1900

Thesis for degree of Bachelor of Science in Mining Engineering [a fire clay of Missouri]

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THESIS

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—IN—

MINING ENGINEERING.

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The clay examined is a fire clay of Missouri, varying in color from white, in pure specimens, to yellow or brown when stained with iron. It is opaque with a luster, varying from dull to pearly, an argillaceous odor and unctous feel.

, It has a hardness of 2.5, scratching gypsum but being scratched by calcite. In the dry state it is brittle, breaking with a conchoidal fracture. If placed in water it may be crumbled between the fingers into pieces the size of a pea, always showing the conchoidal fracture. It will not become plastic upon standing in water in this condition but must first be ground.

The specific gravity was determined in two ways. First, direct weighing, second, by the use of a pygnometer. In the first case a piece was weighed first in air, then in distilled water. The first weight divided by the difference of the two gave the specific gravity.

A pygnometer consists of a bottle with a ground glass stopper ending in a tube with a fine opening. The flask was filled with distilled water and the stopper put in place. The water overflowing was carefully wiped away and the flask weighed. The substance previously powdered and weighed was carefully introduced, causing an overflow equal to the amount of water displaced by the clay. After again replacing the stopper and wiping the flask was weighed.

The difference between the last weight and the weight of the substance, subtracted from the weight of the flask filled with water.

The weight of the clay used divided by this gave the specific gravity.

Table of Specific Gravity Results.

No.	Method	Weight of clay	Weight of Displaced Water.	Specific Gravity
1	Direct weigh.	6.57 g.	3.34 g.	197
2	"	10.21	5.10	2.00
3	"	8.03	3.72	2.15
4	"	11.77	5.75	2.05
5	"	3.19	1.49	2.14
6	By pygnometer	5	2.56	1.95
7	"	5	2.55	1.96
8	"	5	2.40.	1.98

The object of first method is that the clay is porous and required some time to become thoroughly saturated with water. Meanwhile particles separate from the mass, thus diminishing the weight of the substance in water and lowering the specific gravity. This was practically noticable in Number one and two. probably the higher results are the more accurate.

Test of Tensile Strength.

A quantity of the clay was ground finely enough to pass a hundred mesh sieve, to be used in this and some of the succeeding tests. When mixed with water it became plastic and could readily be moulded into briquetted. These were moulded into shape, giving one square inch cross section at its smallest part and fitting the Riehle Bros. Testing Machine.

The following table shows theresult after a week's drying:

Table of Tensile Strength.

No.	Strength.
1	65#
2	38#
3	65#
4	72#
5	68#
6	68#
7	36#
8	65#

Number two and seven showed decided flaws at point of rupture. Throwing these out as valueless the average of the remaining six is sixty-seven pounds. While it is interesting to find that a clay will develope such strength,it would seem to be of no practical value on account of the shrinkage and liability to crack on drying.

Eight other briquettes were made and after drying a week were heated to redness in a muffle . Cracks developed which destroyed their strength. This was probably due to the water still contained which might have been driven off by gentle heating at first.

Absorption Tests.

Rectangular tin dishes were perforated on the bottom, measuring 4 1/2" x 2 1/4" x 1 1/4" and were filled with clay for these tests. The tins were weighed empty. Again when filled and at frrquent intervals after being placed in water.

The following table shows the results:

Table of Absorption Test Results.

No of test.	1	2	3	4	Average.
Weight of clay	15.7g	157	154	159	
after 5 minutes	71.3"	72.0	86.2	69.2	70.2
" 10 "	68.8"	69.5	669	690	68.5
" 20 "	66.2"	68.8	65.6	68.3	67.2
" 40 "	65.0"	67.5	63.0	66.7	65.5
" 70 "	63.1"	66.2	61.7	63.5	63.6
" 2 1/2 hrs.	61.8"	65.6	61.1	62.3	62.7
" 3 1/2 "	61.1"	65.0	61.4	61.0	61.9
" 6 1/2 "	61.1"	65.0	59.8	60.4	61.6
" 22 "	61.8"	65.6	61.1	61.7	62.5

It will be noticed that more water is absorbed in five minutes than can be contained. This must be due to the fine particles filling the interstices between the larger ones and crowding out water. After several hours the percent of water absorbed seems to reach a minimum and then increases again.

Asthe clay absorbs water it is drawn down in the tin by surface tension. To determine the percentage of this shrinkage, carefull measurements were made of the height of the tin above the clay and the volumne computed and compared with the original volumne of the clay.

The results were as follows:

Table of Shrinkage upon Wetting.

Number	Volumne of tin	Volumne of clay	Percent of Shrinkage.
1	12.65	10.63	16.03
2	"	10.29	16.72
3	"	10.31	18.56
4	"	10.63	16.03
		Average	17.32

Shrinkage on Drying.

This was determined by measuring when dry the bricks formed from the above and comparing the volumne with the volumne of the tins.

Table of Srhinkage on Drying.

Number	Volumne of tin	Volumne of clay	Percent of Shrinkage.
1	12.65	5.55	56.08
2	"	5.55	56.08
3	"	5.88	53.48
4	"	6.46	48.90
		Average	53.61

Test of Fusing Point.

