

**A PROCESS FOR EXTRACTING ALUMINA
FROM KANSAS CLAY**

By

E. D. KINNEY

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¹ Metallurgist for Kansas Geological Survey and Department of Mining and Metallurgical Engineering, University of Kansas.

FOREWORD

For the past six years the State Geological Survey of Kansas has been carrying on detailed investigations on the light-firing and refractory clay deposits of the Dakota formation in central and north-central Kansas. These studies were undertaken primarily to determine the quality and reserves of these clay deposits and their possible uses in pottery, light-colored face bricks, refractory shapes including firebrick, and other ceramic products. In the course of this work, the alumina content of many of these clays was determined. The generally high percentage of alumina pointed the way to the investigation described in this report.

When the United States entered the present war the demands for aluminum were increased many times. The ore of aluminum in most common use is bauxite. For several years it has been known that the reserves of this ore within the United States are not large. Large tonnages of ore have been imported from South America and elsewhere. In order to place this country in a safe position as to our future aluminum supplies, several institutions have undertaken researches to develop a practical commercial process of extracting alumina from material other than bauxite. Such an investigation has been carried on for the past year and a half by the State Geological Survey of Kansas. Our research has been devoted to the utilization of Kansas raw materials and has been twofold: (1) The preliminary enrichment of raw clay, and (2) the extraction of alumina, or aluminum oxide, from raw clay or enriched clay, as described in this report. This process can operate on raw materials which occur wholly within the state,—clay, limestone or chalk, salt, and fuel. The proximity of all these materials to central Kansas gives the state a natural economic advantage if this process were to be put into commercial operation.

It should be pointed out that the clay, chalk, and salt reserves of the state are not being seriously depleted. A near-by and abundant supply of gas is an essential requirement to the commercial success of this process. At the present time Kansas possesses adequate reserves of this fuel to last for many years, provided the gas is not exported in excessive quantities from the state for the industrial development of other areas.

JOHN C. FRYE.

A PROCESS FOR EXTRACTING ALUMINA FROM KANSAS CLAY

By E. D. KINNEY

ABSTRACT

A process for the extraction of alumina from clay, utilizing Kansas raw materials, is described. This process is essentially the old soda-sinter process with a new method, devised in the laboratories of the State Geological Survey of Kansas, for controlling the impurities in the solution from which the aluminum hydroxide is precipitated. This method consists of the fractional precipitation of impurities by carbon dioxide gas and their removal by filtering. It is believed that this method may make it possible for Kansas raw materials to compete commercially with other sources of alumina. For a better understanding of the subject, a review of the metallurgy of aluminum is given.

INTRODUCTION

RELATION OF KANSAS TO ALUMINUM PRODUCTION

During the past five years aluminum production in this country has increased tenfold, due largely to an increasing demand from the airplane industry. The annual production is now about 900,000 tons. Domestic resources of high-grade bauxite, the commonly used ore of aluminum, are becoming depleted, and we must look to other domestic sources for aluminum. Clay, because of its wide distribution in large deposits, is the potential aluminum ore of the future, at least in this country. Although the aluminum content of clay may be only half that of bauxite, its use as an ore of aluminum would probably result in only a small increase over the present price in the cost of the metal. The percentage of aluminum in clay is far greater than the metal content of most non-ferrous ores.

Kansas has immense deposits of easily mined clay with an almost inexhaustible supply containing at least 25 percent aluminum oxide. Some other states have richer clays than are now known to exist in Kansas, but, in general, the deposits are of lesser extent. Because of the abundance of cheap gas, limestone, and water—all needed in processing—together with a usable method of extraction that has been developed in the laboratories of the State Geological Survey of Kansas, it is believed that high-grade alumina can be produced in Kansas at a cost which will compare favorably

with production costs elsewhere. This statement is based on the assumption that soda ash, formerly made from Kansas salt and limestone, would again be manufactured and made available for such a process at a reasonable cost.

The alumina process referred to and described in this report is essentially the old soda-sintering process modified to treat clay, with a new purification step, also described in this report, which is necessary to produce a high-grade product. On the basis of large-scale laboratory tests, and not considering possible treatment of the residue, the recovery of high-grade alumina is about 75 percent of the alumina content of the raw clay. Two-thirds of the soda ash employed can be saved for recycling. The process is relatively simple and no unusual difficulty should be experienced in adapting it to large-scale commercial operation.

While the production of aluminum oxide, alumina, in Kansas thus appears entirely feasible, the reduction of alumina to metallic aluminum presents a difficult problem due to lack of cheap and ample electrical power. To produce one pound of aluminum from alumina requires 12.5 kilowatt-hours,—far more power than is needed for the production or refining of any other common metal. For economy, a reduction plant must have a large and constant supply of power. Aluminum plants now in operation in this country are rated at 50,000 to 250,000 horsepower and have an annual output of 15,000 to 75,000 tons of aluminum. The actual cost of this power is not known to me, but it is probably not more than 3 mills per kilowatt-hour (Walter May, 1940). The cheapest wholesale power in the United States is said to be at Bonneville Dam, Washington, where, taken from the transmission lines 8,760 hours a year at 100 percent load factor, the cost is 2 mills per kilowatt-hour (Walter May, 1940).

At the present time, aluminum is being produced from alumina shipped to the following localities where power is cheap: New York, Alabama, Tennessee, North Carolina, Arkansas, Oregon, Washington, and southern California. A government-owned aluminum plant at Malvern, Ark., is the nearest one to Kansas. There is probably little difference in cost between hydroelectric and steam-generated power provided the steam is made from low-cost fuel and both plants are built with private capital. The Bonneville and some other plants now operating were built with Federal funds furnished at low interest rates and thus have the advantage

of low overhead not found elsewhere. Considering our coal deposits and especially the gas reserves of the Hugoton field, it would appear that Kansas is not without resources for producing more and cheaper power than is now produced in the state. Until cheap power is available, it will be necessary to ship any alumina produced in Kansas to other states for reduction.

