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ROLE OF ALUMINA-TO-SILICA MOLE RATIO IN THE LIME-SODA SINTER PROCESS

UNITED STATES DEPARTMENT OF THE INTERIOR

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CONTENTS

ILLUSTRATIONS

TABLES

ROLE OF ALUMINA-TO-SILICA MOLE RATIO IN THE LIME-SODA SINTER PROCESS

by

R. V. Lundquist[1] and D. D. Blue[2]

ABSTRACT

Past reports by the Bureau of Mines have described the extraction of alumina from numerous aluminum silicate materials by the lime-soda sinter process. The results from pilot-plant investigations of six materials, with alumina-to-silica mole ratios from 0.3 to 8.7, indicated a relationship between the alumina-silica ratios and the processing characteristics of the sinter. A series of laboratory tests was conducted to define this relationship. Sinter and leach tests determined that an alumina-to-silica mole ratio of 0.90 or greater resulted in the best alumina and soda recoveries while leaving the leach slurries practically free from any tendency toward gelation. The widely different chemical and mineralogic properties of the various aluminous materials used in compounding the mixes had no perceptible effect on the leaching characteristics of the sinter. The blending of low-cost, high-alumina materials with aluminum silicate materials to give an alumina-to-silica mole ratio of 0.90 or greater would improve alumina and soda extraction and minimize operating problems in the lime-soda sinter process.

INTRODUCTION

Silicon in the form of oxides or silicates creates difficulties in the commercial processing of bauxites for the recovery of alumina by the Bayer process. The lime-soda sinter process was developed many years ago for recovering alumina from siliceous materials other than bauxites, but it was not economically competitive. Ultimately a combination of the two processes permitted the competitive use of bauxite ores with increased silica content. However, the presence of silica still created processing difficulties in the lime-soda sinter portion of the combination process. Even greater problems were encountered when the lime-soda sinter process was applied to anorthosites and other siliceous rocks.

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[1]Formerly supervisory metallurgist, Boulder City Metallurgy Research Laboratory, Bureau of Mines, Boulder City, Nev., now retired.
[2]Supervisory metallurgist, Boulder City Metallurgy Research Laboratory, Bureau of Mines, Boulder City, Nev.

The Bureau of Mines has reported pilot-plant investigations of the lime-soda sinter process for the recovery of alumina from anorthosite (2, 25),[3] kaolin (6), high-silica bauxite (5), diaspore clay (4), and high-iron bauxite (7). The silica in these ores decreased, in the order given, from 52.7 percent in anorthosite to 3.6 percent in high-iron bauxite, and the respective alumina recoveries rose from 65 percent to 95 percent. A summary of this work is presented in table 1.

TABLE 1. - Alumina and soda recovery from pilot-plant extraction of aluminum ores at various alumina-silica mole ratios

Type of ore	Al_2O_3-to-SiO_2 mole ratio	Recovery, percent	
		Al_2O_3	Na_2O
Anorthosite (25)	0.29	65	73
Anorthosite (2)	.29	73	68
Kaolin (6)	.50	85	77
High-silica bauxite (5)	1.44	88	86
Diaspore clay (4)	1.77	89	94
High-iron bauxite (7)	8.73	95	99

Gelation of the leach slurries was an operating problem in all the pilot-plant studies except the one concerned with a high-iron bauxite containing only 3.6 percent silica. Other reports (9, 14, 19-20) also describe a variety of observable effects related to gelation in leach slurries. Little is known about the chemistry of gelation. Perhaps the nearest related knowledge is found in the chemistry of portland cement. Tricalcium silicate ($3CaO \cdot SiO_2$), which is an important phase in cement clinker, contributes strongly to the setting properties of cement (1, 3, 8, 11, 22). This phase is also present in most lime-soda sinters (14). This report describes how gelation of and alumina extraction from the leach slurry are related to the alumina-silica ratio of the lime-soda sinter.

RAW MATERIALS

The raw materials used in preparing sinters were anorthosites from the San Gabriel Mountains, Los Angeles County, Calif. (12); the Carlton Peak area, Lake County, Minn. (10); and the Laramie Mountains, Albany County, Wyo. (25); Arkansas bauxite from the Aluminum Company of America, Bauxite, Ark.; kyanite flotation concentrate from Aluminum Silicates, Inc., Washington, Ga. (21); and a commercial grade of calcined alumina. The compositions of these materials are listed in table 2. Commercial limestone and soda ash were blended with these materials to obtain the desired sinter compositions.