As the degree of heat required to fuse the clay was known to be high the only method of determination was by means of Seger Cones. These are numbered from one to thirty-six inclusive, the higher the number the higher the corresponding fusing point. The fusing point of number thirty-six is supposed to be about 1850 degrees.

A cylinder of about two and one half inches and one inch in diameter, having a concentric cylinderical opening of about four inches in diameter, was used for a furnace. The bottom of this was covered with a perforated iron plate through which a blast could be forced.

Inside on the bottom plate, a solid cylinder bout one and one-half inch in diameter and two and one-half inches long was placed, and and on this a covered cylinderical crucible of the same dimensions, containing a cone of the clay to be tested together with two or three segar cones. A coke fire was then built around this and the blast admitted.

In a comparison with numbers 33, 34 and 35, these numbers were more or less fused while the unknown clay was not. In the attempts of making a comparison with number 36, the results were either no fusion or a fusion of the containing crucible so that no comparison with number 36 was made. As number 35 has a fusing point of about 1830 degrees this still shows the clay to be very refractory.

Chemical Determinations.

The chemical analysis was carried on as follows: one gram of the clay finely powered was placed in aplatinum crucible and heated to drive off all volatile matter. This had to be started at a moderate temperature as so much water was present that the escaping steam would throw the substance out of the crucible. After the greater part of the water was driven off the process wascompleted by heating to constant weight over a blast lamp.

After determining the percentage of water the charge was fused with sodium carbonate and treated with hydrochloric acid. As this formed gelatinous silicic acid, it was necessary to evaporate

to dryness to form silica. This was then treated with acid and water filtered and the precipitate weighted. The precipitate was then treated with hydrochloric acid and heated to drive off the silicon fluoride and again weighed. The residue this obtained was probably aluminia mechanically held by the silica and therefore was afterwards added to the alumina as determined.

The alumina was precipitated as hydroxide heated to the oxide and weighed. From the filtrate, the calcium was precipitated as calcium oxalate, filtered redissolved in hydrochloric acid and titrated with a standard solution of potassium permanganate.

In another charge the calcium oxide was determined by boiling with hydrochloric acid after ignition but before fusion, filtering, washing and again weighing charge. The change in weight represents all substance present except the compound of silica and alumina. The result thus obtained was about .05 of one per cent above the other determinations of calcium oxide. This might be due to inaccuracies or to the iron and magnesium, both of which are present in very small quantities.

The Results obtained were as follows.

Number	SiO_2	Al_2O_3	Loss on ignition	CaO
1	41.16	35.00	24.38	0.40
2	41.34	33.60	24.63	0.37
3	43.02	35.82	22.07	0.45
Average	41.84	34.81	23.69	0.41

As the loss on ignition varied in these charges, it being due to air dry a ground sample was next tried.

Ten grams were weighed out in a watch glass and placed in a protected place to dry on February first. On April third it weighed

9.555 grams but as the weather was damp it was returned and weighed again on April seventh, which was a clear dry day. It then weighed 9.150 grams, showing readily the influence of atmospheric conditions.

Drying at 100 degrees C. was accomplished by heating for four hours in an oven. Watch glasses provided with a clamp were used to precent change of weight while being dried and weighed. The cooling was done in a sulphuric acid dessicator.

Number one and two were from the above air dired sample and three and four from a freshly pulverized sample.

The results were as follows:

No.	Weight.	After drying at 100 C.	After Ignition	Combined. H_2O	Total loss Percentage
1	2.4609	2.2788	2.0200	11.36%	17.92
2	2.1807	2.0175	1.7062	15.00	18.00
3	1.6468	1.4836	1.2594	15.11	23.52
4	1.2841	1.1620	.9817.	15.52	23.54

The column headed combined water gives the percent of the sampled dried at 100 degrees C. which is lost on ignition.

By accident the thermometer with number one was allowed to rise to 130 degrees C. which probably accounts for the low percentage of combined water. Regarding this result as inaccurate the average combined water is 15.21 percent.

The clay was thought to be Halloysite which according to Dana has a firmula 2 $S:O_2$ Al_2O_3 2 H_2O Aqua, and giving percentage as follows: $S:O_2$, 43.5% Al_2O_3 36.9% , H_2O 19.6 %.

This corresponds to 2 SiO_2 Al_2O_3 3 H_2O but from the manner in which the formula is written one molecule of water seems to be uncertain. As even the air dried specimen of this mineral contains

only 18% H_2O, if it is Halloysite, this must have a different formula.

Reducing the average of the chemical determinations to the basis of combined water, we have SiO_2, 46.04%, Al_2O_3 38.30% H_2O 15.21 %, CaO. 45%.

Assuming the calcium oxide to be an accidental impurity, these percentages correspond closely to the formula:

8 SiO_2, 4 Al_2O_3, 9 H_2O, which gives the following percentage, SiO_2 45.70, Al_2O_3 38.88, H_2O 15.42. This formula is too complicated to be certain.

If we assume that a slight mount of carbonaceous matter is present as shown by the black color during ignition the percentage would correspond closely to the formula 2 SiO_2, Al_2O_3, 2 H_2O with the following percentage. SiO_2, 46.44 %, Al_2O_3 39.58, H_2O 13.98.

As thisis much more simple than the previous formula it is probably more correct. The formula is the same as for Kaolinite but the mineral does not have a crystaline structure .