OBJECT OF THE INVESTIGATION

This investigation was undertaken as a research project of the State Geological Survey to determine if the extensive deposits of clay that occur within the state could be used as a commercial source of aluminum. Such an investigation was deemed especially important at this time due to the urgent need for aluminum in the war effort.

POSSIBLE ALUMINUM ORES IN KANSAS

Up to the present time practically all aluminum has been obtained from the ore bauxite, a hydrous aluminum oxide ($\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$). The best bauxite contains 50 to 60 percent alumina (Al_2O_3), but deposits of this grade, which occur in the south-central and southeastern parts of the United States, are becoming seriously depleted. We are importing about 60 percent of our needed bauxite from British and Netherlands Guiana at a freight rate said to be \$6.00 per ton. Other potentially important sources of aluminum are low-grade bauxite, alunite ($\text{K}_2\text{Al}_6(\text{OH})_{12}(\text{SO}_4)_4$), feldspars (potassium, sodium, or calcium aluminum silicates), cryolite (Na_3AlF_6), and clay.

Clay is probably destined to become the most important aluminum ore of the future in this country because of its great abundance. The principal constituent of the usable clays is the mineral kaolinite ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) which contains 39.5 percent alumina. Clay occurs in many parts of the United States, some of the beds being very large and of high alumina content. In Kansas there are very extensive deposits of variable composition, and an immense tonnage containing 25 percent alumina is available. The clay occurs in low dipping beds 8 to 10 feet thick with relatively little overburden. The low cost of mining, together with near-by abundant supplies of natural gas, limestone, salt, and water, which are needed in processing, offers unusual opportunities for the production of alumina in the state.

ACKNOWLEDGMENTS

Special thanks are due to several staff members of the State Geological Survey of Kansas who gave valued aid in connection with this study. Norman Plummer, an authority on Kansas clays, furnished all of the samples used in the laboratory and gave much information on the clay deposits of the state. Raymond F. Thompson and Russell Runnels made the chemical analyses and assisted in the tests. Mr. Oliver C. Ralston of the U.S. Bureau of Mines furnished valuable information concerning the effect of concentration of sodium aluminate solutions on the solvency of impurities.

OUTLINE OF THE METALLURGY OF ALUMINUM

There are two separate operations in any process for the production of aluminum: (1) the extraction of alumina from the ore by leaching, and (2) the reduction of alumina to aluminum by electrolysis. The various processes differ only in the first operation. In the second step, the extraction of the metal from alumina, all employ the Hall-Herault process. This process consists of the electrolysis of alumina in a fused bath of cryolite.

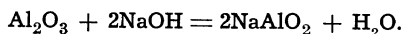
Processes for extracting the alumina from the ore are classified as acid or alkaline depending on the reagent used in leaching. Alkaline processes are the more important. When sulphuric acid is used, iron from both ore and equipment contaminates the solution and is not easily separated from the alumina. Silica is not attacked. Conversely, alkaline processes bring silica into the solutions, which is also difficult to separate from the alumina, but the alkaline reagents do not dissolve iron. Ore to be treated by alkaline processes should be low in silica, and that to be treated by acid processes should be low in iron.

There have been two successful aluminum processes in the history of the industry: the soda-sintering process, used years ago in France, and the modern Bayer process which has been recognized as the standard since about 1890. Both are alkaline processes designed to treat low silica bauxites and not clay.

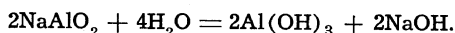
THE BAYER PROCESS

Typically the bauxite used in the Bayer process contains 56 to 59 percent alumina (Al_2O_3), 20 to 28 percent iron oxide (Fe_2O_3),

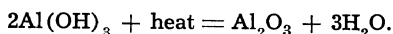
3 to 8 percent silica (SiO_2), 0.5 to 3 percent titanium dioxide (TiO_2), and 10 to 15 percent water. The ore is treated with a solution of 41 percent sodium hydroxide (NaOH) (specific gravity 1.45) for 2 to 8 hours in an autoclave under 60 pounds of pressure and at a temperature of 155°C . The alumina dissolves as follows:



The leach is allowed to stand for several hours to settle the residual ore which contains undissolved iron and titanium oxide. Some silica is dissolved, in part forming sodium aluminum silicate and the balance being soluble sodium silicate. The sodium aluminate solution is filtered and the aluminum is precipitated as hydroxide by diluting the solution to a specific gravity of 1.23. The solution is then seeded with fresh hydroxide and allowed to stand. The reaction is as follows:



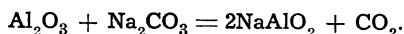
The hydroxide is calcined at $1,000^\circ\text{C}$. to convert it into oxide:



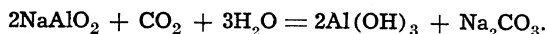
The alumina is converted to aluminum by the Hall-Herault process in an electric furnace. The principle involved is the electrolytic dissociation of alumina dissolved in cryolite and the fluorides of one or more metals more electropositive than aluminum, such as sodium, potassium, or calcium. About 2 pounds of alumina are required to produce 1 pound of aluminum. Also, 0.8 pound of carbon electrode, 0.1 pound of cryolite, and 12.5 to 13 kilowatt-hours of power are consumed in the production of 1 pound of metal. The best metal produced contains 99.7 percent aluminum.