[3] Underlined numbers in parentheses refer to items in the list of references at the end of this report.

TABLE 2. - Composition of aluminous raw materials used in tests, percent

	Al_2O_3	SiO_2	CaO	Na_2O	Fe_2O_3	LOI[1]
Anorthosite:						
California..................	27.0	53.8	9.5	6.1	2.4	([2])
Minnesota..................	30.8	47.8	14.9	2.3	1.9	([2])
Wyoming....................	26.7	52.6	11.3	3.3	2.9	([2])
Bauxite: Arkansas............	43.7	29.4	0	.33	4.1	19.7
Kyanite: Georgia.............	49.5	39.3	.06	.08	7.5	1.2
Calcined alumina.............	98.6	.03	([2])	.50	.04	.75

[1]Loss on ignition at 1,000° C.
[2]Not determined.

EXPERIMENTAL PROCEDURES

About 60 sinters were prepared to cover the Al_2O_3-to-SiO_2 mole ratio range of 0.29 (anorthosite) to 4.5 (approximating a relatively good bauxite ore). The natural materials, and in some cases calcined alumina, were proportioned in various combinations to obtain the selected mole ratio in the sinter. Three sinters were prepared for every mole ratio and material to verify observed data points and process characteristics.

The sinter mixes were prepared by proportioning the raw, minus 200-mesh materials to obtain mole ratios for $CaO:SiO_2$ and $Na_2O:Al_2O_3$ of 2.0 and 1.0, respectively; extra Na_2CO_3, equivalent to 12 percent of the total Na_2O required for the desired ratio, was added to the mix to compensate for sodium lost by volatilization during sintering. The proportioned raw materials were thoroughly blended dry for 30 minutes in a steel ball mill and then fed to a laboratory disk pelletizer.

A vibrating feeder controlled the feed rate of the dry sinter mix and an adjustable, fine, water spray controlled the moisture fed to the pelletizer. The rate of feed, the tilt, and the speed of rotation of the pelletizer disk were adjusted to produce 1/4- to 3/8-inch-diameter pellets. Excellent pellets were obtained with 20 percent or less moisture content. The compressive strengths of the green pellets ranged from 4 to 20 pounds, and the strengths after drying the pellets at 110° C for 12 hours ranged from 15 to 60 pounds per square inch of cross section.

An updraft sintering furnace, developed in earlier work (12), was modified as shown in figure 1. The modification consisted of installing a silicon carbide tube liner, raising the iron grate about 2 inches, and supporting the grate with two iron bars. This modification reduced the furnace capacity from 10 to 5 kilograms of feed. The modified furnace was easier to operate, and the sintering was more easily controlled.

In starting the updraft sintering furnace, a 1-inch-thick layer of crushed petroleum coke was placed on the grate and ignited with a gas flame and downdraft. A mixed charge of pellets and coke was next placed on top of the ignited coke layer, the downdraft was shut off, and the updraft was started. The hot sintering zone moved up the length of the furnace shaft in

