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# **EXTRACTION OF ALUMINA FROM CANADIAN AND AMERICAN ANORTHOSITE BY THE LIME-SODA- SINTER PROCESS**

D.H.H. Quon

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EXTRACTION OF ALUMINA FROM CANADIAN AND AMERICAN  
ANORTHOSITE BY THE LIME-SODA-SINTER PROCESS\*

by

D. H. H. Quon\*\*

- - -

SUMMARY

The lime-soda-sinter process for extracting alumina from Canadian and American anorthosites was investigated. The alumina yield was correlated with sintering conditions and reactant compositions.

Extractions of greater than 97% and 90%  $\text{Al}_2\text{O}_3$  were obtained from the Canadian and American anorthosites respectively, from sinter mixes having mole ratios of  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3 = 1.0$ , and  $\text{CaO}/\text{SiO}_2 = 2.0$  to 2.2.

The optimum condition of sintering has been established at approximately  $1275^\circ$  to  $1300^\circ\text{C}$  for a period up to 30 minutes. Extremes of high or low sintering temperature and long soak-time show a decrease in alumina extraction.

Although the technical feasibility of the process has been established, its economic viability along with that of other potential processes will be the subject of further investigations in a continuing research program.

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\* This report relates essentially to the samples as received

\*\* Research Scientist, Ceramic Section, Mineral Sciences Laboratories, CANMET, Department of Energy, Mines and Resources, Ottawa, Canada.

Extraction de l'alumine des anorthosites du Canada  
et des Etats-Unis par le procédé chaux-soude\*

par

D.H.H. Quon\*\*

RESUME

L'auteur étudie le procédé chaux-soude d'extraction de l'alumine et son application aux anorthosites du Canada et des Etats-Unis. Il établit la corrélation existant entre le rendement en alumine d'une part, et les conditions de frittage et la composition des réactifs mis en jeu, d'autre part.

Il a obtenu des rendements d'extraction de l' $\text{Al}_2\text{O}_3$  supérieurs à 97 pour cent et 90 pour cent pour des anorthosites provenant du Canada et des Etats-Unis respectivement, à partir de frites présentant les rapports molaires suivants:  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3 = 1,0$  et  $2,0 < \text{CaO}/\text{SiO}_2 < 2,2$ .

Il montre que les conditions optimales de frittage se situent approximativement entre  $1275^\circ\text{C}$  et  $1300^\circ\text{C}$  pour une durée allant jusqu'à 30 minutes. Lorsque la température de frittage est trop longue, on observe une diminution du rendement d'extraction de l'alumine.

Bien qu'on ait établi la praticabilité technique du procédé, sa rentabilité ainsi que celle d'autres techniques feront l'objet de plus amples recherches dans le cadre d'un programme de recherche continu.

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\* Ce rapport a essentiellement trait aux échantillons tels que reçus.

\*\* Chercheur scientifique, Section de la céramique, Laboratoires des sciences minérales, CANMET, ministère de l'Energie, des Mines et des Ressources, Ottawa, Canada.

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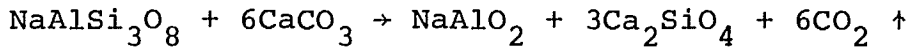
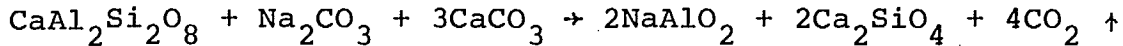
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## INTRODUCTION

The world's bauxite reserves are slowly being depleted while the demand for aluminum is increasing rapidly. Several of the major aluminum consuming and producing nations have little or no bauxite but they do have vast quantities of low-grade alumina-bearing materials and these have become a potential future source of aluminum. Processes for winning alumina from such low-grade materials as clays, anorthosite and nepheline syenite have been developed, but a satisfactory commercial process has not yet been developed<sup>(1-4)</sup>. Because anorthosite is relatively abundant in Canada and contains typically 25 to 30% alumina, a further investigation of the processes for extracting alumina from this material becomes important.

Anorthosite is a member of the gabbro family and consists of 95 to 99% of the plagioclase feldspar, labradorite, which is a solid solution of albite ( $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ ) and anorthite ( $\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ). Methods have been developed in recent years for recovering alumina from feldspar by the melt-quench technique<sup>(5)</sup>, the lime-sinter technique<sup>(6)</sup> and the lime-soda-sinter technique<sup>(7-9)</sup>. Of these three methods, the lime-soda-sinter process has been investigated most extensively. In this process, anorthosite reacts with limestone and soda ash at a high temperature to give a mixture of dicalcium silicate and sodium aluminate. The solid-state reactions that occur during sintering are represented by the following equations:





The sodium aluminate is readily soluble in caustic solution and can be removed from the sinter by leaching.