THE SODA-SINTERING PROCESS

In the soda-sintering process a calculated charge of bauxite ore and soda ash is heated to sintering temperature and the soluble sodium aluminate which is formed is removed by water leaching. The sintering reaction is as follows:



Aluminum hydroxide may be recovered by seeding or by the use of carbon dioxide gas. The reaction is as follows:



The remaining treatment is identical with that of the Bayer process.

A commercially successful process for extracting alumina from bauxite or clay must show a good extraction of high-quality alumina and must include the recovery of much of the soda used. Alumina extraction in the Bayer process, excluding possible treatment of residues, is probably 75 to 85 percent.

At present, 80 percent of our aluminum is required for airplane sheet. The alumina for this high-grade metal must conform to the specifications listed in table 1.

TABLE 1.—*Specifications for high-grade alumina*

Impurity	Permissible amount (percent)	Remarks
Silica (SiO_2)	0.02	tolerance to 0.06
Iron oxide (Fe_2O_3)	0.02	tolerance to 0.04
Calcium oxide (CaO)	0.04	
Titanium dioxide (TiO_2)	0.004	
Magnesium oxide (MgO)	0.02	
Potassium oxide (K_2O)	0.05	
Sodium oxide (Na_2O)	0.60	

Silicon, iron, and titanium must be low in the alumina used in the electrolytic process as these elements are deposited with the aluminum. The alkalis and alkaline earth metals entering the cell call for equivalent amounts of aluminum fluoride to make the corresponding cryolite. The alkalis and alkaline earth metals do not deposit, but accumulate as the cell operates and cause trouble by changing the normal percentage composition of the electrolyte. To produce alumina low in silica is a major problem. One of the aluminum alloys, aluminum silicon, can be made from high silica alumina, but little of this metal is used.

NEW PROCESSES

The need for a successful process for the extraction of alumina from clay has long been recognized. Recently a number of new methods have been developed. Some of these have passed the pilot plant stage and are about to begin operation on a commercial scale. Brief descriptions of the more important processes follow.

Soda-lime-sinter process.—The soda-lime-sinter process is a modification of the soda-sinter process designed to treat clay. This method is the basis of the new process developed in the laboratories of the State Geological Survey of Kansas and it will be described

later in this report. Other modifications of the process are now being used to treat high silica bauxites and Bayer process residues, and will soon be used on Wyoming feldspar.

Lime sinter-soda leach process.—The lime sinter-soda leach process is an alkaline process in which clay is first sintered with lime and subsequently leached with caustic alkali solution. A plant which will utilize this process has been approved by the War Production Board for erection in South Carolina.

Kalunite process.—The Kalunite process is one of several designed to extract both aluminum and potassium from the mineral alunite. In Utah, where a plant is in operation, the alunite is dehydrated at 550°C . This treatment makes it soluble in sulphuric acid. An excess of calcine is leached with potassium sulphate and 10 percent sulphuric acid, but in order to keep impurities low the solution is not allowed to become acid. The pregnant solution is filtered and normal potassium alum formed by crystallization. The crystals are dissolved with a dilute potassium sulphate solution and treated in an autoclave at 200°C . The potassium alum is broken down into a basic alum plus sulphuric acid and potassium sulphate. The sulphuric acid and potassium sulphate are recycled to leach fresh ore, while the basic alum is calcined into alumina and potassium sulphate. The calcine is leached with hot water to remove potassium sulphate, leaving the alumina behind. The potassium sulphate is recovered by crystallization. This process can also be used for treating clays.

Ammonium-sulphate-exchange process.—In the ammonium-sulphate-exchange process the essential steps are a reaction of a mixture of clay and ammonium sulphate at 373°C . to 400°C . in a rotary kiln to form ammonium aluminum sulphate (alum) and ammonia. The mass is then leached with dilute sulphuric acid and filtered. The alum is recovered by crystallization, dissolved in dilute sulphuric acid, and aluminum hydroxide is precipitated by ammonia saved from the first operation. Ammonium sulphate formed in the last step is recycled and the aluminum hydroxide calcined to alumina.

Sulphuric acid leaching process.—Several acid processes have been developed, but in general it can be said that these processes are not entirely satisfactory either because of high iron in the alumina produced or because of corrosion of the metal equipment used.

LABORATORY TESTS

Our investigation began with the idea that if limestone were used in the soda-sintering process to form an insoluble silicate with the large amount of silica present, a process could be developed for extracting alumina from clay. This method, at first thought to have been original with us, was later found to have been investigated by several others (Kammermeyer and White, 1940). The process finally developed by me makes use of the basic procedure outlined, but differs in some details from any other process known to me. The different operations required in the soda-lime sintering process are given below:

- Mixing the clay, soda, and lime in proper proportion
- Sintering the mixture at high temperature
- Leaching to separate soluble sodium aluminate
- Purifying the sodium aluminate solution
- Precipitation of aluminum hydroxide
- Calcination of aluminum hydroxide to alumina.

Our work, after first demonstrating the process to be theoretically sound, was to determine optimum conditions of operation so that various important factors, such as composition of the sinter, temperature, time, and concentration and purity of solutions, could be controlled. The chemical composition of the raw materials used is shown in table 2.

MIXING THE CHARGE AND SINTERING

The first sintering charges contained 1 gram of calcined clay; later the amount was increased successively to 4, 31.6, and 44.6 grams. All material was ground in a mortar to pass through a 100-mesh sieve. The total weights of the different charges used are shown in table 3.

