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ALUMINUM INVESTIGATION PROGRESS REPORT

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collaborating

Object.— The purpose of this investigation is to determine the possibility of commercially practicable recovery of aluminum from high grade Kansas clays.

Preliminary investigation.— A review of the history of the aluminum industry led to the conclusion that a process, sometimes called the "Deville-Pechiney process", had promising possibilities as applied to production of alumina from alumina-rich clay. The method, designed to treat bauxite having a moderate silica content, consists of heating the ore with sodium carbonate to form sodium aluminate, which is recovered by leaching with water. Most of the contents of ore, except the alumina, are supposed to remain in the leached residues as insoluble compounds. Mention is made of the possibility of employing calcium carbonate to form an insoluble calcium silicate that would also remain in the residues. The process was considered best suited for treatment of low-silica bauxites. Published descriptions of the process led to the idea that addition of a sufficient quantity of calcium carbonate to combine chemically with the silica of the clay, so as to form insoluble calcium silicate, might free the alumina of certain Kansas clays. Also considered was the observation in testing work on some Kansas clays that at certain temperatures components of the kaolin present are more or less dissociated into silica and alumina. This critical temperature may be at approximately 600° C, but in any event all of our tests were made on clay previously heated to that temperature and subsequently kept in dessicators until required for further study.

1/ The Aluminum Industry, Edwards, Frary and Jeffries, vol. 1, p. 132.

We believe the results as shown in the tests given later in this report are, unless otherwise stated, technically correct, but it should be appreciated that on account of the limited capacity of our equipment, especially in regard to sintering crucibles and proper grinding equipment, the tests were run for the most part on small samples and the resulting sinter had to be ground down by hand in a porcelain mortar. Calcined alumina is highly hygroscopic, which accounted for unreliable analyses in our first tests. This was controlled later on, however.

Results.— Eight tests now have been run. These were made on one-gram samples of clay, which, with the fluxes, were sintered in platinum crucibles protected in covered clay crucibles placed in a gas-fired pot furnace. Thus far, the mixtures for sintering have had approximately the following ratios: CaO: SiO_2 = 2:1; MgO:Al_2O_3 = 1:1. The temperatures of firing the samples have ranged from 1375° C to 1475° C. The resulting cake, unless otherwise stated, has been well sintered in every case, coherent and somewhat porous. One 45-gram sample of clay was run. Our procedure was to leach the cake quickly for 5 minutes in 250 cc of water at 80° C, and then to treat the sample with four hot water washes. The residue was boiled in 50 cc normal NaOH for 5 minutes and washed four times with hot water. An analysis of the calcined clay used in all tests is as follows.

Analysis of calcined clay from Dakota formation, north central Kansas

SiO_2		63.60 per cent
Al_2O_3		31.60
Fe_2O_3		2.38
CaO		.50
MgO		.50
	Total	98.58

The first four tests showed a considerable extraction of alumina in the form of sodium aluminate, both by water and caustic leaching. By means of water leaching alone, 25 to 60 per cent of the Al_2O_3 of the clay sample was obtained. Our laboratory technique on the first runs was not well developed and inasmuch as the results on recovery of alumina did not check with later work, they are not reported in detail. There was no iron in the leach solution and except for some that appeared in certain alkali leaches we have encountered no problems in dealing with iron. There was considerable silica in some of the first four leaches, the ratio of silica to alumina recovered being given in the following table.

Analyses of solutions from sintered clay charge on first four tests

	SiO_2	Al_2O_3	Temperature of sintering
No. 1 water leach	.0016 gm	.076 gm	1380° C
alkaline leach	not run		
No. 2 water leach	.0014	.1188	1400°
alkaline leach	.0023	.1600	
No. 3 water leach	.0016	.0998	1450°
alkaline leach	.0147	.1137	
No. 4 water leach	.0110	.1631	1400°
alkaline leach	.0170	.1547	

From here on the analytical work was done with great care. Test 5 is approximately representative of results which we believe can be expected except that the silica in the caustic leach seems low for some reason. The solutions were run twice with check results but the residue was not checked.

Analysis of solutions from sintered clay charge; fifth test

No. 5	SiO_2	R_2O_3	Al_2O_3	Fe_2O_3	CaO	Ig loss
Water leach	.0005 gm.	.214 gm.	.214 gm.	Nil	.041 gm.	
NaOH leach	.0004	.056	.056	Nil	.042	
Residue 2.0 grams	29.40%	5.2%	4.2%	0.84%		None

Extraction of Alumina

Water leach 67.7 percent Al_2O_3

NaOH leach 17.7 percent Al_2O_3

Total 85.4

Molecular ratios

$CaO:SiO_2$ = 2:1; $Na_2O:Al_2O_3$ = 0.87:1

Temperature - 1480° C 45 minutes

Metallurgical Balance

Grams of Al_2O_3 in clay = 0.3160

Grams recovered water leach = .214

Grams recovered NaOH leach = .056

Grams in residue = .084

Total .354

It will be noted there is an excess of Al_2O_3 in the products of .038 grams over that in the original clay. The 12 percent excess we believe to be due to an error in the residue analysis for alumina.