Previous investigators<sup>(7,8)</sup> have indicated that the lime-soda-anorthosite process is complicated by gelation occurring during the leaching stage. This gelation phenomenon has not yet been satisfactorily explained - the literature contains a number of contradictory hypotheses that attempt to explain this behaviour. In general, most of the investigators attribute gelation to the crystallization of unwanted phases in the sinter.

The aim of this study is to determine the effects of variables such as temperature, time of sintering, composition of reactants and the effect of various crystalline phases present in the sinter on the efficiency of the extraction process.

This research is a work element of a Project on alumina from non-bauxite sources. The objectives of this project are to develop the knowledge base required to advise government on policies related to developing alternatives for imported bauxite, and to reduce the dependence of Canadian industry on imported raw materials.

#### RAW MATERIALS

The anorthosite and limestone used for this study was supplied by the Aluminum Company of Canada Limited from the Lac St. Jean region of Quebec, and by the United States Bureau of

Mines, (hereafter referred to as Canadian or American anorthosite or limestone). The soda ash was a technical grade obtained from a commercial supplier.

#### Mineralogy of the Canadian Anorthosite

Canadian anorthosite is a dark grey, coarse-grained rock consisting of euhedral to subhedral plagioclase feldspar having a grain size varying from 2.0 to 30 mm. In a thin section, the plagioclase feldspar shows twinning of albite, albite-carlsbad and pericline types. The composition of the plagioclase is labradorite ( $An_{50-54}$ ) and some of the feldspar exhibits undulatory extinction resulting from localized straining. Accessory minerals, which constitute less than 3% of the rock, are pyroxene, magnetite and pyrite. The alteration products are hornblende and sericite.

#### Mineralogy of the American Anorthosite

The American anorthosite is light grey and consists of anhedral to subhedral grains of plagioclase feldspar measuring 5 to 20 mm. The rock consists of about 98% feldspar and the composition of the feldspar is labradorite, ( $An_{55-57}$ ), as determined from the albite twins. The accessory minerals in the anorthosite are orthopyroxene and magnetite and the alteration product is amphibole.

#### Mineralogy of Limestones

The Canadian limestone is fine grained and greyish. Microscopically, the rock is fossiliferous and consists of calcite with traces of pyrite and limonite.

The American limestone is a fine grained, light grey rock and consists of well-crystallized calcite.

### Chemical Analyses of Anorthosites and Limestones

Prior to the experimental work, the chemical compositions of both the anorthosites and limestones were determined by X-ray fluorescence and optical emission spectroscopy. The analyses of the major constituents in both the anorthosites and limestones are given in Table 1. Semi-quantitative analysis for the concentration of the trace elements in the anorthosites is given in Table 2; it can be seen that the trace-element concentrations of the two anorthosites are very similar.

### EXPERIMENTAL PROCEDURE

The experimental stages of this study proceeded through the following steps: (1) grinding of the raw materials, (2) mixing of the raw materials, (3) sintering of the mixtures, and (4) grinding and leaching of the sinters. Samples were selected at every step and were characterized mainly by X-ray diffraction and chemical analysis. Selected samples were examined by means of differential thermal and thermogravimetric analyses, infrared spectroscopy, optical and scanning electron microscopy.

#### Grinding

The rock samples as received were about 2.5 to 5 centimetres in size, and were reduced initially to about 1 to 2 centimetres using a jaw crusher. Final grinding was carried out in a micro-pulverizer using a 200-mesh screen. Only the minus 200-mesh fraction was used for preparing samples for sintering. The soda ash as received was a coarse, lumpy, granular material, which was also ground to minus

TABLE 1  
CHEMICAL ANALYSES OF RAW MATERIALS

Oxides	Weight per cent			
	Anorthosite		Limestone	
	Canadian	American	Canadian	American
Al <sub>2</sub> O <sub>3</sub>	26.88	30.35	0.87	0.72
CaO	10.49	11.65	51.73	54.92
Na <sub>2</sub> O	4.80	4.25	0.01	0.01
K <sub>2</sub> O	0.51	0.51	0.31	0.08
MgO	0.23	0.35	0.55	0.30
Fe <sub>2</sub> O <sub>3</sub>	0.64	1.68	0.46	0.09
SiO <sub>2</sub>	53.06	51.52	3.51	0.75
TiO <sub>2</sub>	0.12	0.04	0.04	0.02
Total	96.73	100.35	57.48	56.89