During the course of the experimental work described in this report, the proportions of the various ingredients found to be most satisfactory were approximately as follows: 2 gram mols of calcium oxide (CaO) to 1 gram mol of silica (SiO_2) and 1 gram mol of sodium oxide (Na_2O) to 1 gram mol of alumina (Al_2O_3). In the practical operation of this process it must be recognized that Al_2O_3 is replaced by R_2O_3 — R_2O_3 consists of Al_2O_3 plus varying amounts of Fe_2O_3 with or without TiO_2 . It was found by experiment that

TABLE 2.—*Chemical analyses of raw materials*
(Analyses by R. F. Thompson and Russell Runnels, in the Geological Survey Laboratories)

Material	Composition (percent)								
	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	K ₂ O + Na ₂ O	TiO ₂	Ign. loss	Total
Clay ¹ (dried 105° C.)	58.40	2.12	27.24	0.64	0.43	0.84	1.19	7.84	98.70
Clay ¹ (calcined 700° C.)	63.36	2.30	29.55	0.69	0.47	0.91	1.29	0.00	98.57
Sodium carbonate (Na ₂ CO ₃)			chemically pure						
Calcium carbonate ² (CaCO ₃)			chemically pure						
Limestone from Smith county, Kansas ²	2.12	0.59	1.15	53.36	0.56	³	³	42.02	99.80

¹ Dried clay and calcined clay were used separately in different charges.

² Calcium carbonate and limestone were used separately in different charges.

³ Not determined.

an excess or deficiency of CaO or an excess or deficiency of Na₂O would result in a lower alumina extraction.

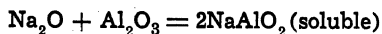
The charges were sintered under oxidizing conditions in platinum vessels that were placed in covered fire-clay crucibles set in an upright cylindrical gas furnace. A thermocouple tube was placed through a hole in the furnace to within one-half inch of the clay crucible. The temperature and the time of sintering were varied with the type of clay and the nature of the charge. It was found that the presence of iron oxide in the charge lowers the sin-

TABLE 3.—*Weights of sintering charges*

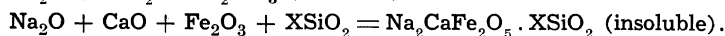
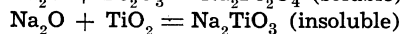
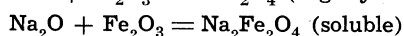
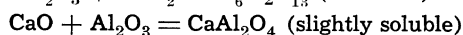
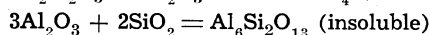
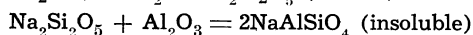
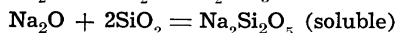
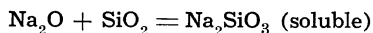
Material	Weight, in grams			
Raw clay	1.0	4.0	31.6	44.6
Calcium carbonate (CaCO ₃)	2.19	8.76	69.2	97.67
Sodium carbonate (monohydrate) (Na ₂ CO ₃ · H ₂ O)	0.40	1.60	12.6	17.80
Total weight of charge	3.59	14.36	113.4	160.07

tering temperature by a measurable amount, and it is believed that the presence of titanium oxide will raise the sintering temperature. Best results have been obtained by raising the temperature gradually and holding it at 1,375° C. for one-half hour. It was found that maintaining peak temperature for a longer time did not increase extraction and may cause a loss of soda. No check was made on soda other than to determine the amount recovered in the sodium aluminate solutions. At 1,375° C. the extraction of alumina is 75 percent. It was found by the U.S. Bureau of Mines (Personal communication from Oliver C. Ralston dated April 1, 1943) in testing clays from other areas that the top permissible temperature was 1,140° C. When the Kansas raw material studied was sintered at this peak temperature an extraction of only 20 percent alumina was obtained. A good sinter from the material studied by me will be greenish-brown in color, hard, compact, uniform except for a slight surface glaze, and will show much shrinkage. A light-colored sinter with little shrinkage, which is porous and easily crushed, will give poor extraction. There is probably some danger of the hydrolyzation of sodium aluminate to insoluble aluminum hydroxide; therefore, a sinter should be leached as soon as possible.

On sintering a charge containing clay, calcium carbonate, and sodium carbonate, the principal reactions are as follows:



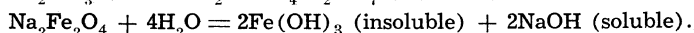
An ideal sinter in which all of the main constituents of the charge are found in these compounds is never realized because of other reactions, some of which follow:



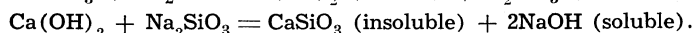
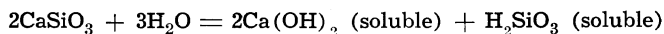
Some of these reactions occur to a considerable extent; others less so. The aluminum silicates and sodium titanate represent losses of aluminum and sodium.

LEACHING THE SINTER

The important factors in leaching are the volume and temperature of the water solvent, fineness of the sinter, and time of leaching. Sodium aluminate dissolves with the addition of water, and the following reactions may also occur:



Some calcium silicate will dissolve and may remain in solution or be reprecipitated, as follows:



The first reaction causes an alumina loss but prevents the entrance of silica into the filtrate.

On filtering, the sodium aluminate enters the filtrate, as do small amounts of silica, iron, lime, titanium, and alkalies—all objectionable because of the difficulty of separating them from the aluminum hydroxide when it is precipitated. The silica is probably present as silicic acid (H_2SiO_3), sodium silicate (Na_2SiO_3), or aluminosilicic acid; lime as calcium aluminate (CaAl_2O_4) or calcium hydroxide ($\text{Ca}(\text{OH})_2$); alkalies as sodium or potassium silicates or hydroxides. Iron and titanium theoretically should not be in soluble form at the end of the leaching period. Their presence in the alumina, unless by "salting" in the case of iron, is difficult to explain.

