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The development of a new process for the manufacture of insulating refractories

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THE DEVELOPMENT OF A NEW PROCESS
FOR THE MANUFACTURE OF INSULATING
REFRACTORIES

BY

MILES EDWARD TYRRELL

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

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Approved by

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of
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The Development of a New Process For the Manufacture of Insulating Refractories

Because of the importance of economy in fuel consumption, insulating refractories have been receiving serious consideration in the heat treating field. The insulating brick is a recent development and is still in the neophyte stage. Insulators such as infusorial earth have been used back of refractories for a number of years, but only within the last five years has insulation on the inside of the furnace been considered and this has given rise to many new problems in the production of insulators.

Some good insulating brick have been made for use at low and medium temperatures.¹ Up to the present time these bricks have been replacing firebrick with little change in furnace design but at present there is a tendency to adapt furnaces to take full advantage of the properties of these brick.

I. Rueckel W. C. "Physical Properties of Some Insulating Brick." Jour. Amer. Cer.

Soc. V18, 18, 1935

The chief limitation of insulating brick used in modern furnaces, is their lack of resistance to slags at high temperatures. Other disadvantages are low resistance to abrasion, low spalling resistance, and poor mechanical strength.

The use of refractory insulating brick in furnaces produces savings in fuel and heating time as much as 60 to 70 percent. These economies are a result of the low thermal conductivity of the brick and its light weight which is responsible for its low heat capacity.

None of the porous types of brick can be used where they will be exposed to abrasion, slagging action or flame impingement. But where an abrasive combustion cannot be avoided, a facing material can be applied that will stand the direct force of the flame.

One of the most important phases of insulating brick manufacture is the machining to size. When this type of brick is fired it deforms considerably and therefore must be ground to size. The grinding is accomplished by means of emery and carborundum wheels.

A nine inch wall of average insulating brick has about the same heat resistance as $22\frac{1}{2}$ inches of firebrick. This fact makes a much smaller grate area possible and at the same time production is speeded up.²

The characteristics desired in an insulating refractory are, in order of their importance:

1. Uniformity of pore structure.
2. Minimum shrinkage.
3. Spalling resistance.
4. Physical strength.
5. Insulating value.
6. Thermal capacity.³

Four general methods have been used to produce insulating refractories:

1. Formation of bubbles in the soft mix.
2. Addition of porous material to the clay body.
3. Burning out particles of organic matter.
4. Sublimation of solid particles in the mix.

2. Wood, T. E., "Light Weight Cellulose Brick Made for Furnace Installation", Ceramic Age, V26, (96-98) Sept. 1935

3. Hepburn, W. M., "Insulating Refractories", Jour. Amer. Cer. Soc. V18, 1, 1935.

The method used to produce bubbles are varied: in one method lime is used, it is added to the mix and then is acidified forming CO_2 , another method is the use of aluminum and an alkalis to form hydrogen bubbles ⁴.

J. R. Parsons has just patented a new process for making light weight brick by the bubble method. In his method an alkalie reacting substance such as black liquor is added to a mixture of clay and water to effect deflocculation of the clay, a tenacious foam is added and a reagent such as gypsum is incorporated to produce a delayed flocculation with entrapment of foam bubbles, the mixture is then shaped, dried and fired ⁵.

The most common method of producing insulation brick is method number three above. By this procedure some organic material (wool, cork, dust, peat or coal) is mixed with the clay and then burned out leaving open spaces in the body. This method tends to make the resulting brick less refractory because of the fluxing of the ash of the organic material ².

2. Wood, T. E., "Light Weight Cellulor Brick Made for Furnace Installation", Ceramic Age, V26, (98-98) Sept. 1935.

4. Norton, F. H., "The Manufacture and Use of Insulating Firebrick in the United States", Trans. Cer. Soc. Eng., V35, 7, 1937.