1  
5  
1

TABLE 2  
CONCENTRATION OF TRACE ELEMENTS IN ANORTHOSITES

Elements	Weight per cent	
	Canadian	American
Mn	0.01	0.009
Mo	0.01	0.006
Pb	0.08	0.04
Cr	0.03	0.03
Cu	0.02	0.03
Ba	n.d. < 0.01	n.d. < 0.01
P	n.d. < 0.025	n.d. < 0.025
Sb	n.d. < 0.01	n.d. < 0.01
As	n.d. < 0.01	n.d. < 0.01
W	n.d. < 0.01	n.d. < 0.01
Sn	n.d. < 0.004	n.d. < 0.004
Bi	n.d. < 0.01	n.d. < 0.01
Ga	n.d. < 0.04	n.d. < 0.04
Nb	n.d. < 0.008	n.d. < 0.008
Ta	n.d. < 0.08	n.d. < 0.08
Ge	n.d. < 0.01	n.d. < 0.01
V	0.01	n.d. < 0.002
Li	n.d. < 0.1	n.d. < 0.1
In	n.d. < 0.004	n.d. < 0.004
Zr	n.d. < 0.001	n.d. < 0.001
Ag	n.d. < 0.001	n.d. < 0.001
Zn	n.d. < 0.3	n.d. < 0.30
Ti	n.d. < 0.02	n.d. < 0.001
Ni	0.02	n.d. < 0.025
Co	n.d. < 0.01	n.d. < 0.01
Sr	0.1	n.d. < 0.01

n.d. lower than detection limit of the instrument

200 mesh and oven-dried prior to use. The lime, soda and anorthosite powders were mixed in varying proportions by weight, as shown in Table 3. The composition of the mixtures was expressed in terms of the mole ratios of  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  and  $\text{CaO}/\text{SiO}_2$ .

#### Mixing

Mixtures were prepared by dry-blending the appropriate amount of the individual constituents for two hours in a ball mill charged to 40% of the total mill volume with 1.5-centimetre diameter steel balls. Eighty-gram aliquots of the mixed samples were taken and pressed into discs 4.5 centimetres in diameter and 3.5 centimetres in height, using a pressure of 10,000 psi (69 MPa). These discs were stored in an oven at  $100^\circ\text{C}$  prior to sintering.

Spherical pellets were also prepared for testing. Pellets of 0.5 to 1.0 centimetres in diameter were obtained by spraying sufficient water into the mix and rolling the mixture into pellets. The finished pellets were dried in an oven at  $100^\circ\text{C}$  to develop sufficient green strength to withstand the subsequent handling and sintering stages. The samples were held in a platinum-lined zirconia boat during the sintering process in an attempt to avoid any contamination which could arise by reaction with the container.

#### Composition of the Reactants

The composition of the mixtures used for the sintering stage is given in Table 3. In calculating the amount of limestone and soda ash required for the appropriate  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  and  $\text{CaO}/\text{SiO}_2$  mole ratio, the  $\text{K}_2\text{O}$  and  $\text{MgO}$  mole weights were considered equivalent to  $\text{Na}_2\text{O}$  and  $\text{CaO}$  respectively. Compositions having 0.8-1.8, 0.8-2.0,

TABLE 3

## COMPOSITION OF THE MIXTURES

Source of Materials	Na <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub> Mole Ratio	CaO/SiO <sub>2</sub> Mole Ratio	Weight per cent		
			Anorthosite	Limestone	Soda Ash
CANADIAN	0.8*	1.8*	35.59	59.40	5.01
	0.8	2.0	32.70	62.70	4.60
	1.0	2.0	32.14	61.50	6.36
	1.0	2.2	29.75	64.36	5.89
	1.0	1.8	34.77	58.25	6.88
	1.0	2.4	27.66	66.86	5.48
	1.0	2.6	25.80	69.10	5.10
	1.2	2.0	31.50	60.50	8.00
	1.4	2.0	30.90	59.40	9.77
AMERICAN	0.8	1.8	38.80	54.40	6.80
	0.8	2.0	36.50	57.00	6.50
	1.0	2.0	35.76	55.74	8.50
	1.0	2.2	33.57	58.44	7.99
	1.0	1.8	38.20	52.63	9.17
	1.0	2.4	31.60	60.84	7.56
	1.0	2.6	29.87	62.99	7.14
	1.2	2.0	35.10	54.80	10.10
	1.4	2.0	34.20	53.30	12.50