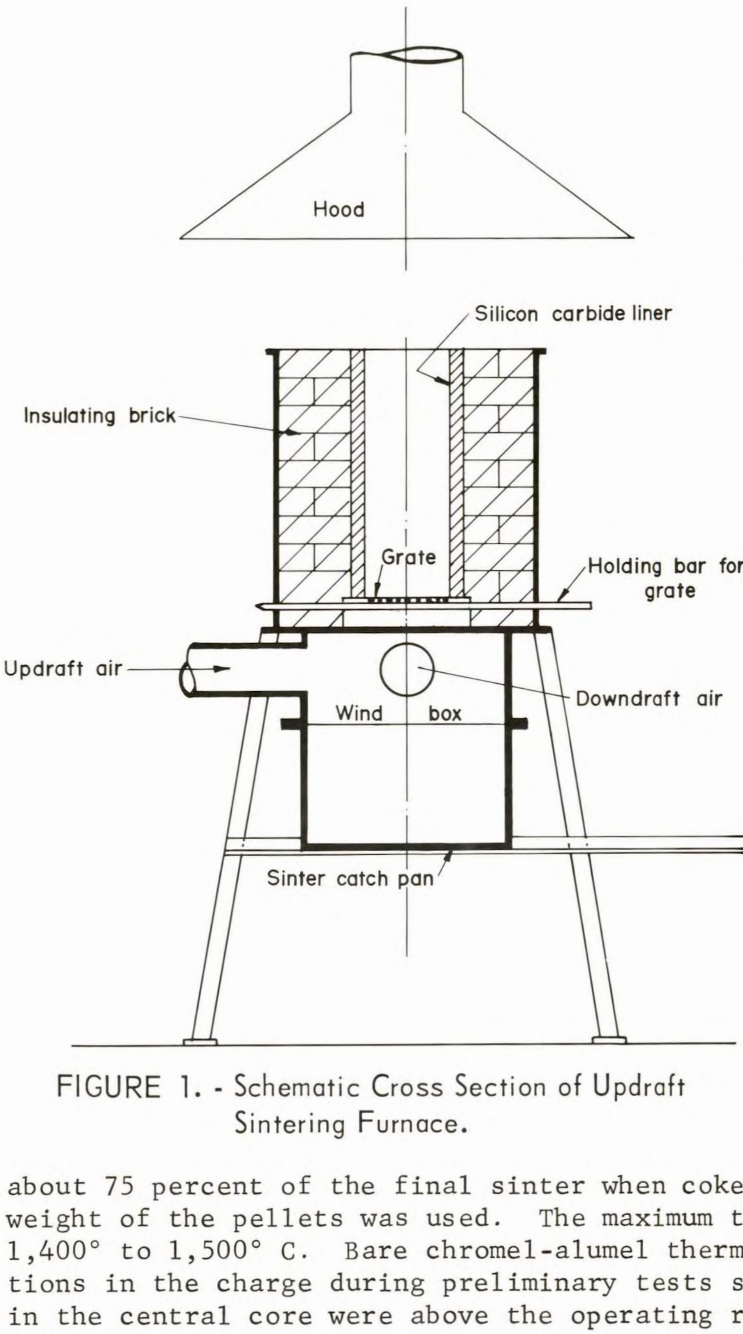

FIGURE 1. - Schematic Cross Section of Updraft Sintering Furnace.

about 20 minutes. The flame at the end of sintering was heavily laden with sodium vapor. Updraft air was continued for 30 minutes after completion of combustion to cool the sinter to about room temperature before removing it from the furnace. Heating and cooling rates during and after sintering were in the range of 60° to 100° C per minute.

Withdrawing the two iron bars dropped the sinter and grate into the pan below the furnace. About 3,500 grams of sinter was produced per furnace charge. Ignition and dust loss varied from 24 to 34 percent of the weight of the pellet charge.

The sinter formed in the updraft furnace was readily divided into two fractions: a hard central core in which the pellet surfaces had melted enough to flow and stick the pellets together to form a porous sinter in which the pellet shapes lost their identity, and a surrounding, relatively soft material in which no fusion had occurred. The quantity of central core was about 75 percent of the final sinter when coke equal to 30 percent of the weight of the pellets was used. The maximum temperature was estimated at 1,400° to 1,500° C. Bare chromel-alumel thermocouples placed in various locations in the charge during preliminary tests showed that maximum temperatures in the central core were above the operating range of the couples, some of which registered on the recorder up to 1,400° C. In a continuous operation the soft fraction would represent a recycle product, and the proportion would depend on the characteristics of the sintering furnace.

The hard fractions were ground to minus 48-mesh; more than 50 percent was minus 100-mesh. The ground sinters were subjected to a standard leach procedure to determine the recovery of alumina and soda and to a separate standard procedure to measure gelation. The sinters and residues were analyzed for Al_2O_3, SiO_2, Na_2O, and CaO (free and combined) content. The recovery of alumina and soda was calculated from the analysis of the sinters and the corresponding leached residues.

In the standard leach procedure, slurries with two ratios of solid to solution were used, one with 75 grams of sinter to 150 ml of leach solution, and the other with 50 grams of sinter to 200 ml of leach solution. The leach solutions contained 45 to 50 grams Na_2O per liter as either NaOH, Na_2CO_3, or equal proportions of each. In the leaching investigations, sucrose equal to 1 percent of the sinter weight was added with the sinter to depress gelation. The leach solutions were heated to 70° C in a water bath, and the sinter and sucrose were then added. The leach slurries were maintained at this temperature for 30 minutes while being vigorously stirred to keep the solids in suspension. The slurries were then immediately filtered. The filtered residues were washed by repulping in hot water and were then refiltered. The total volume of wash water was about 3 times the volume of the initial leach solution.