Results of a sixth test are given in the following tabulation. This was also based on a 1-gram clay sample.

Analysis of solutions from sintered clay charge; sixth test

No. 6	SiO_2	RO_3	Al_2O_3	Fe_2O_3	CaO	Ig. loss
Water leach	.0037 gm.	.227 gm.	.227 gm.	Nil	.037 gm.	
NaOH leach	.0365	.0085	.0073	.0012	.0355	
Residue 1.88 gms.	27.62%	4.00%	3.14%	0.88%	49.4%	12.16%

Extraction of Alumina

Water leach	71.8 percent Al_2O_3
NaOH leach	2.3 percent Al_2O_3
Total	74.1 percent Al_2O_3

Metallurgical Balance

Grams Al_2O_3 in charge	= .3160
Grams Al_2O_3 in water leach	= .2270
Grams Al_2O_3 in NaOH	= .0073
Grams Al_2O_3 in residue	= .059
Total	.2933

$\frac{.3160 - .2933 \times 100}{.3160}$ = 7.1 percent Al_2O_3 unaccounted

Molecular ratios $CaO:SiO_2$ = 2:1; $Na_2O:Al_2O_3$ = 0.87:1

Temperature of sintering was up to 1475° C for 45 minutes. The charge sintered into a very hard compact mass. This may have something to do with the high silica.

A seventh test, following the same procedure as in the previous ones, gave results that corroborate the earlier findings. An analysis follows.

Analysis of solutions from sintered clay charge; seventh test

Test 7	SiO_2	R_2O_3	Al_2O_3	Fe_2O_3	CaO	Ig. loss
Water leach	.0078 gm	.217 gm	.217 gm	Nil	.033 gm	
NaOH leach	.0305	.006	.0047	.0013	.065	
Residue 1.98 grams	27.30%	5.98%	3.36%	0.60%	49.60%	12.16%

Extraction of Alumina

Water leach	71.8 percent Al_2O_3
NaOH leach	2.3 percent Al_2O_3
Total	74.1 percent Al_2O_3

Metallurgical Balance

Al_2O_3 in charge	0.316 grams
Grams Al_2O_3 in water leach	0.217
Grams Al_2O_3 in NaOH leach	0.0047
Grams Al_2O_3 in residue	0.0068
Total	0.2285

$\frac{.3160 - .2285 \times 100}{.3160}$ = 8.7 percent Al_2O_3 unaccounted

Molecular ratios in charge; $CaO:SiO_2$ = 2:1; $Na_2O:Al_2O_3$ = 1:1

The charge sintered the same as number 6, possibly accounting for high silica. Temperature to 1475° C, 45 minutes.

Tests nos. 8 and 9 were planned primarily to determine the quantity of silica in the solutions from leaching. Results are shown in the following statements.

Partial analysis of solutions from sintered clay charge; eighth test

Amount of silica in the leaches is as follows

Water leach	SiO_2 = 0.0001 gm
NaOH leach	SiO_2 = 0.0195 gm.

This represents a harmless amount of silica in the water leach. This analysis was on a 1-gram clay sample sintered at 1450° C.

Test no. 9 was made in order to determine accurately amounts of iron and silica that may be present in the leaches. Amount of clay used, 46.6 grams; temperature of firing, 1450° C. for 45 minutes. Ratios $CaO:SiO_2$ = 2:1; $Na_2O:Al_2O_3$ = 1:1. The extraction of alumina from the charge was incomplete for the reason that fusion occurred and in the time available the coarse slag particles could not be broken down in a muller by hand to give the proper size for leaching.

Partial analysis of solutions from sintered charge of 46.6 grams of clay,

ninth test

	SiO_2	Al_2O_3	Fe_2O_3	
Water leach	0.0304 gm	6.50 gm	Not run yet	None or a trace only
NaOH leach	0.099	1.84	Not run yet	None or a trace only
Residue		Not run		

Extraction of Alumina

Water leach	44.3 percent Al_2O_3
NaOH leach	10.5 percent Al_2O_3
Total	54.8 percent Al_2O_3

The amount of SiO_2 present is far less than that found in solutions from which alumina is precipitated in the commercial Bayer process.