\*Referred to in text as 0.8-1.8

1  
8  
1

1.0-2.0 and 1.0-2.2 for both the Canadian and American anorthosites were investigated extensively at different temperatures and times of sintering. Samples with varying ratios of  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  and  $\text{CaO}/\text{SiO}_2$  were prepared to determine the effect of changing mole ratios on the efficiency of the alumina extraction, after the preliminary investigation was completed.

### Sintering

Sintering was carried out in an electrically heated resistance furnace having silicon-carbide heating elements (Globar); typically the heating rate decreased with increasing temperature (Fig. 1). The temperatures of the samples were measured by means of a Pt-Pt:10%Rh thermocouple placed directly above the samples. The temperature during the soak period was controlled within  $\pm 0.5^\circ\text{C}$ . The furnace power was turned off on completion of the sintering period and the samples were allowed to cool to room temperature before they were removed from the furnace.

Selected samples were sintered in a gas-fired furnace to examine the effect of the heating rate on the extraction of alumina. The heating rates were  $42^\circ\text{C}/\text{minute}$  and  $72^\circ\text{C}/\text{minute}$ .

### Leaching

The sintered products were ground to minus 65 mesh and leached in 1.5N caustic-soda solution. The leaching procedure was conducted at  $70^\circ\text{C}$  for a period of 30 minutes. The sodium hydroxide solution was stirred constantly while twenty-five grams of ground sinter was added slowly to give a solid:solution ratio of 1:2. Stirring was continued throughout the leaching period and upon completion the slurry was filtered through a Buchner filter