Gelation of lime-soda sinters (2, 4-6, 9, 19-20, 25) is described in numerous reports. Several of these investigations (4, 9, 19-20) relied on the use of various types of viscometers for measuring the gelation of the slurries. A drawback of such measurements was that they were obtained by intermittent readings of the viscometers. A continuous-recording viscometer was assembled to overcome the difficulties encountered in the interpretation of intermittent data.

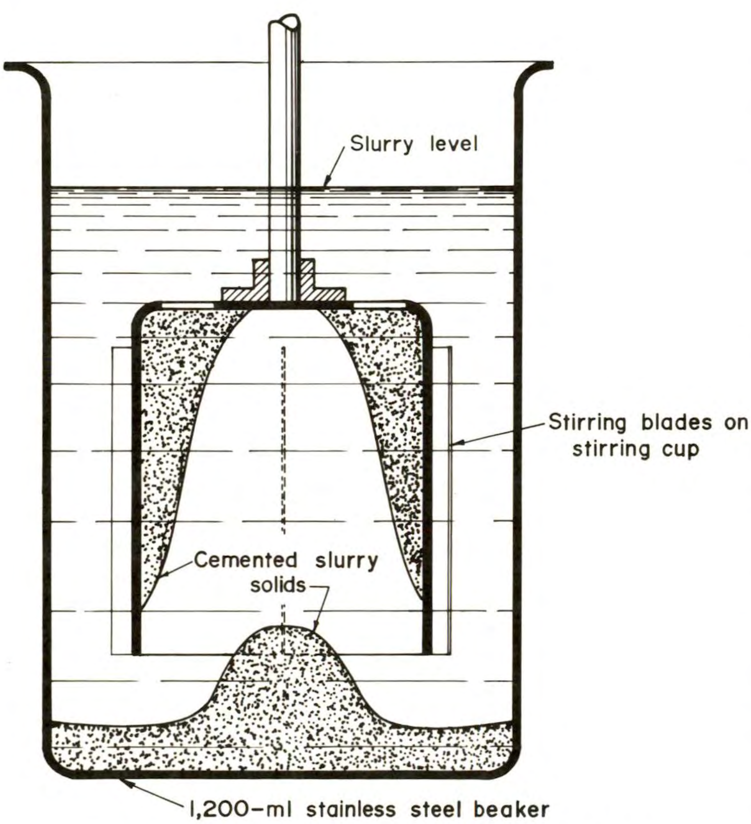

FIGURE 2. - Schematic Cross Section of Beaker Assembly Used With Continuous-Recording Viscometer.

The principal parts of this continuous-recording viscometer were a specially designed stirring motor and control unit, a recorder, and a beaker assembly for containing and stirring a lime-soda sinter slurry. The control unit for the motor automatically varied the power input to the motor to compensate for the changes in output torque required to maintain a constant, selected, motor speed as the consistency of the slurry changed. Recording the power input provided a continuous record of changes in output torque, which by appropriate calibration, was easily converted to centipoises. The beaker assembly consisted of a 1,200-ml stainless steel beaker and an inverted 150-ml stainless steel beaker converted into a stirring cup, as shown in figure 2. A 1/4-inch blade was formed by cutting an

L-shaped slit in the side of the stirring cup and bending the resulting flap outward tangentially to the cylinder to serve as a scoop for circulation of the slurry as the cup was rotated. Holes in the top of the stirring cup permitted upward circulation of the slurry. A circular recording chart was calibrated for reading apparent viscosities.

The recording viscometer was calibrated from 1 to 1,200 centipoises by using glycerol (1.25 specific gravity at 25° C) and mixtures of glycerol and water at temperatures of 10° to 50° C (23-24). A change of 24 millivolts of power input corresponded to a viscosity range of 1 to 1,200 centipoises. The data from the recording chart were readily converted to a rate of change of the logarithm (20) to compare tendencies for gelation.