It was found that high concentrations of aluminate solutions give a means of partially controlling impurities. For practical reasons the aluminate concentration should be as high as possible. Such highly concentrated solutions have much less solvency for impurities, especially compounds of silica and lime. In our first experiments the concentration of alumina was less than 1 gram per liter, and while extraction was good, the quality of the alumina was poor. Subsequently, by using our purification process, to be described later, and the same concentration, a medium-grade alumina was made. Our present practice employs solutions of 10 to 30 grams per liter, uses purification, and yields high-grade alumina. One known difficulty with rich solutions, which we never experienced, is the tendency of aluminum hydroxide to settle out before filtering is finished. In our tests, one of the 30 gram solutions stood 2.5 hours after filtering without any sign of

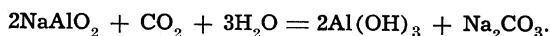
precipitation. The Bayer process uses solutions of a concentration of 80 grams. Another process in pilot plant stage uses 20 grams. For various reasons, it was impractical in our laboratory to produce solutions of greater concentration than 31 grams, although this should not be difficult when operating our process on a larger scale.

The proper temperature for water to be used in leaching is not definitely known. Sodium aluminate is reported as soluble in cold water and very soluble in hot water. Within the limits of our own experience, hot water has given slightly better extraction than warm water. For a test, a sinter was ground, mixed thoroughly, divided into two equal portions, and each leached under identical conditions as to volume of water and time, one with water at 45° C. and the other with water at 95° C. The lower temperature water extracted 74.0 percent of the alumina, whereas the higher temperature water extracted 74.35 percent. The sinter in all cases was ground to pass through a 20-mesh sieve. Mr. Oliver C. Ralston of the U.S. Bureau of Mines, however, wrote me stating that "Leaching with 80° C. water is almost certain to decrease extraction and our own experience has been that 50° C. is about as high a temperature as can be used."

Impurities are present in greater quantity in alumina produced by hot water leaching, as shown in table 5. This disadvantage probably outweighs that of better extraction; therefore, all things considered, hot water should probably be avoided. Different volumes of water were used in our leaching tests depending on the solution concentration desired. In general, the sinter was leached once for five minutes and given four separate washes, all with moderate agitation. The total time of leaching and filtering was about one-half hour.

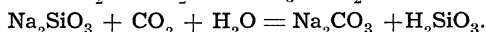
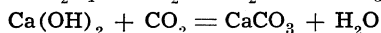
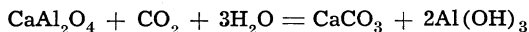
PURIFICATION OF SOLUTIONS AND PRECIPITATION OF ALUMINUM HYDROXIDE

In practice, aluminum is recovered from solutions as the hydroxide either by crystallization or precipitation by carbon dioxide, as follows:



In our process, carbon dioxide is a by-product of sintering, so this gas is used. Our first alumina, which was of poor quality,

was produced from the hydroxide obtained from unpurified solutions of low concentration. This alumina contained an average of 0.40 percent silica and 0.60 percent lime. While other investigators believed that high-grade alumina could be produced in this manner, we finally abandoned the technique as unsuccessful when used on the raw materials being investigated. In order to complete the precipitation of aluminum hydroxide, it is necessary to make the solution acid with carbon dioxide. When the gas is being added but before the solution becomes acid, calcium carbonate and silicic acid precipitate. The following reactions are believed to occur:



When the solution becomes acid, calcium carbonate dissolves and freshly precipitated silicic acid is believed to dissolve.

In some cases the aluminum hydroxide precipitate formed was found to contain impurities soluble in water. In spite of their solubility, it was impossible to remove them completely from the precipitate by washing. For this reason, it is necessary to precipitate the aluminum hydroxide from nearly pure solutions in order to produce a good grade of alumina. The carbon dioxide method of fractional precipitation for the removal of impurities from solutions was developed through our efforts to produce pure alumina. So far as known, the method is a new contribution to the soda-lime sintering process. This procedure is based on the principle that when a small amount of carbon dioxide is added to aluminate solutions the soluble impurities present in the alkaline solution precipitate in preference to the aluminum compounds, and can be removed by filtration. The reactions involved are shown above. No doubt the flocculent calcium carbonate precipitate aids in settling the silicic acid.

The quantity of gas used in purification varies with the amount of impurity present, which in turn depends on the concentration of the aluminate solutions. In our tests, approximately 30 c.c. per liter was used on impure solutions containing 1 gram per liter of alumina, while for purer solutions, containing 30 grams of alumina per liter, 15 c.c. per liter was employed. Determination of the correct amount is not difficult as there is an appreciable time interval after the lime is precipitated before the alumina begins to precipitate. Also the solution can be tested with calcium oxalate,

if necessary, to determine if lime still remains in solution. In our practice, the solutions were heated to 50°C. before the first injection of carbon dioxide, and later to the boiling point to remove the excess carbon dioxide before filtering off the precipitated impurities. The cooled solution was then heated to 75° C. and the aluminum hydroxide precipitated with additional carbon dioxide.

Table 4 shows the weight and composition of the purification precipitate and the alumina recovery in the alumina extraction precipitate on various tests made with 4-gram calcined clay samples.