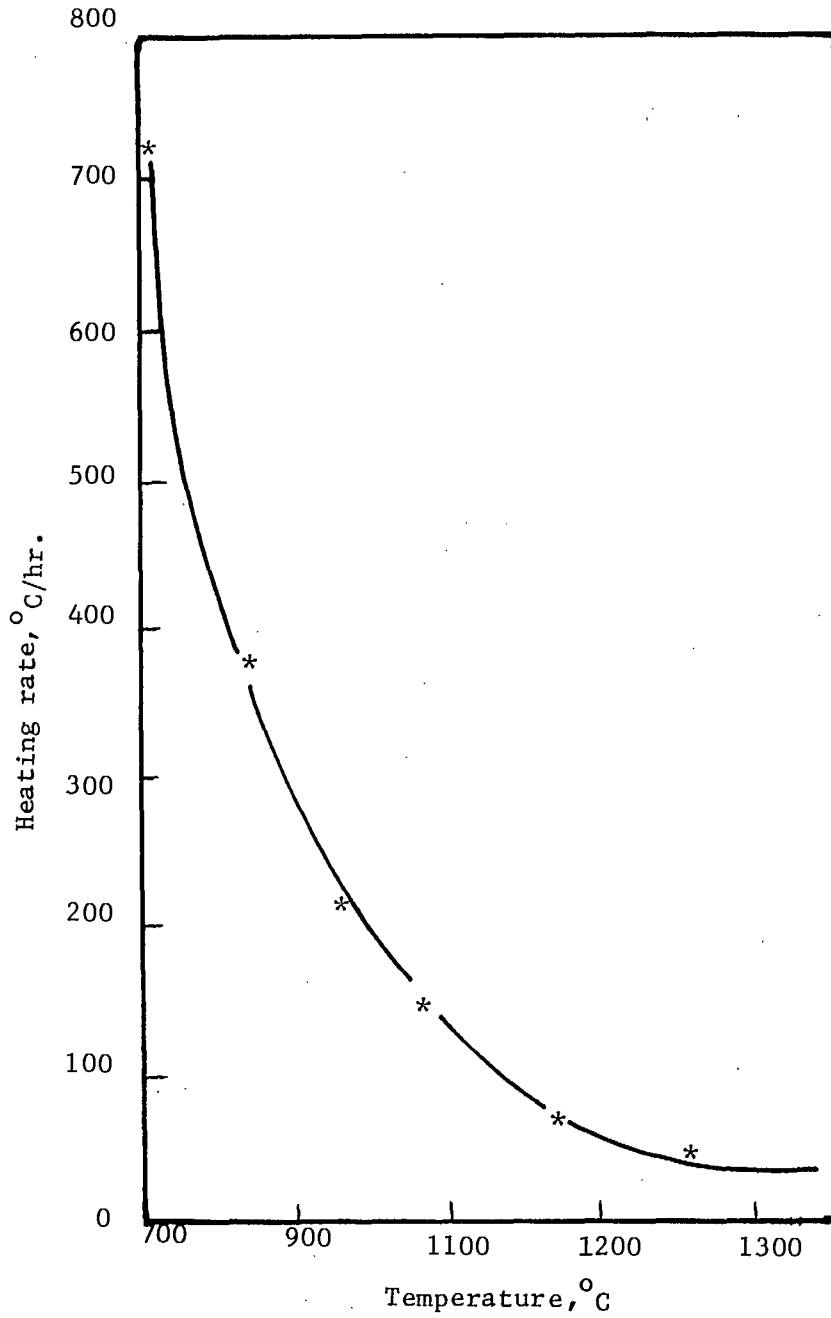


FIG. 1. Heating Rates of the Resistance Furnace at Various Temperature Ranges.

with Whatman No. 30 paper, and washed with 200 ml of hot distilled water in small portions of approximately 30 ml each.

Aluminum hydroxide precipitated from the filtrate during cooling and hence a 1:1 aqueous solution of hydrochloric acid (commercial 36.5%) was slowly added to redissolve the precipitate. The cold solution was transferred to a 500 ml volumetric flask with distilled water. Aliquots of this solution were used for the determination of the aluminum and silicon content using atomic-absorption spectroscopy.

## RESULTS AND DISCUSSION

Several important parameters are known to influence the extractability of alumina from anorthosite by the lime-soda-sinter process<sup>(7-9)</sup>. These parameters include the temperature of sintering, soak-time, composition of the mixtures (principally the  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  and  $\text{CaO}/\text{SiO}_2$  mole ratios), grain size of the raw mixtures and the leaching process. In this investigation, emphasis was placed on the variations of the reactant compositions, sintering temperature and soak-time because these were considered to be the most important parameters. Other variables such as grain size and the leaching procedure were kept constant throughout this study.

### (a) Effect of Sintering Temperature and Composition of the Reactants

#### Canadian Anorthosite

Mixtures containing mole ratios of  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  and  $\text{CaO}/\text{SiO}_2$  equal to 0.8-1.8, 0.8-2.0, 1.0-2.0, and 1.0-2.2 were examined in order to establish the optimum reactant composition and sintering

temperature. These four mixtures were sintered at temperatures of 1250, 1275, 1300 and 1350°C for a soak-period of 40 minutes in a resistivity-heated electric furnace. The percentage of alumina extracted from materials sintered under these conditions is shown in Figure 2. The results indicate that the alumina extraction was 96% of the theoretical value for the composition 1.0-2.2 but only 30% for the composition of 0.8-1.8. The optimum extraction from sinters at a soak-time of 40 minutes occurred between 1275 and 1300°C. Increasing the sintering temperature to 1350°C decreased the alumina yield for compositions 0.8-2.0 and 1.0-2.0, and this higher temperature also led to a partial melting of the composition 0.8-1.8. The cause of the lower alumina recovery was due mainly to the thickening or gelation of the leach solution. For the composition 0.8-1.8, a slight increase in alumina yield was observed with respect to an increase in sintering temperature. There was no substantial improvement in the alumina recovery for the composition 0.8-2.0 with increased sintering temperature, and this is reflected by the maximum alumina extraction being about 75%.

#### American Anorthosite

Figure 3 shows the extraction of alumina from the American anorthosite with respect to the composition and temperature for a soak-time of 40 minutes. The results indicated that the maximum alumina extraction was obtained from a composition of 1.0-2.0 sintered at 1330°C, whereas a composition of 0.8-1.8 yielded the minimum amount of alumina in all the temperature ranges investigated. The gelation or thickening of the leach solution was not observed with