5. Parsons, J. R., "New Plastic Insulating Refractories", Brick and Clay Record, V85, (203-7) Dec. 1934.

PURPOSE

In as much as the grinding of insulating brick to size after the firing operation is one of the most important and at the same time one of the most expensive operations involved in their manufacture, it was felt that the development of a method for manufacture of insulating brick which would do away with the necessity of grinding after firing would be a very worth while project.

With this object in mind it was decided that the logical method would be to incorporate the pore structure in the mix while green and at the same time to add some agent which would prevent loss of the pore structure before the body was strong enough to support it's own weight.

We decided to produce the pore structure in the brick by means of a soap froth and to add liquid paraffine to the slip before it was poured into the mold. The paraffine would solidify quickly and permit the removal of the green brick from the mold with no danger of its slumping or losing its shape before being fired.

Materials used:

The clay used in this study was "Empire" fire clay from A. P. Green Firebrick Company in Mexico, Missouri.

The soap used for the froth was a granulated soap costing ten cents per pound, F. O. B. the Proctor and Gamble wholesale house at St. Louis, Missouri.

Paraffine manufactured by the Standard Oil Company and selling for nine and a half cents per pound in small lots was used as the setting agent.

Method of procedure:

The first set of test bodies was made up to determine the proper amounts of each ingredient which should be used to give the best green body.

The water, soap and electrolyte were heated to 70°C on the hot plate. When the mixture reached this temperature it was whipped with an egg beater until a good froth was obtained. The clay which was ground to pass 60 mesh was added slowly and the whipping continued until the mixture was a homogeneous mass. After the clay had been added the paraffine was introduced and the slip poured into cold beakers.

The series of bodies given in Table 1 were used in this part of the study.

TABLE 1

Body	1	2	3	4	5	6
Clay	75	75	75	75	75	75
Water	25	25	25	25	25	25
Na ₂ CO ₃	1	1	1	1	1	1
Paraffine	0	5	10	0	5	10
Soap	2 $\frac{1}{2}$	2 $\frac{1}{2}$	2 $\frac{1}{2}$	5	5	5

The proportions in the above table are given as parts.

DISCUSSION OF RESULTS

After permitting the test pieces to dry in the beakers they were taken out and examined so that the best body might be determined.

The bodies which contained only 2 $\frac{1}{2}$ parts of soap did not have a very uniform pore structure, this indicated that a body with a greater percentage of soap must be used.

The bodies which contained 5 parts of soap had a uniform pore structure and the size of the pores seemed to be very good, therefore it was decided that approximately this percentage of soap should be used in later studies.

(8)

The bodies which contained no paraffine did not retain their pore structure when poured into the mold, this proved that a setting agent was necessary in the production of this type of brick. The bodies which contained ten parts of paraffine showed an excess which floated to the top of the mix before it cooled. The bodies containing five parts of paraffine kept their pore structure very well and since there was no noticeable excess it was decided to use approximately this percentage in the future studies.

PROCEDURE 2

When an attempt was made to make a full sized brick using the batch decided upon in the first part of the study it was found that the proportions were not proper to give the best results, so it was decided to make up several full size brick and vary the batch composition somewhat from that originally decided upon.

It was also found that the egg beater was not large enough to thoroughly beat the mixtures so a Kitchen Aid mixer was obtained for the mixing.

The water and soap were heated as in procedure 1 and then beaten into a good froth. It was found that when the froth just filled the kettle, the best results were obtained. After the soap was well beaten, the clay and the liquid paraffine were introduced and the slip poured into the mold.

It was decided that for best results a collapsible mold should be used, so a wooden mold with four folding sides was used.

The series of bodies used in this part of the study are given in Table 11.

