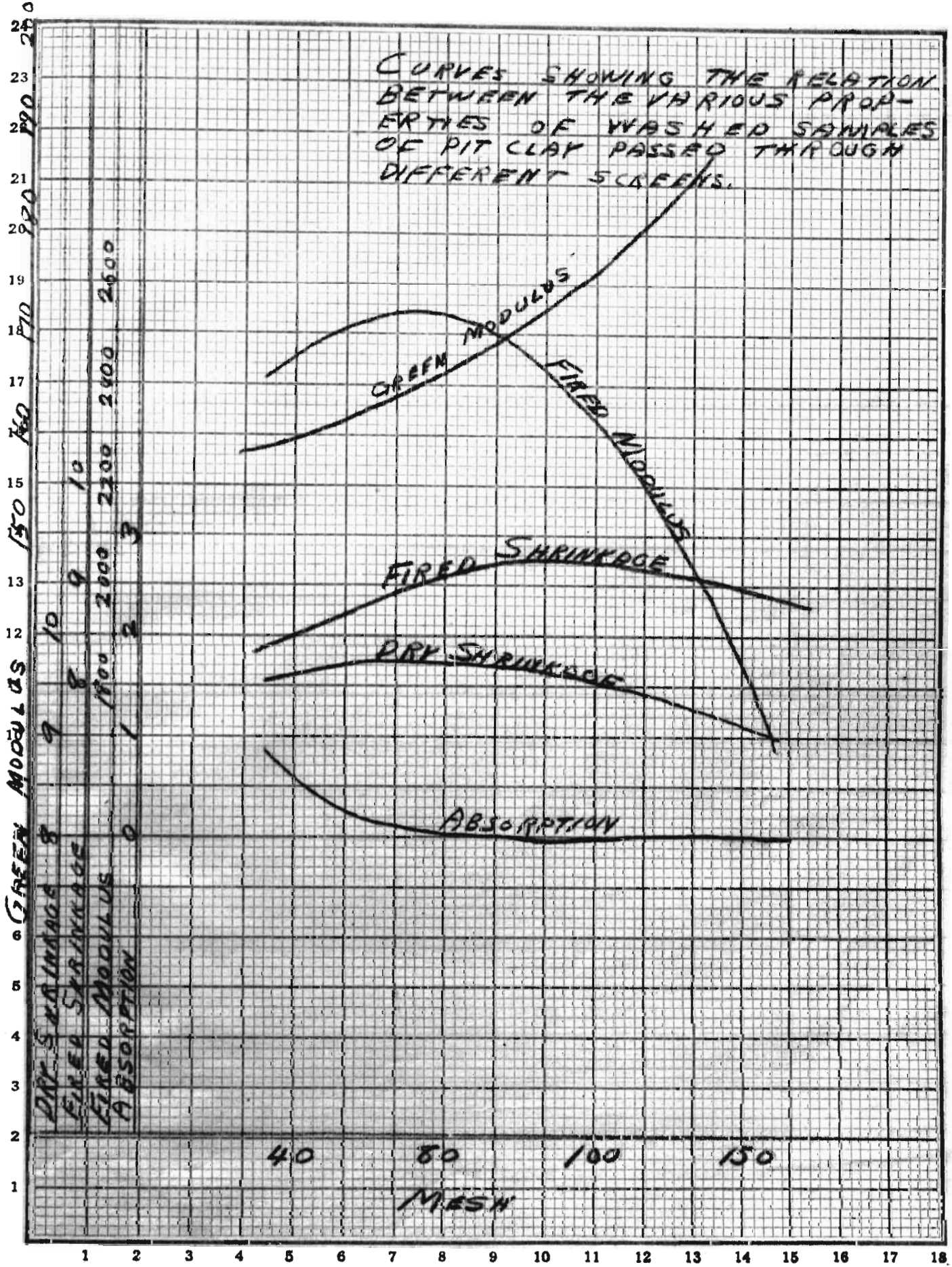
ODDIS20

1	5	4.68	4.44	6.8	5.2	.91	.88	220		2350	62.5	71	2.1
2	5	4.68	4.42	6.8	5.6	.89	.86	290		3340	88	88.5	.57
3	5	4.72	4.44	5.9	5.9	.90	.85	260		3020	78	78	0
4	5					.96	.92	11.2	104				
5	5					.90	.87	270		2960	72	73.5	2.1
Average				6.5	5.5				104	2920			1.2