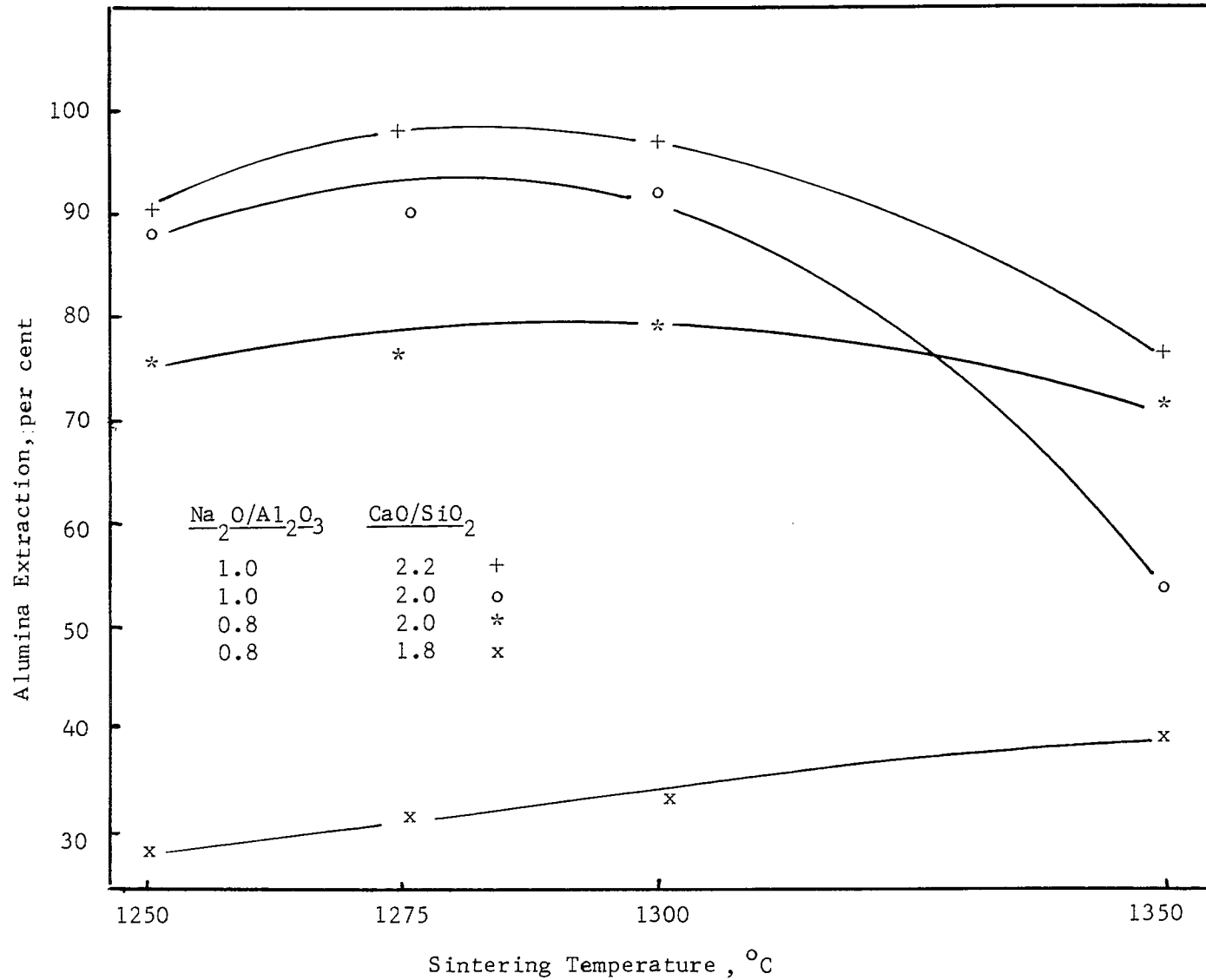


FIG. 2. Alumina Recovery as a Function of Sintering Temperature and Composition from the Canadian Anorthosite at a Soak-Time of 40 Minutes.