(10)

TABLE 2

Body # 1

Constituent	Weight in grams	%
Clay	1500	92.5
Soap	60	3.7
Paraffine	60	3.7
Na_2CO_3	1.5	.1
Water	450	

Body # 2

Clay	1500	92.0
Soap	70	4.3
Paraffine	60	3.6
Na_2CO_3	1.5	.1
Water	450	

Body # 3

Clay	1300	91.0
Soap	70	4.7
Paraffine	60	4.2
Na_2CO_3	1.5	.1
Water	450	

TABLE 2 (cont)

Body # 4

Constituent	Weight in grams	%
Clay	2000	91.0
Soap	105	4.8
Paraffine	90	4.1
Na_2CO_3	4	.1
Water	675	

Body # 5

Clay	1800	90.0
Soap	105	5.25
Paraffine	90	4.5
Na_2CO_3	4	.25
Water	675	

These bodies were left in the mold for one hour, after which they were taken from the mold and air dried for two weeks. When thoroughly air dried the test brick was dried at 110° for one week and then fired to 1300°C , in a high temperature, oil fired furnace.

DISCUSSION OF RESULTS

In Body # 1 the paraffine solidified before being poured into the mold. This resulted in a dense body whose pores were uneven and much too fine.

This body had a good pore structure even though percentage of soap was not great enough and the test piece collapsed after about 5 minutes in the mold.

Body # 3 was stirred rapidly just before pouring, resulting in a very good pore structure. This body was less dense than either of the first two but the amount of soap froth was still not great enough and the brick collapsed.

Body # 4 contained too much clay in proportion to the other constituents. This resulted in a dense body with poor pore structure.

Body # 5 was whipped at an intermediate speed and had a very good pore structure. This body set well and just filled the mold.

It was decided that Body # 5 was the body which would give the best insulating brick so several test pieces were prepared using this composition.

They were dried and fired as stated in the procedure.

These test pieces were badly cracked in the firing process and therefore were of no value for test purposes. The pieces were also covered with a brown scum and hard scale which would make them useless with out first grinding the six surfaces. The fired pieces had a good pore structure so it was decided to keep the same body as a base in future study.

PROCEDURE 3

In this study the composition of Body # 5 in Table 2 was used as the base.

The formation of the scale on the test pieces which were fired in the preceding study had to be eliminated if the purpose of the investigation was not to be defeated. It was decided to add boric acid to the mix to repaporize the alkaili in the soap during the firing process.

The soap and water were heated to 70°C as in the previous studies and frothed in the mixer. Then the clay which had also been preheated to prevent the solidification of the paraffine before the slip was poured,

(14)

was added, the paraffine introduced and the slip poured into the mold as in Procedure 2.

It was estimated that 10% of Boric acid would be required to neutralize the action of the soap so this amount was added to the mix when the clay was introduced into the mixer.

After air drying for 2 weeks and drying in the electric drier for 1 week the test pieces were fired to cone 10 in the high temperature furnace.

Since the above mixture is the one which was used for the bricks on which the tests were run, the following cost account will be the only one rendered.

Cost of raw materials per thousand bricks:

3334 pounds of flint clay @ .3¢	= \$10.00
200 pounds of paraffine @ .9½¢	= 19.00
100 pounds of soap @ .10¢	= 10.00
Total cost per thousand	= \$39.00

RESULTS OF PROCEDURE 3

The test pieces which were prepared and fired using this procedure were quite badly cracked but it was possible to get several pieces of the fired brick which were large enough to make the necessary tests for physical properties.

The addition of the boric acid accomplished its purpose since there was no scum or scale on the fired specimens. The addition of the boric acid also seemed to aid in holding the pore structure while the brick was in the green state.

The tests run on the fired brick were:

1. Fired modulus of rupture.
2. Fired crushing strength.
3. Insulating value.
4. P. C. E.
5. Bulk specific gravity.

The above tests were also run on 8 different samples taken from the insulating refractory bricks manufactured by three of the largest producers in the United States. The purpose of running these tests were to obtain a comparison between standard bricks and the new brick.

TEST METHODS

Modulus of Rupture.

The cross breaking strength of the fired brick was obtained by sawing pieces 6" x 1" x 1" from the brick and breaking them on the laboratory cross breaking machine.

Fired Crushing Strength.