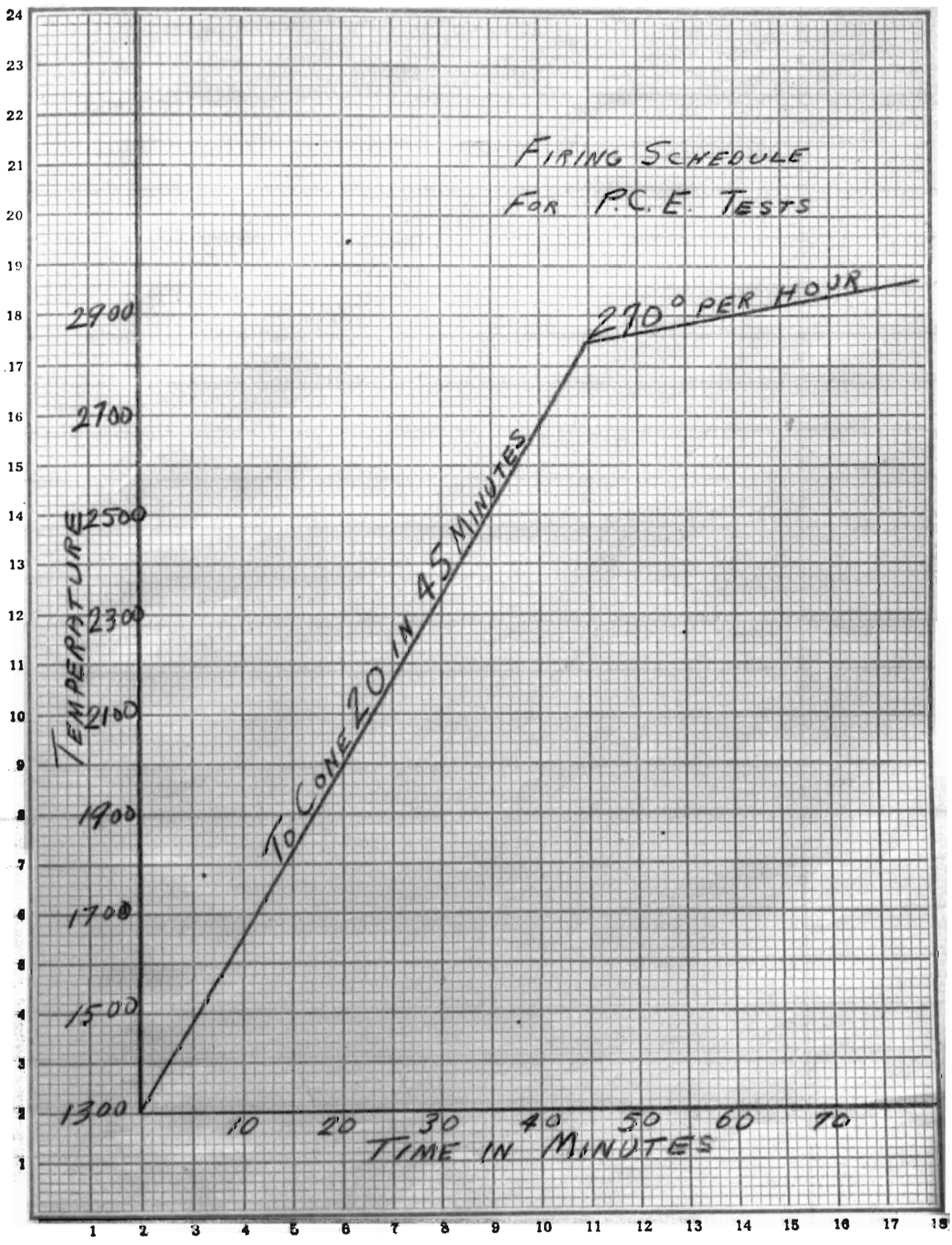
CURVES SHOWING THE RELATION
 OF THE VARIOUS PROPERTIES
 OF SAMPLES OF DUMP CLAY
 PASSED THROUGH VARIOUS
 SCREENS AFTER SLAKING