TABLE 4.—*Chemical analyses of purification precipitates and extraction of alumina from charges containing 4 grams of calcined clay.*

No.	Weight of precipitate (grams)	Purification precipitate					Alumina	
		Composition of precipitate (percent)					Grams of alumina	Extraction (percent)
		SiO ₂	CaO	Fe ₂ O ₃	Al ₂ O ₃	Total		
41	0.29	5.10	78.6	0.1	13.1	96.9	0.83	70
43	0.083	3.60	84.0	0.3	9.7	97.6	0.84	71
44	0.063	6.00	80.3	0.8	10.2	97.3	0.92	79
46	0.133	3.07	86.2	0.4	6.7	96.4	0.88	75
52	0.22	3.46	85.0	0.2	8.0	96.6	0.83	70

In commercial practice it might be found desirable to recycle the purification precipitate to the sinter feed. A portion of the small amount of alumina present in the form of Al(OH)₃ in this precipitate was precipitated from calcium aluminate by carbon dioxide, as shown previously. Some aluminum hydroxide is also present for the reason that the carbon dioxide added to the solution forms bubbles which rise to the surface as a froth, and the gas in the bubbles in contact with the aluminate solution causes precipitation of the hydroxide.

The method of purification described above was found to greatly improve the quality of our alumina. Average samples of alumina produced by this method contained about 0.15 percent silica and 0.40 percent lime. Up to this point in all of our experiments we used charges containing only 4 grams of clay which produced an aluminate solution of less than 1 gram of alumina per liter. Greater improvement might have been effected under these

conditions, but in the meantime we found that by using larger charges and more concentrated solutions high-purity alumina could be more easily obtained.

RESULTS USING NATURAL LIMESTONE FLUX

A few tests were made using natural limestone from Smith county, Kansas, in sintering mixtures containing 4 grams of clay in place of pure calcium carbonate. The best results, taking into consideration the additional alumina in the limestone, showed a 75 percent extraction of alumina containing 0.18 percent silica and 0.46 percent lime. The use of natural limestone did not yield good results on first trial, but, theoretically at least, there is no reason why, with proper fluxing, this material should not yield results comparable with those obtained using pure calcium carbonate. Further tests using natural limestone are needed; as yet none have been made using large charges where this process shows the best results.

RESULTS USING LARGE CHARGES

In the first stages of our work, charges containing a total of 14.36 grams were considered as large as our equipment would permit. Later realizing the value of larger units, as was also suggested by Oliver Ralston (Personal communication dated April 1, 1943) of the U.S. Bureau of Mines, we enlarged our equipment to sinter as much as 160 grams at one time. The use of larger charges has the advantage of approaching conditions found in commercial practice. In treating these larger charges in the laboratory the same amount of leaching water was found to produce a greater concentration of alumina in solutions of much greater purity. A disadvantage in our case was the impossibility of determining alumina extraction accurately on account of the large amount of sinter lost by its sticking to the platinum crucible and later in grinding. A chemical analysis of alumina recovered from a 160-gram charge and specifications for alumina for reduction to airplane sheet aluminum are shown in table 5. In this particular case, the sinter was divided, one half being leached with water at 45° C. and the other half with water at 75° C. The results shown in table 5 are characteristic of the alumina that was produced by this process using large charges and with carbon dioxide purification of solutions. Chemical analyses of the sintering charge, sinter, and residue are shown in table 6.

TABLE 5.—*Chemical analysis of alumina from a 160-gram charge and specifications for alumina for reduction to airplane sheet aluminum*

Constituent	Chemical specifications for alumina for reduction to airplane sheet aluminum	Alumina sample No. 6 leached with water at 45° C	Alumina sample No. 6 leached with water at 75° C
	(percent)	(percent)	(percent)
SiO ₂	0.02, tolerance to 0.06	0.017	0.06
Fe ₂ O ₃	0.02, tolerance to 0.04	0.04	0.04
CaO	0.04	0.056 ¹	0.07 ¹
TiO ₂	0.004	Trace too small to determine	Too small to determine
MgO	0.02	Trace	Trace
K ₂ O	0.05	Trace	Trace
Na ₂ O	0.60	Na ₂ O + K ₂ O = 0.23 K ₂ O = Trace	Not determined

¹ The slightly high lime is a matter of little importance; otherwise the comparisons are favorable.

TABLE 6.—*Chemical analyses of the sintering charge, sinter, and residues*

Material	Weight (grams)	Composition (percent)									
		SiO ₂	Fe ₂ O ₃	CaO	Al ₂ O ₃	MgO	K ₂ O + Na ₂ O	TiO ₂	CO ₂	H ₂ O	Total
Sintering charge using calcined clay	14.36	17.59	0.64	34.36	8.20	0.01	5.80	0.35	30.55	1.61	99.11
Sinter 1	8.1	25.64	1.00	51.06	13.93		not determined				91.63
Residue 1	5.3	29.30	1.02	59.30	5.1		not determined				94.72

TREATMENT OF THE RESIDUE

The weight of the residue, containing approximately 25 percent of the alumina originally present in the clay treated, was found to be much less than half the weight of the original charge. The residue may be of value for recycling or for its possible use in making cement, rock wool, building blocks, or other by-products.

On resintering and leaching the residue, about one half the alumina present can be recovered without further fluxing, which would bring the total recovery in the process to 85 percent. This alumina, however, contains as much as 3 percent silica; because efforts to increase its purity have thus far failed, we have not incorporated this operation in the main process. Resintering is inex-

pensive in regard to gas, and little, if any, high-cost soda ash is required. According to Norman Plummer of the State Geological Survey, who has made a detailed study of the refractory clays of Kansas, the alumina which could be recovered by resintering and leaching would be sufficiently pure for use in the manufacture of high-alumina refractories. The plastic fire clays which are here considered as an ore of alumina would also be suitable for use as a bonding material in such refractories. The composition of the residue approaches Portland cement and it might be used in cement or rock-wool manufacture. The material sets to a hard mass on standing and might be used in the manufacture of easily molded building blocks. Therefore, the residue appears to have some value, but its best utilization must await results of future research.