The fired crushing strengths were determined by use of a modification of the cross breaking machine. Pieces 1" x 1" x 1" were sawed from the test pieces and placed between 2 heavy metal plates. The bottom plate was placed on the knife edges and the breaking edge of the machine was rested on the top plate. Weight was then applied at a uniform rate until the test piece gave way.

Insulation Value.

Pieces of the brick 6" x 1" x 1" were sawed from the test specimens, and a hole $\frac{1}{2}$ " deep was bored into the piece the long way, to take care of a thermometer. The test piece was placed in a 1" x 1" opening in the furnace door and the furnace fired for 24 hours at 1800°F. At the end of the 24 hour period the temperature indicated by the thermometer was noted and the loss of heat through the brick calculated in B.T.U's. in the usual way.

Softening Point.

The softening point of the brick was found using the regular A. S. T. M. standard test, C 24 - 33.

Bulk Specific Gravity.

The bulk specific gravity of the test pieces were found by using the weight and volume of a 2" cube of the paraffine coated test brick in kerosene.

SUMMARY OF DATA

Brick	Fired M of R #/sq.in.	BTU Loss per sq. ft. per hour	Fired Crushing Strength #/sq.in.	P.C.E.	Bulk Sp. Gr.	Color
EF-22	133	1400	300	27	.690	White
Ef-21	250	1458	293	28	.698	White
A-25	141	1511	273	23	.698	White
G-25	28	1700	262	30 $\frac{1}{2}$.724	Cream
Non-par.	81	1170	134	5 $\frac{1}{2}$.459	Red
HW-111	155	1173	384	14	.815	Brown
HW-111-A	77	1400	231	13	.721	Red
HW-777	36	1017	123	7	.559	Red
Average	113	1354	250	16	.671	
Research Brick	210	1506	412	32	.931	Cream

The dry crushing strength of the research brick is 40 pounds per square inch.

The total shrinkage is 25.52%.

Bricks EF-22, EF-21, A-25 and Non-par, were samples of insulating refractory brick manufactured by the Armstrong Cork Brick Company. Brick G-25 was manufactured by the A. P. Green Firebrick Company. Brick HW-111, HW-111-A and HW-777 were manufactured by the Harbison - Walker Refractories Company.

DISCUSSION OF DATA

In comparing the average of the data taken on the bricks of other companies and the brick made by Procedure 3 it may be seen that this brick equals and even surpasses the best brick on the market in every one of its important physical properties.

A brick with much greater mechanical strength than the brick manufactured today would be a great help to industry. The new brick is much stronger than any of the others which were tested. This fact is probably due to the very fine and regular pore structure which made up the brick.

The heat loss of the brick compared favorably with the loss through other brick and although it seems to be quite high the insulating value of the brick is as good as is needed.

The brick is somewhat heavier than the other bricks tested but this is to be expected with the greatly increased strength.

The total drying and firing shrinkage of the brick is 25.52%. This shows that there is very little loss of pore structure in the drying and firing operations and it should not be difficult to produce a uniform brick which would require no grinding.

CONCLUSIONS

1. The proportions of the clay, soap and paraffine used in the brick are an important factor in determining the structure of the fired brick.
2. The physical properties of the brick compare very favorably with the physical properties of all the bricks tested.
3. The cost of the brick would not be so great so to make it un-economical to manufacture.
4. Considering the cost and the physical properties the manufacture of the brick would be simple, practical and profitable.
5. The brick can be made by Procedure 3 and will require no grinding to shape after firing.

RECOMMENDATIONS FOR FURTHER RESEARCH

The main problem which is still unsolved in the production of this brick is its tendency to crack in the drying process.

The cracking is very likely due to the manner in which the brick is dried since no strains are set up in the molding which would tend to cause cracking. Because of this I would suggest that several drying behavior test be run on the green brick, in this way the correct temperature and drying rate could be determined. I might also suggest that a procedure of drying be used which would permit turning the brick over at regular and short intervals.

The brick had a tendency to stick to the bottom of the mold and I would suggest that several lubricants be tried until one is found which will release the brick with no tearing.

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