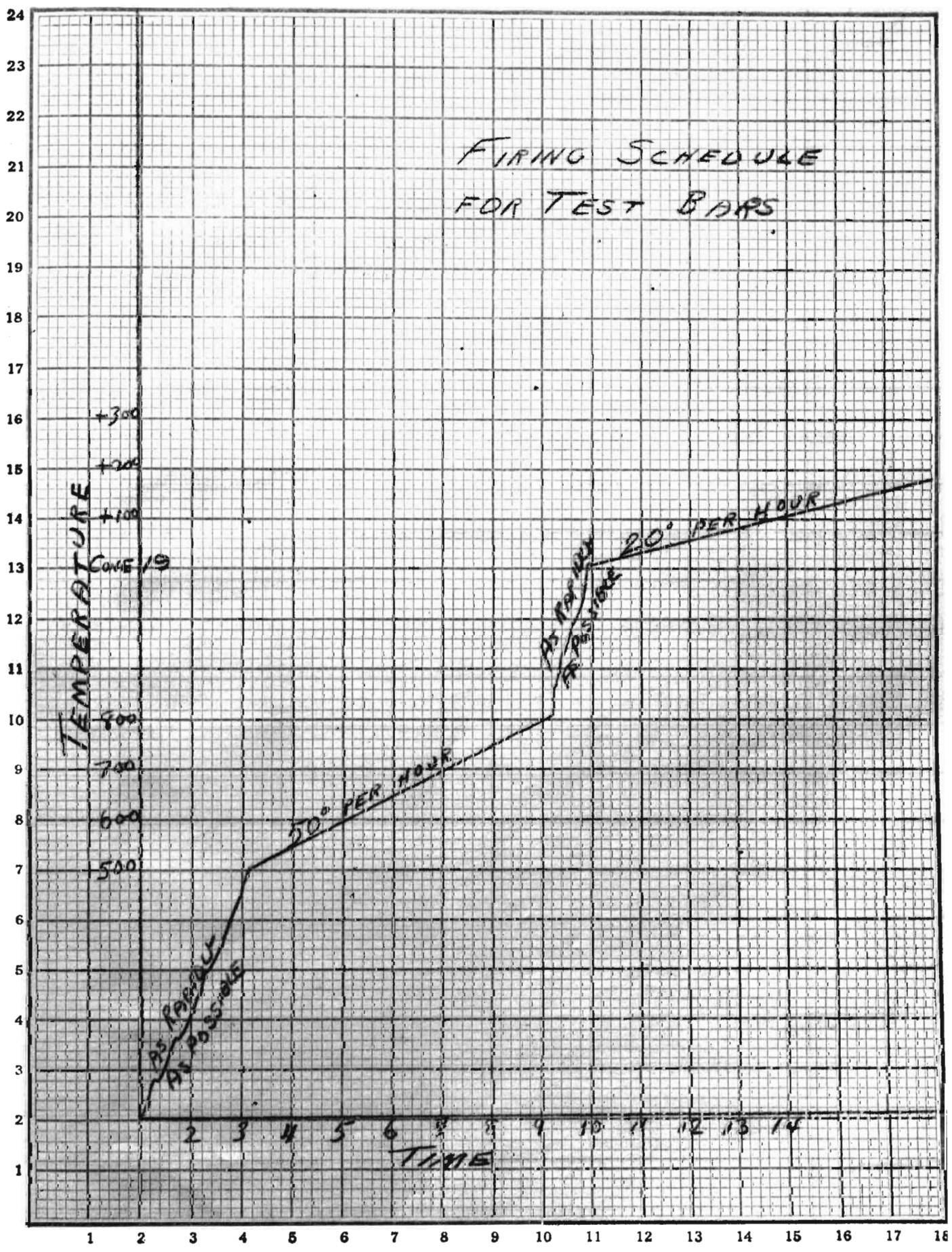
CURVES SHOWING THE RELATION BETWEEN THE VARIOUS PROPERTIES OF WASHED SAMPLES OF PIT CLAY PASSED THROUGH DIFFERENT SCREENS.



FIRING SCHEDULE
FOR P.C.E. TESTS



FIRING SCHEDULE FOR TEST BARS



Discussion of Data

The firing schedule used for the tests was evidently too fast and the bars showed signs of overfiring in nearly every case. The schedule allowed for volatilization and soaking of the heat near the maturing temperature. In the case of the washed dump clay the bars were so badly broken down that no tests at all could be obtained. The bars which were made from the disintegrated material showed the least signs of overfiring. This indicates that the quality of these bars is superior to the remainder. However, the maturing temperature was too high and much of the data will be distorted.

The test cones for P.C.E. determination were all made in the same manner but much trouble was encountered with the furnace. Indicative results were obtained and this was all that was desired. In the P.C.E. was below cone 30 no definite value was determined.

The longer weathering tests indicated that the P.C.E. might be raised somewhat by an extended weathering period. The pit samples showed much promise along this line although the dump samples showed very little signs of improvement.

In the washed materials the cone equivalent was fairly good for the coarser meshes but the finer mesh material gave a much lower fusion point. Washing lowered the fusion point in nearly all cases and so washing may help the clay in some ways but not so far as the fusion point is concerned.

The disintegrated material showed the most promise. The quality of the material was greatly increased. The strength was good and the P.C.E. was cone 32. This indicates the reason for the little overfiring on the part of these bars.

shrinkage
Drying is relative but it is interesting to note that in the case of the dump clay after one month of weathering about 10 percent drying shrinkage was evident while after two months weathering the same clay showed 1 percent expansion on drying.

The washed clays alone gave excessively high drying shrinkage. Some of them ranged as high as 13 percent which is too high for fire clays.

The green modulus is important only to a limited degree. Only the original pit sample and the disintegrated pit sample gave too low green modulus for good fire clay.

The fired modulus dropped in the weathered material as the weathering progressed. This indicates that some of the fluxes were washed out of the material. The washed samples showed a higher modulus but the material through the finer meshes tended to drop indicating that the fluxes present were of a fairly coarse texture. The disintegrated material developed a wonderful fired modulus.

The importance of absorption depends on the use for which the ware is intended. The absorptions in this group ranged from 0 to 9 percent as shown on the data sheets.

The accompanying curves are self explanatory.

CONCLUSIONS

Although the tests in this research were not complete or entirely conclusive it is evident that the dump clay is valuable in many respects and the pit clay has excellent chances of developing into a high grade fire clay.

All of the methods of purification pursued gave a better clay although in many cases the clay was not developed into a better or higher grade fire clay. Some of the purified products would work excellently in some other phase of the ceramic industry.

Of all of the methods employed the disintegration of the dump sample gave by far the best results. This method of purification made the necessary change in P.C.E. value from below to above cone 31 and at the same time developed a low absorption and a high strength both in the green and the fired state.

The writer recommends this method for further research in an effort to develop low grade fire clays.