THE RECOVERY OF SODIUM CARBONATE

The sodium present in sodium aluminate leached from the sinter remains in solution when aluminum hydroxide is removed by filtering. The ratio of Na_2O to Al_2O_3 in $\text{Na}_2\text{Al}_2\text{O}_4$ is 0.61 : 1.0. The amount of Na_2O present in the solution is directly proportional to the weight of alumina recovered. With a 75 percent alumina extraction, 67 percent of the soda in the original charge theoretically should be available and could be recovered by evaporation and crystallization.

ORE-DRESSING TREATMENT OF CLAY

The ores of the nonferrous metals, being for the most part of low grade, are usually given an ore-dressing treatment for the removal of much of the worthless portion before extraction of the metal is attempted. Kaolin, which is one type of clay, consists dominantly of kaolinite or hydrous aluminum silicate. This mineral contains 39.5 percent alumina. The type of Kansas clay used in these experiments contains less alumina and more impurities (mostly silica) mechanically admixed with the clay minerals. Norman Plummer has developed a sedimentation or gravity process for treating clay pulps in which a fractional separation of much of the heavier free silica is made, leaving an enriched alumina product. On the basis of laboratory results, 5 tons of raw clay containing 27 percent alumina will yield 2.5 tons of a product con-

taining 37 per cent alumina and the same weight of low-grade material. Since clay is cheap, the latter can be discarded. The estimated total cost of the enriched clay is \$0.62 per ton.

The small additional cost for preliminary concentration of raw clay is more than compensated for by the saving in labor and materials in the alumina extraction process, as shown on pages 133-134.

ESTIMATED COST OF PRODUCING ALUMINA FROM KANSAS RAW MATERIALS

The estimated cost of producing 1 ton of alumina in a plant with a capacity of 20 tons per day from raw clay containing 27.24 percent alumina and assuming an extraction of 75 percent, is given below:

Raw materials

Clay: 4.9 tons @ \$0.20	\$ 0.98
Limestone: 10.2 tons @ \$1.50	15.30
Soda ash ¹ : 0.52 tons (lost in process) @ \$20.00	10.40

Gas consumed

Sintering 16.7 tons (charge required to produce 1 ton of alumina) at 1,375° C. The quantity of gas consumed has been calculated as follows: 1 gram × specific heat of clay × degrees centigrade × conversion of calories per gram to B.t.u. × pounds per ton × weight of charge ÷ calorific power of gas in B.t.u. × efficiency factor = thousands of cubic feet.

$$\frac{1 \times 0.2 \times 1,375 \times 1.8 \times 2,000 \times 16.7}{900 \times 0.57} = 32.20 \text{ M cu. ft.}$$

Calcining 1 ton $\text{Al}(\text{OH})_3$ to Al_2O_3 at 1,000° C.

$$\frac{1 \times 0.2 \times 1,000 \times 1.8 \times 2,000 \times 1}{900 \times 0.57} = 1.40 \text{ M cu. ft.}$$

Total gas: 32.20 + 1.40 = 33.60 M cu. ft. @ \$0.10..... 3.36

Waste heat used on other processing

Water 0.25

Power

Crushing 4.9 tons clay	
Crushing 10.2 tons limestone	
Total crushing 15.1 tons @ \$0.10	\$ 1.51
Rotate kiln with 16.7 tons @ \$0.10.....	1.67
Other power and light	0.25
Total power	3.43

¹Based on 67 percent recovery in processing.

Labor

Three shifts of 15 men each: $\frac{45 \times \$5.00}{20}$ 11.25

Overhead expense

Plant cost \$150,000, interest 6 percent, depreciation 10 percent

$\frac{150,000 \times 0.16}{20 \times 365}$ 3.28

Total cost per ton¹ \$48.25

Total cost per pound 0.02412

The estimated cost of producing 1 ton of alumina in a plant with a capacity of 20 tons per day from concentrated clay containing 37 percent alumina and assuming an extraction of 75 percent, is given below:

Raw materials

Clay: 3.6 tons @ \$0.62 \$ 2.23

Limestone: 6.2 tons @ \$1.50 9.30

Soda ash²: 0.51 tons (lost in process) @ \$20.00 10.20

Gas consumed

Sintering 11.32 tons (charge required to produce 1 ton of alumina) at 1,375° C. The quantity of gas consumed has been calculated as follows: 1 gram \times specific heat of clay \times degrees centigrade \times conversion of calories per gram to B.t.u. \times pounds per ton \times weight of charge \div calorific power of gas in B.t.u. \times efficiency factor = thousands of cubic feet.

$\frac{1 \times 0.2 \times 1,375 \times 1.8 \times 2,000 \times 11.3}{900 \times 0.57} = 21.8 \text{ M cu. ft.}$

Calcining 1 ton $\text{Al}(\text{OH})_3$ to Al_2O_3 at 1,000° C.

$\frac{1 \times 0.2 \times 1,000 \times 1.8 \times 2,000 \times 1}{900 \times 0.57} = 1.40 \text{ M cu. ft.}$

Total gas 21.8 + 1.40 = 23.2 M cu. ft. @ \$0.10 2.32

Waste heat used on other processing

Water 0.20

Power

Crushing 3.6 tons clay

Crushing 6.2 tons limestone

Total crushing 9.8 tons @ \$0.10 \$ 0.98

¹Value of residue not included.

²Based on 67 percent recovery in processing.