To measure the viscosities of lime-soda sinter slurries, 800 ml of NaOH-Na_2CO_3 leach solution was heated to about 70° C. To this solution, 400 grams of sinter without any sucrose was added, and the mixture was vigorously stirred for about 1 minute. The slurry was transferred to the recording viscometer to determine changes in viscosity. The beaker containing the slurry was covered to minimize loss of water by evaporation, which was held to about 30 to 35 ml over a 24-hour period. The slurries cooled naturally to room temperatures (25° to 30° C) in 4 to 6 hours. The stirring cup was rotated at 240 rpm with a probable error in regulation of ±5 percent. The apparent viscosity readings taken from the recording chart were not corrected for ambient temperature changes or water evaporation.

EXPERIMENTAL RESULTS

The sinter mixes were compounded of the three anorthosites with bauxite, kyanite, and alumina to give the desired mole ratios of Al_2O_3 to SiO_2. The leaching and viscosity data obtained for the various sinters did not exhibit any characteristics that could be attributed to the raw materials used in preparing the mixes. The data presented are the weighted averages for the many determinations made on all of the sinters that had the same Al_2O_3-to-SiO_2 mole ratios without regard to the source of the raw materials.

The hard center core of the furnace product was produced under semimolten conditions during sintering. Some of the glassy phases had a hardness about equal to that of orthoclase. Chemical analyses of the hard sinter for calcium not chemically bound with silicates or aluminates showed less than 0.40 percent free CaO.

The soft, earthy, concentric sinter had a free calcium content of up to 5 percent. Extraction tests on the soft sinter invariably gave much lower soda and alumina recoveries. The soft sinter also exhibited greatly accelerated gelation characteristics. The only test data reported on such sinters are included in table 4 to show the effect of free lime on gelation rates and to characterize a typical fast-gelling sinter, as shown on the recording viscometer.

Alumina and soda recoveries were determined from leach slurries with a sinter-solution ratio of 1 to 2. The leach-recovery determinations made from

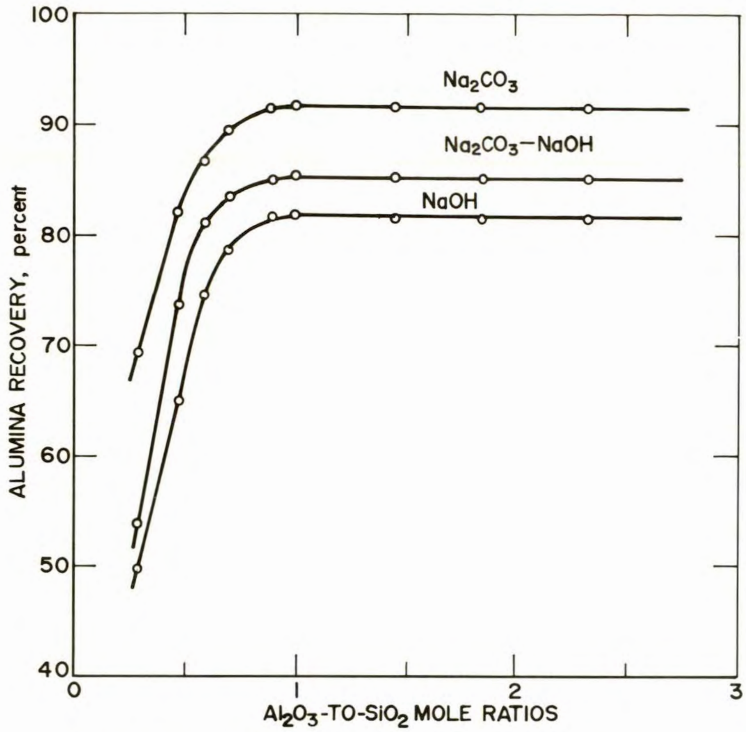

FIGURE 3. - Effect of Al_2O_3-SiO_2 Mole Ratios and Extraction Mediums on Alumina Recovery.

leach slurries with a sinter-solution ratio of 1 to 4 showed a slight increase in alumina recovery. The decrease of sinter solids, however, did not provide a sufficient advantage to warrant inclusion of the data.

Alumina recovery data obtained by leaching a sinter in solutions of NaOH and Na_2CO_3, either singly or as a mixture of equal volumes, are plotted versus the Al_2O_3-to-SiO_2 mole ratio in figure 3. Recovery increased sharply to a mole ratio of about 0.90 to 1.00 and remained relatively constant thereafter. Maximum recovery of alumina of 91.5 and 81.7 percent occurred in the Na_2CO_3 and NaOH leach solutions, respectively, at a mole ratio of 1.00. Intermediate recovery occurred in the mixed solutions of Na_2CO_3 and NaOH. This result shows that the optimum mole ratio for obtaining good alumina recovery in the lime-soda sinter process ranges from 0.90 to 1.00. Lower ratios decreased recovery and larger ratios did not show any apprecialbe increase. The data in figure 3 are in substantial agreement with the pilot-plant data shown in table 1.