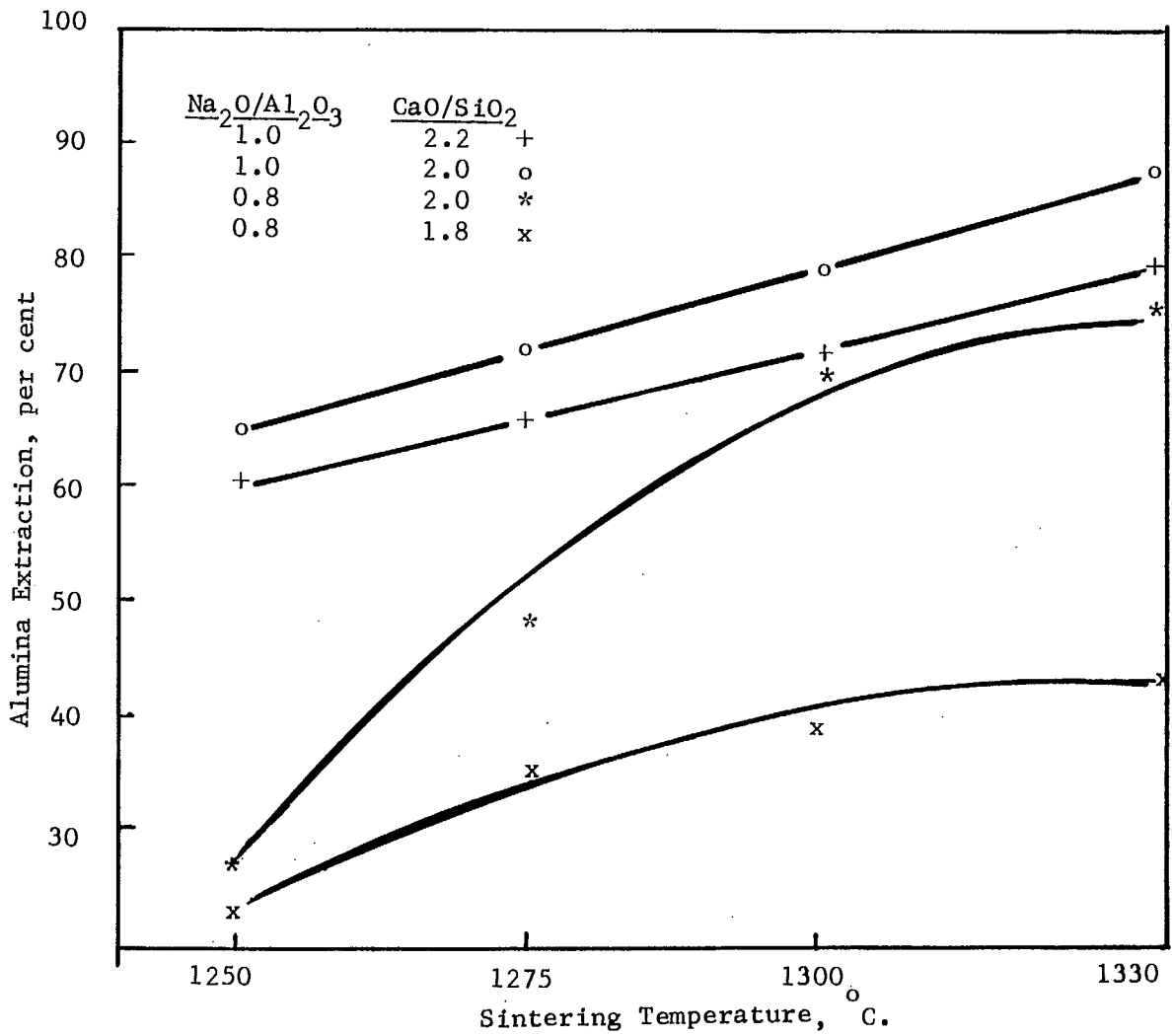


FIG. 3. Alumina Recovery as a Function of Sintering Temperature and Composition from the American Anorthosite at a Soak-Time of 40 Minutes.

the American anorthosite.

The American and Canadian anorthosites behaved in a similar manner in that low recovery of alumina occurred when the  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  mole ratio was equal to 0.8.

(b) Effect of Soak-Time on the Alumina Recovery

The Canadian and American anorthosites were sintered at 1250, 1275, and 1300°C for periods ranging from 10 to 40 minutes to establish the optimum sintering time at different temperatures in the range 1250 - 1300°C that would lead to the maximum recovery of alumina. The reactant compositions investigated were 0.8-1.8, 0.8-2.0, 1.0-2.0 and 1.0-2.2.

Canadian Anorthosite

The results showing the amount of alumina extracted from the Canadian anorthosite sintered at 1250°C for soaking periods of 10, 20, 30 and 40 minutes are given in Figure 4. These results indicate that there was very little noticeable change in the alumina yield with respect to increased soak-time; the maximum alumina yield was obtained from materials sintered for only 10 minutes. Figure 5 shows that the alumina yield from each composition at 1275°C is higher than that obtained from 1250°C sinters. Increasing the soak-time to 1275°C results in only a slight increase in the amount of recoverable alumina. For the composition 1.0-2.0 sintered at 1275°C (Figure 5) there appears to be a slight decrease in the amount of alumina extracted with increasing sintering time; for the other mixtures the maximum yield was obtained from materials sintered for 20 minutes.

