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BENEFICIATION TESTS ON LATAH COUNTY CLAYS

By

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INTRODUCTION

Because of the huge demand for aluminum in the aircraft industry and the fact that our reserves of high-grade domestic bauxite are definitely limited, the search for new raw materials and methods for producing alumina has taken on great significance. Compared with the 1939 production of metallic aluminum, the figures for 1942 were more than three times as large, and it is estimated that the total production for 1943 will be seven times that of 1939. [1]

Although it was originally believed that bauxite was a definite mineral compound, the term has come to include several of the hydrous aluminum oxides. In nature these are always found mixed with varying amounts of iron oxide, silica, and other impurities in small amounts. The impurities are present as mechanical mixtures, however, and not as chemical compounds.

Clay represents an almost inexhaustible source of aluminum, but as yet the cost of recovering the metal is too high to compete with bauxite. The aluminum-bearing mineral in clay is kaolinite, although as with bauxite, this term has come to include several similar minerals not originally identified. Kaolinite is a hydrous aluminum silicate. The theoretical composition of the mineral is alumina 39.5%, silica 46.5%, and water 14.0%, but very few clays actually come up to this alumina content. Unlike bauxite, in which the silica is present as a mixture, it is quite obvious that the silica content of clay cannot be brought below the theoretical composition by any ore dressing method.

All clays are of secondary origin, being the products of weathering of rocks such as granite, that contain aluminous minerals. They may be roughly divided into two classes, namely (1) residual, and (2) transported. In the first class are included all clays that have been weathered in place and roughly maintain the structure of the original rock. These are usually found to contain quartz, partially decomposed feldspars, mica, and any other weather-resistant minerals that may have been in the original rock. In the second class are placed the clays that have been transported, generally by running water, to some place other than the original location. Since running water tends to classify, according to particle size and specific gravity, any material it is carrying, the fine clay minerals are transported farther than the coarse, undecomposed material. On account of this natural concentration, transported clays are generally less amenable to beneficiation than the residual ones.

It is a well-known fact that the Bayer Process, which is standard for producing alumina from bauxite (low silica and high iron), is not applicable in the treatment of clays (high silica and low iron). A multiplicity of processes have been proposed for producing, from clay, alumina that is suitable for use in

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[1] Wilson, Philip D., Aluminum production: Mining & Metallurgy, Feb. 1943, p. 71.

either the Hall-Heroult or Soderberg electrolytic cell. The most promising of these appear to be either an acid leach or the soda-lime sintering process. A discussion of these is beyond the scope of this paper which covers only the possible beneficiation by ore dressing to produce a high-grade kaolinite.

The samples on which this work was done were submitted by the U. S. Bureau of Mines as cut in their exploration work on the Latah County clays. Samples of both the transported and residual material were received but most of the work was done on the residual clay. The prime object was to produce a concentrate that would be satisfactory feed material to the proposed dilute sulfuric acid leaching. In assaying, except where otherwise indicated, only the percentage of alumina or R_2O_3 that would be available to an acid leach was reported.

The writer wishes to thank Mr. A. W. Fahrenwald for his many helpful suggestions; also Mr. C. F. Zeuch, formerly Analyst with the Idaho Bureau of Mines and Geology, for the assays made on the test products.

TESTING PROCEDURE AND DATA

A.) Tests on Transported Clay -

Of the several samples of drillings from the transported deposits that were submitted, all looked about alike and work was done on only one. This sample was so extremely fine-grained that no appreciable concentration could be made in a separation by particle size. An assay of the head sample showed that the alumina content was only 4 per cent below and the silica 3 per cent above that of pure kaolinite. No great improvement in grade could be expected, so only a screen analysis was made, the results of which are given in the following table.

TABLE I.

Analyses of Various Screen Sizes of Sample

from Hole No. 18 (17.9'-28')

Screen Size	Weight, %	SiO_2 Assay %	Fe_2O_3 Assay %	Al_2O_3 Assay %
+100 mesh	3.4	60.1	4.62	26.7
-100 +400 mesh	15.4	53.3	2.16	32.1
-400 mesh	81.2	48.4	1.73	36.7
Composite	100.0	49.5	1.9	35.5

Note: The above assays represent total amounts obtained by fusion.

Kaolinite that has not been calcined is relatively insoluble in dilute sulfuric acid. A series of ignition tests was made on material that had been thoroughly dried at 110° C. to determine the temperature required for driving off the water of hydration. The sample was calined to constant weights at intervals of approximately 100° C. Some difficulty was encountered in reaching constant weights in the intermediate range, but it was proved that most of the loss occurs between 400 and 500° C. A summary is given in the following table.

TABLE II.