Figure 4 shows that excellent soda recovery occurred in NaOH solutions when the Al_2O_3-to-SiO_2 mole ratios were greater than 0.60. However, solutions of Na_2CO_3 alone appear to depress soda recovery somewhat from those obtained in NaOH solutions. Soda recovery from mixed solutions of NaOH and Na_2CO_3 was about equal to recovery from NaOH solutions. The recovery of soda does not present any difficult problems.

Gelation of the leach slurry is a problem in the production of alumina by the lime-soda sinter process. The most prominent observable feature of gelation is a general thickening of the slurry, sometimes accompanied by cementation of the solids into lumps or cake and on to the surfaces of the extraction vessels. In the recording viscometer tests, cementation was a common occurrence. Figure 2 shows the areas in the viscometer where cemented solids were found. The hardness of cemented solids varied from relatively soft, meaning those easily broken, to hard, similar to freshly set concrete. In a few scattered instances, the cemented solids appeared as gravel about one-fourth inch in diameter in the slurry. The thickness of the cemented solids in the stirring cup varied from less than one-eighth inch to more than one-half inch; in

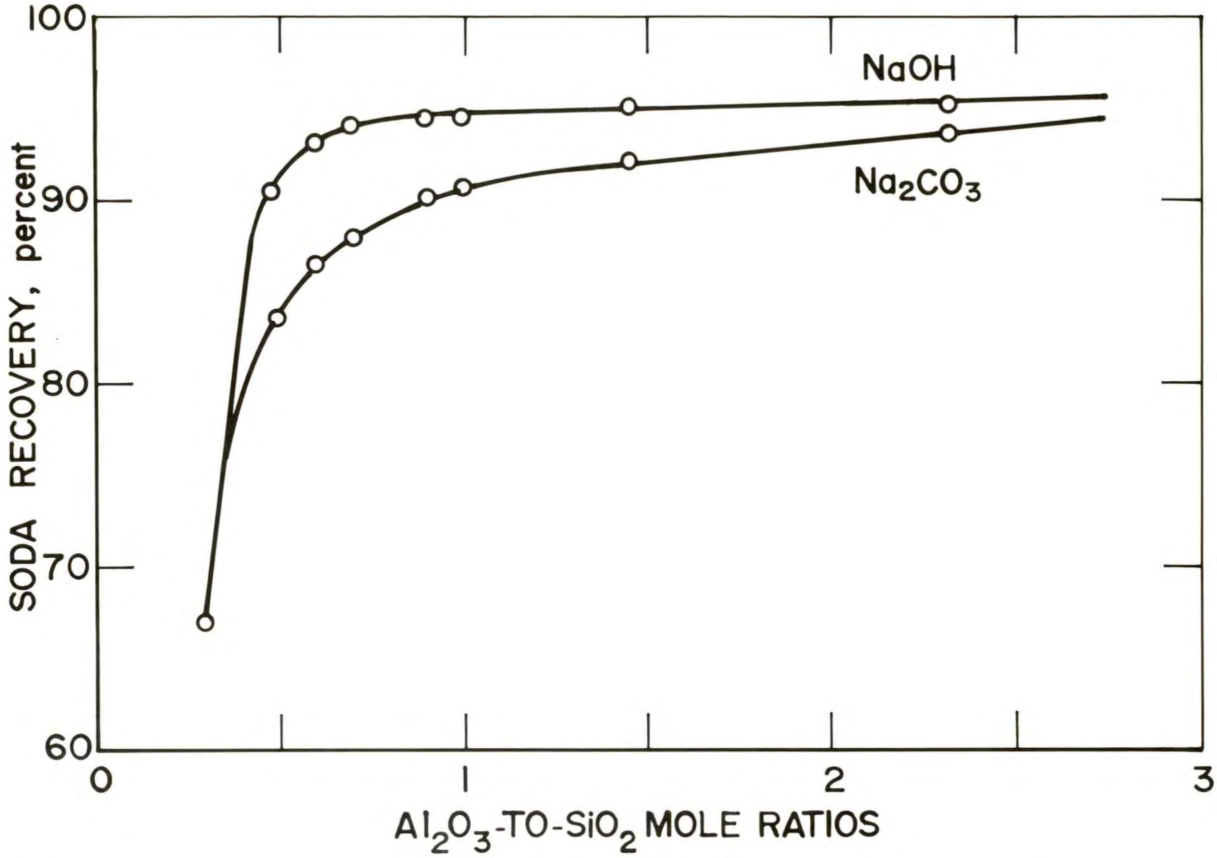

FIGURE 4. - Effect of Al_2O_3-SiO_2 Mole Ratios and Extraction Mediums on Soda Recovery.

the slurry beaker the height of the cemented mound varied from less than three-fourths inch to more than 1.5 inches.

A comparison of the sinters showed that, for those with Al_2O_3-to-SiO_2 mole ratios less than 0.90, cemented solids appeared in the stirring cup in 57 percent of the tests and 75 percent of these were hard; in the slurry beaker 100 percent appearance of cemented solids occurred of which 64 percent were set hard. Cementation of sinter solids was not observed in any slurry made from sinters with mole ratios greater than 0.90. In these tests, the sinter solids in the bottom of the slurry beaker were soft and possessed the properties of normally settled materials, even after standing 24 hours undisturbed.

The continuous measurement of viscosity provided a convenient method for determining the rate at which thickening or gelation occurred. Table 3 presents averaged values for gelation characteristics of sinters as related to Al_2O_3-to-SiO_2 ratios above and below 0.90 and to particle size of the sinter. The gelation characteristics are expressed in terms of initial viscosity, rate of change of viscosity, solids expansion, and chemically bound water in the