On the basis of these tests it is believed that as our technique improves an extraction of 70 percent of the alumina present can be obtained regularly by water leaching, yielding a product sufficiently pure to meet the requirements of alumina to be used for aluminum production.

The question of utilizing the more impure alumina produced by the caustic leach, and the recovery of soda from such leach solutions must be postponed for further investigation. The samples of alumina from leach 9 are available for analyses. They are pale green to white in color.

Estimated costs.— Aside from relative simplicity and low plant cost, several factors indicate that this process should have a low operating cost in Kansas. Among especially favorable conditions are the following: There is an abundance of natural gas that is available at low cost from two pipelines traversing the area of clay deposits. Readily developed in exhaustible supplies of chalky limestone lie adjacent to the clay deposits. Analyses of this limestone show 97 percent calcium carbonate; it requires little effort to crush. Immense deposits of clay of very uniform chemical and physical characters have been explored in north central Kansas;

they carry some 51 percent Al_2O_3. On the basis of laboratory tests so far made, only the most tentative figures as to the cost of alumina produced by the process are warranted. Estimates, which are explained in a following break-down, indicate that a plant producing twenty tons of alumina per day could produce this amount at a cost of about three cents per pound f.o.b. Kansas. Two pounds of alumina are required to produce one pound of aluminum, and of course electric power is required for this operation. Presumably the alumina manufactured from treatment of the clay would be shipped to a point of economical final metallurgical treatment.

Explanation of Tentative Cost Estimates for Production of Aluminum in a 20-ton per day Plant

(Items carefully considered but not exhaustively checked)

Required for 1 ton alumina (Al_2O_3):

Clay 4.7 tons (assum. 75% ext. and 10% H_2O) @ $0.15 =		0.70
Limestone 10.3 tons (4.7 x 2.19 = 10.3) @ $1.50 =		15.46
Soda ash 1.6 tons (4.7 x 0.35 = 1.6) @ 15.00 =		24.00
Gas: Calcining clay at 600° C $\frac{1 \times 0.2 \times 600 \times 1.8 \times 2000 \times 4.7}{1100 \times .57}$ = 3.2 M cu. ft.		
Gas: Kiln, firing mix 1450° C $\frac{1 \times 0.2 \times 1450 \times 1.8 \times 2000 \times 11.2}{1100 \times .57}$ = 18.6		
Gas: Calcining 1 ton Al_2O_3 at 1000° C $\frac{1 \times 0.2 \times 1000 \times 1.8 \times 2000}{1100 \times .57}$ = 1.3		
Gas: Heating leach water to 80° C $\frac{1 \times 1 \times 80 \times 1.8 \times 2000 \times 33.5}{1100 \times .30}$ = 17.4		
Total gas	40.5 M cu. ft. @	
Water		4.05
Water. Assume $5.00 per day		0.25
Power: Crush clay 4.7 tons @ $0.10 = $0.47		
Power: For kiln $\frac{224 \text{ tons} \times \$0.10}{20}$ = 1.10		
Power: For calciner $\frac{332 \text{ tons} \times \$0.10}{20}$ = 1.66		
	3.23	3.23
Labor. 3 shifts of 15 men $\frac{45 \times \$4.00}{20}$ =		9.00
Overhead. Assume plant cost $150,000.00. Interest 6%, Depreciation 10% $\frac{\$150,000.00 \times .16}{20 \times 365}$ =		3.29
Total cost per ton Al_2O_3		59.96
Total cost per pound		0.0299
Total cost Al_2O_3 per one pound aluminum		0.0598

This estimate is based on results expected by water leaching only; it makes no consideration of the higher extraction and lowered costs to be expected from the recovery of sodium from the leaching solutions, either as carbonate or caustic, and their use in the process.

Power requirement 1 ton aluminum - $50.00 Per pound - $0.025
Cheapest U. S. Power, Bonneville Dam 25000 Kw. hr. @ .002 - $50.00

Summary.— Preliminary laboratory studies have demonstrated the practicability of separating Al_2O_3 from alumina-rich clay. These investigations indicate the possibility of producing aluminum on a commercial basis at costs as low as those utilizing bauxite as a source and possibly at lower costs. If this is true, tremendous quantities of source materials in Kansas and probably other parts of the United States await development. Enlargement of the investigation so as to permit tests on considerably increased quantities of clay is justified and recommended strongly. If this work proves the results already obtained, as we are sure will be the case, pilot plant tests should be provided. Because the manufacturing operations are comparatively simple, it should be possible to arrange for such pilot plant studies without large outlay of capital or delay in time.

— E. D. Kinney