Weight Loss on Ignition at Various Temperatures, Sample from Hole No. 18 (17.9'-28')

Ignition Temperature	Loss in Weight - Cumulative %	
	Sample I	Sample II
300° C.	1.30	1.53
414° C.	3.03	3.3
512° C.	11.1	11.3
609° C.	11.9	12.1
750° C.	12.55	12.7
847° C.	12.7	12.85

B.) Tests on Residual Clay -

Washing Tests

The sample of approximately 150 pounds of residual clay contained a large amount of quartz, partially kaolinized feldspars, and muscovite mica, which made it apparent that a considerable improvement in grade could be expected from a washing operation. A fusion assay on the head sample gave the following analysis: ignition loss= 6.55%, SiO_2 = 68.6%, Fe_2O_3 = 3.03%, Al_2O_3 = 21.2%. Only the "available" contents (to a dilute H_2SO_4 leach) were determined in all assays of the test products. Compared to total amounts, the "availables" on the head sample were: R_2O_3 = 18.5%, SiO_2 = 1.6%, Fe_2O_3 = 2.16%, Al_2O_3 = 14.7%.

In order not to grind the quartz and other sand particles while breaking up the clay lumps, the porcelain mill was equipped with rubber-coated steel rods as grinding media. A batch of 500 grams of clay was mixed with 1500 cc. of water and run in the mill until all clay lumps were broken up. The sample was then transferred to an enameled bucket where a separation at the desired particle size was made by sedimentation. Sodium silicate was added as required to prevent flocculation. Three tests were made with cuts at 200, 400, and 800 mesh, respectively. All settling times were calculated from Stoke's law of settling by viscous resistance. The metallurgical results of these tests are tabulated below.

TABLE III.

Metallurgical Results of Sedimentation Tests

Test No.	Product	Weight %	Assay Avail.R_2O_3 %	Recovery Avail.R_2O_3 %	Assay Avail.Al_2O_3 %	Recovery Avail.Al_2O_3 %
2 Overflow at -200 mesh	Sands (2-1)	44.2	4.5	10.8	2.7	7.8
	Overflow(2-2)	55.8	29.4	89.2	25.2	92.2
	Composite	100.0	18.4	100.0	15.25	100.0
3 Overflow at -400 mesh	Sands (3-1)	49.9	6.0	16.5	3.7	12.0
	Overflow(3-2)	50.1	30.2	83.5	26.9	88.0
	Composite	100.0	18.1	100.0	15.3	100.0
4 Overflow at -800 mesh	Sands (4-1)	58.2	7.8	25.1	5.6	21.4
	Overflow(4-1)	41.8	32.3	74.9	28.5	78.6
	Composite	100.0	18.0	100.0	15.2	100.0

Thickener Tests

Although good metallurgical results can be obtained by a simple washing operation on this clay, some satisfactory method for dewatering the concentrate must be found. Raw clay has to be calcined before the alumina can be leached from it in any acid process, and unless a reasonably dry concentrate can be delivered to the calcining kilns the heat cost will be exorbitant.

To determine the thickening rate of the washed concentrate, pulps of various densities were prepared and placed in 1000 cc. graduate cylinders for observation of the settling rate. Lime was added to flocculate the solids. Without this, no definite water-pulp line could be observed; the colloidal clay particles staying in suspension indefinitely. When lime is added, good flocculation is attained and the water above the pulp is almost clear. However, the pulp reaches the compression stage of settling at a high water to solid ratio and all thickening rates are extremely slow. One test was made to determine the maximum thickening density. After 29 hours settling the water to solid ratio was still 1.95 to 1 by weight.

Once kaolinite has been calcined to drive off the water of hydration, it loses its plasticity and does not regain it on being mixed with water. Since it was found that the thickening of the concentrate of raw clay would be impractical, a series of tests was made to determine the possibility of calcining before any beneficiation was tried. It was thought that if a satisfactory concentrate could be made from the burned material, it would be much easier to handle later on.

Such a concentrate would be ready for the acid leach and could contain an appreciable amount of water that would not be tolerated where the concentrate had to be sent to kilns for calcining.

A sample of the raw clay was calcined at 800° C. and then submitted to the same grinding and washing procedure as had been used on the raw material. It was found that the clay lumps form hard aggregates on burning and that it is difficult to break them up without grinding the quartz and other sandy impurities. For this reason, the grade of the concentrate and also the recovery were much lower than had been previously attained.

The thickening tests showed that, although the calcined material settled several times faster than the raw, it was still too slow for commercial operation. Again, it was found that the compression stage of thickening was reached at a high water to solid ratio and, consequently, the settling rate fell off rapidly as the pulp density increased. After settling overnight, the ratio of water to solids, by weight, was 1.48 to 1 in the thickened pulp.

These tests proved that this method of treatment has too much against it to be commercially successful. It was also found that some of the R_2O_3 is rendered insoluble by ignition. Instead of showing a higher percentage of available R_2O_3, as it should on account of the ignition loss, the assays were slightly lower than for the raw clay. As evidence of the loss of plasticity on burning, calcined clay, after being mixed with water, dries to a fine powder instead of the solid cake formed on drying raw clay.