residue. The data on rate of change in viscosity show that sinters with Al_2O_3-to-SiO_2 ratios of less than 0.90 are very prone to gelation, which is aggravated by fine-grinding. Sinters with mole ratios greater than 0.90 are almost free from gelation, and fine-grinding appears to inhibit gelation. A secondary measurement of gelation is the swelling of leach residues. Greatly swollen leach residues reduce the volume of leach liquor recovered after filtration and increase filtration and washing problems. Table 3 shows that these problems are aggravated for sinters with Al_2O_3-to-SiO_2 mole ratios less than 0.90. Chemically bound water shows the extent to which the sinter phases are hydrated during leaching.

TABLE 3. - Relation of gelation to Al_2O_3-SiO_2 mole ratios and particle sizes of sinter

Al_2O_3-to-SiO_2 mole ratio...........	<0.90	<0.90	<0.90	>0.90	>0.90	>0.90
Minus 100-mesh particles in sinter percent..	50-69	70-79	80-100	50-69	70-79	80-100
Slurry characteristics:						
Initial viscosity.............cp..	44	54	120	57	69	79
Rate of change in viscosity log of viscosity/hr..	0.0140	0.0165	0.0181	0.0081	0.0032	0.0018
Time to reach 400 cp.......hours..	68	54	29	105	238	369
Residue characteristics:						
Volume expansion in 24-hour leaching................percent..	217	228	259	169	111	111
Chemically bound H_2O........do....	6.6	10.5	10.4	12.2	11.8	14.0

Table 4 shows typical viscosity data obtained by the recording viscometer. The presence of appreciable free CaO in a sinter had a strong influence on the thickening of the leach slurry. The availability of free calcium ions through the presence of free CaO and through hydrolysis of calcium aluminates and silicates is a primary cause for processing difficulties. Free calcium ions exist in lime-soda sinter leach solutions only on a transitory basis before precipitating as another insoluble calcium compound. Uncontrolled precipitation of the calcium brings about the thickening of slurries (20) and low alumina recovery (15, 17). Directed sequestering (9, 20) or controlled precipitation (13, 15-16, 18) of the calcium ion brings about relaxation in gelation and an improvement in alumina recovery.

TABLE 4. - Viscosities of leach slurries as a function of free CaO in sinters, centipoises

Al_2O_3-to-SiO_2 mole ratio	Free CaO, percent	Time, hours						
		0	0.5	1	4	8	12	20
0.37	0.28	25	35	35	88	88	97	102
.37	2.67	35	43	61	130	210	240	275
.37	4.40	61	88	175	570	630	700	700
1.37	.19	25	25	35	43	53	53	53
1.57	.39	61	53	61	71	71	80	88

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