Rotate kiln with 11.32 tons @ \$0.10.....	1.13	
Other power and light	0.25	
Total power		2.36
Labor		
Three shifts of 13 men each: $\frac{39 \times \$5.00}{20}$		9.75
Overhead expense		
Plant cost \$150,000, interest 6 percent, depreciation 10 percent		
$\frac{150,000 \times 0.16}{20 \times 365}$		3.28
Total cost per ton ¹		\$39.64
Total cost per pound		0.01982

PRICES

In general, aluminum producers control all necessary operations from the mining of the ore to the production of the metal. This includes the production of alumina, which is rarely sold on the open market as it is prepared by producers for their own use. Market quotations are available for aluminum ore, but not for alumina.

Mr. R. C. Rand of the Non Ferrous Metals Division, Office of Economic Warfare, Washington, D.C., states in a letter dated September 17, 1943: "I can only give you my estimate that if there were a free market, domestic prices for domestic high-grade alumina would be between 2¾ and 3¼ cents per pound." This is equivalent to \$55.00 to \$65.00 per short ton. Other information indicates that alumina is being sold in the St. Louis area for \$50.00 per ton and shipped to the State of Washington at a freight cost of \$10.80 per ton.

In the absence of an official market price, it would seem that a conservative estimate of the present value of high-grade alumina would be between \$50.00 and \$55.00 per short ton. The most recent quotation available to me of the price of aluminum is 15 cents per pound.

CONCLUSIONS

The commercial success of an ore-treating process depends on the cost of production and the quality of the product produced.

¹Value of residue not included.

These are controlled by the efficiency of the method used, labor, and the cost of supplies. The latter two factors may differ locally. No accurate comparison of production costs of the soda-lime sintering process with other processes recently developed can be made because of lack of data on the other new processes. The operation of this process is simple and the results obtained are considered favorable. It seems probable that the process can be further improved by continued research. Kansas contains a greater percentage of the native raw materials needed for the operation of this process than would be used in any other process known at the present time. The only material not available in the state is soda ash, the manufacture of which requires limestone, salt, and ammonia. We have an abundance of limestone and salt and only a small amount of ammonia is needed as this is recovered and recycled in the process. For commercial success, the required soda ash should be manufactured locally. Also, it would no doubt be a great help if the clay were given a preliminary ore-dressing treatment before processing.

Laboratory tests on a fairly large scale have demonstrated this process to be both theoretically sound and practical; it should, however, be operated on a pilot plant scale to secure additional data before considering the erection of a commercial plant.

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STATE GEOLOGICAL SURVEY OF KANSAS

Recent Publications

- BULLETIN 27. Ground-Water Resources of Kansas, by Raymond C. Moore with chapters by S. W. Lohman, J. C. Frye, H. A. Waite, T. G. McLaughlin, and Bruce Latta, 112 pages, 1940. Mailing charge, 25 cents.
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BULLETIN 41

1942 REPORTS OF STUDIES

- PART 1. GROUND-WATER SUPPLIES AVAILABLE FOR NATIONAL DEFENSE INDUSTRIES IN SOUTH-CENTRAL KANSAS, by Stanley W. Lohman, pp. 1-20, figs. 1-5, February 28, 1942.
- PART 2. GROUND-WATER SUPPLIES AVAILABLE IN KANSAS FOR NATIONAL DEFENSE INDUSTRIES, by S. W. Lohman, J. C. Frye, H. A. Waite, V. C. Fishel, T. G. McLaughlin, B. F. Latta, and G. E. Abernathy, with a summary of stream flow in Kansas, by G. S. Knapp and J. B. Spiegel, pp. 21-68, figs. 1-3, pls. 1-4, April 24, 1942.
- PART 3. KANSAS MINERAL RESOURCES FOR WARTIME INDUSTRIES, by John M. Jewett and W. H. Schoewe, pp. 69-180, figs. 1-13, May 9, 1942.
- PART 4. MAP: MINERAL RESOURCES OF KANSAS, by R. C. Moore and J. C. Frye, 45 by 32 inches (5 colors), April, 1942.
- PART 5. LOPOPHYLLID CORALS FROM LOWER PENNSYLVANIAN ROCKS OF KANSAS AND OKLAHOMA, by Russell M. Jeffords, pp. 185-260, pls. 1-8, figs. 1-2, June 12, 1942.
- PART 6. PLEISTOCENE MAMMALS FROM KANSAS, by Claude W. Hibbard, pp. 261-269, pl. 1, August 3, 1942.
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- PART 8. MINERAL RESOURCES OF PHILLIPS COUNTY, by Kenneth K. Landes and Raymond P. Kerohar, pp. 277-312, pls. 1-4, figs. 1-5, August 22, 1942.
- PART 9. STRATIGRAPHY OF THE PRE-GREENHORN CRETACEOUS BEDS OF KANSAS, by Norman Plummer and John F. Romary, pp. 313-348, pls. 1-2, figs. 1-4, November 30, 1942.
- PART 10. KANSAS BENTONITE: ITS PROPERTIES AND UTILIZATION, by E. D. Kinney, pp. 349-376, pls. 1-2, fig. 1, December 14, 1942.

BULLETIN 47

1943 REPORTS OF STUDIES

- PART 1. NEW CARBONIFEROUS AND PERMIAN SPONGES, by Ralph H. King, pp. 1-36, pls. 1-3, figs. 1-2, February 27, 1943.
- PART 2. KANSAS OIL FIELD BRINES AND THEIR MAGNESIUM CONTENT, by Walter H. Schoewe, pp. 37-76, figs. 1-3, June 30, 1943.
- PART 3. DEEP WATER WELL AT THE JAYHAWK ORDNANCE WORKS IN CHEROKEE COUNTY KANSAS, by G. E. Abernathy, pp. 77-112, figs. 1-4, September 10, 1943.
- PART 4. A PROCESS FOR EXTRACTING ALUMINA FROM KANSAS CLAY, by E. D. Kinney, pp. 113-136, December 10, 1943.