Data on the thickening rates for both the raw and calcined clay are given in the following tables.

TABLE IV.

Summary of Thickening Tests on Calcined Clay Concentrates

Test No.	Weight of Solids(gm)	Volume of pulp at start (cc.)	Ratio H$_2$O:Solids by weight at start	Ratio H$_2$O:Solids by weight at finish	Settling rate, Ft. per hour
19	69	1000	14.0:1	9.0:1	2.03
19	69	850	11.9:1	7.6:1	1.74
19	69	700	9.7:1	6.0:1	1.51
20	120	1000	7.9:1	4.7:1	1.36
22	144	1000	6.5:1	4.9:1	0.84
22	144	850	5.5:1	4.2:1	0.63
22	144	700	4.5:1	3.6:1	0.437
23	179	1000	5.2:1	3.8:1	0.855
23	179	850	4.3:1	3.3:1	0.644
23	179	700	3.5:1	2.8:1	0.453

TABLE V.

Thickening of Raw Clay Concentrate - Test 18-A

Time of Reading, Hours	Reading at Water-Pulp Level (cc)	Ratio H$_2$O:Solids by weight	Settling rate from Previous density Ft./hour
0	1000	5.75:1	----
$\frac{1}{4}$	975	5.61:1	0.116
$\frac{1}{2}$	920	5.27:1	0.256
$\frac{3}{4}$	860	4.90:1	0.279
1	805	4.56:1	0.256
1$\frac{3}{4}$	630	3.50:1	0.271
3	550	3.00:1	0.074
4	535	2.90:1	0.017
6	510	2.75:1	0.015
7	500	2.68:1	0.012
10	475	2.53:1	0.010
22	410	2.13:1	0.006
27	385	1.97:1	0.006
29	380	1.95:1	0.003

Note: Weight of dry solids = 162 gm.
S.G. of solids = 2.5
1 foot on graduate = 860 cc.

Air Classification

The possibility of separating the fine kaolinite from the coarse-grained impurities by air classification was considered as another scheme of beneficiation. In the original tests the material to be classified was dropped through a horizontal stream of air from a blower. The metallurgical results of these tests did not come up to expectations and no data on them will be included.

A more definite separation, according to particle size, can be made by sorting in a vertical column. Any particle whose mass multiplied by the acceleration of gravity is greater than the resistance force of the rising air will fall to the bottom of the column, and if the force of the air against a particle

is greater than its weight, it will necessarily rise. This gives a definite cut-off instead of the gradation from coarse to fine obtained by blowing in a horizontal plane.

A classifier was constructed by attaching a vertical pipe, 2-3/4 inches in diameter by 36 inches in length to a centrifugal blower. A cloth was stretched over the blower outlet as a constriction plate to restrict the air velocity and to give a more uniform flow across the section of the sorting column. By dropping the feed into the top of the pipe, the sand portion was collected in the bottom while the fine was blown out through the top. To collect the concentrate, the classifying column was encased in a box of suitable size with a small opening in the top, directly over the column, to hold a funnel for charging the feed. Dusting losses amounted to approximately 4 per cent of the feed, which was assumed to be from the fine portion in the calculations.

Although air classification was tried on both raw and calcined clay, the best results were obtained on the raw material. After being thoroughly dried at 110° C., the lumps of raw clay were quite readily pulverized. The metallurgical results of the several tests are tabulated below and reference to Table III will show how these compare with water classification.

TABLE VI.

Air Classification of Raw Clay

Test No.	% of Weight in Concentrate	Assay - Avail.R_2O_3 in Concentrate %	Recovery - Avail. R_2O_3 in Concentrate %	Calculated Head Available R_2O_3 %
11	43.2	26.2	59.5	19.0
12	43.3	25.2	60.2	18.2
13	48.2	25.6	67.3	18.4
14	40.0	26.2	57.0	18.2
15	45.8	24.4	65.2	17.1
16	52.5	25.8	71.8	18.8
17	55.2	24.6	73.6	18.5

SUMMARY

The results of this testing program may be summarized as follows:

1) A concentrate of satisfactory grade for the proposed dilute sulfuric acid leaching process can be made by direct washing of the raw clay.

2) The washed concentrate cannot be dewatered by ordinary thickening and filtering procedures.

3) With some sacrifice in grade and recovery, definite beneficiation can be obtained by classifying the dried raw clay in a rising air current. When thoroughly dry, the lumps are easily broken up. The large amount of fine mica causes some difficulty in air classification.

4) Air classification of the calcined clay is impractical because it is too difficult to break up the lumps after burning.

5) Washing the calcined clay gives a lower grade concentrate and a lower recovery than washing the raw clay. Although the washed product is more readily thickened than that of the plastic clay, it is still too slow to be commercially feasible.

6) Flotation tests on both raw and calcined clays gave poor metallurgical results.