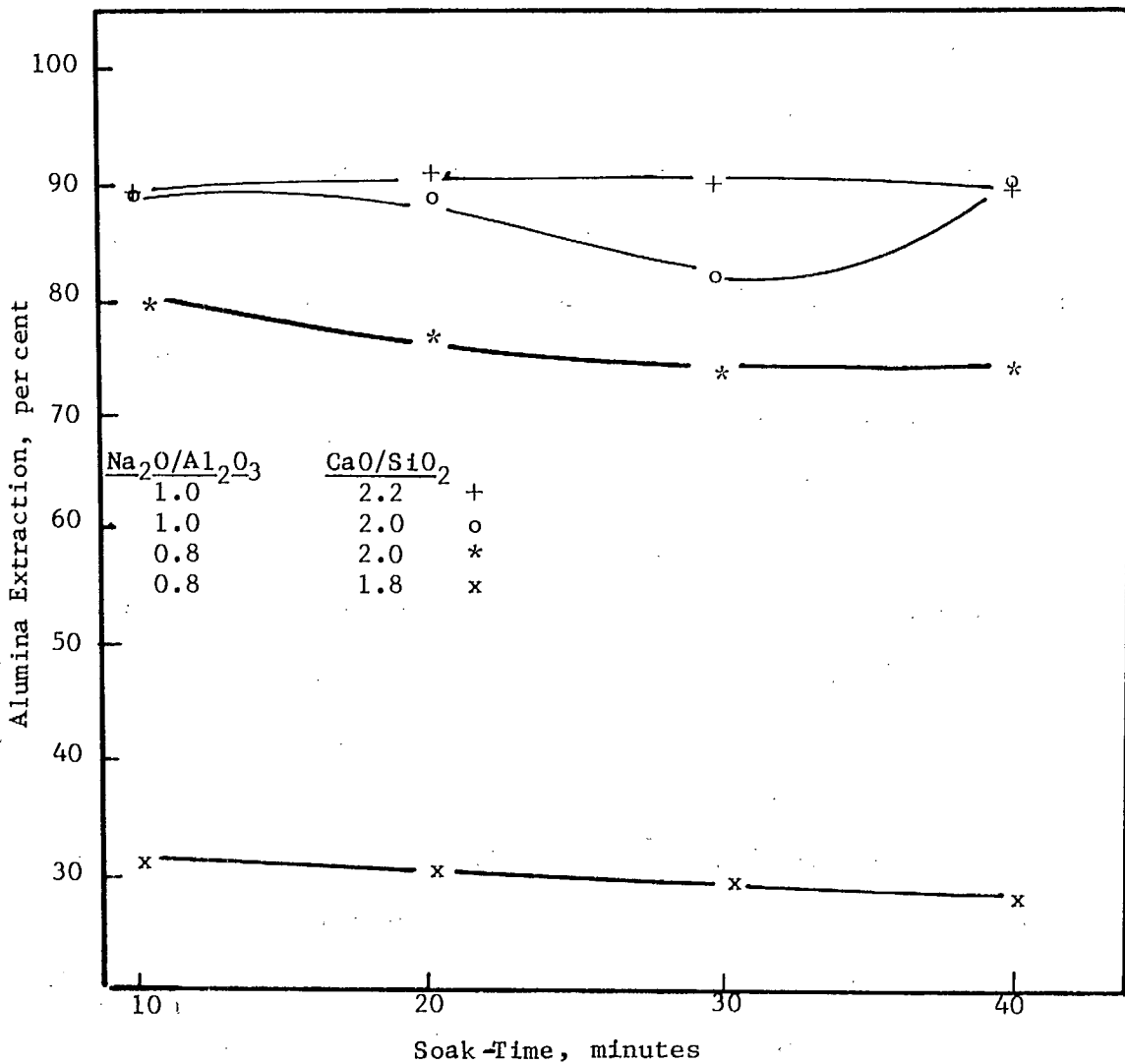


FIG. 4. Alumina Extraction from the Canadian Anorthosite as a Function of Composition and Soak-Time at 1250°C.

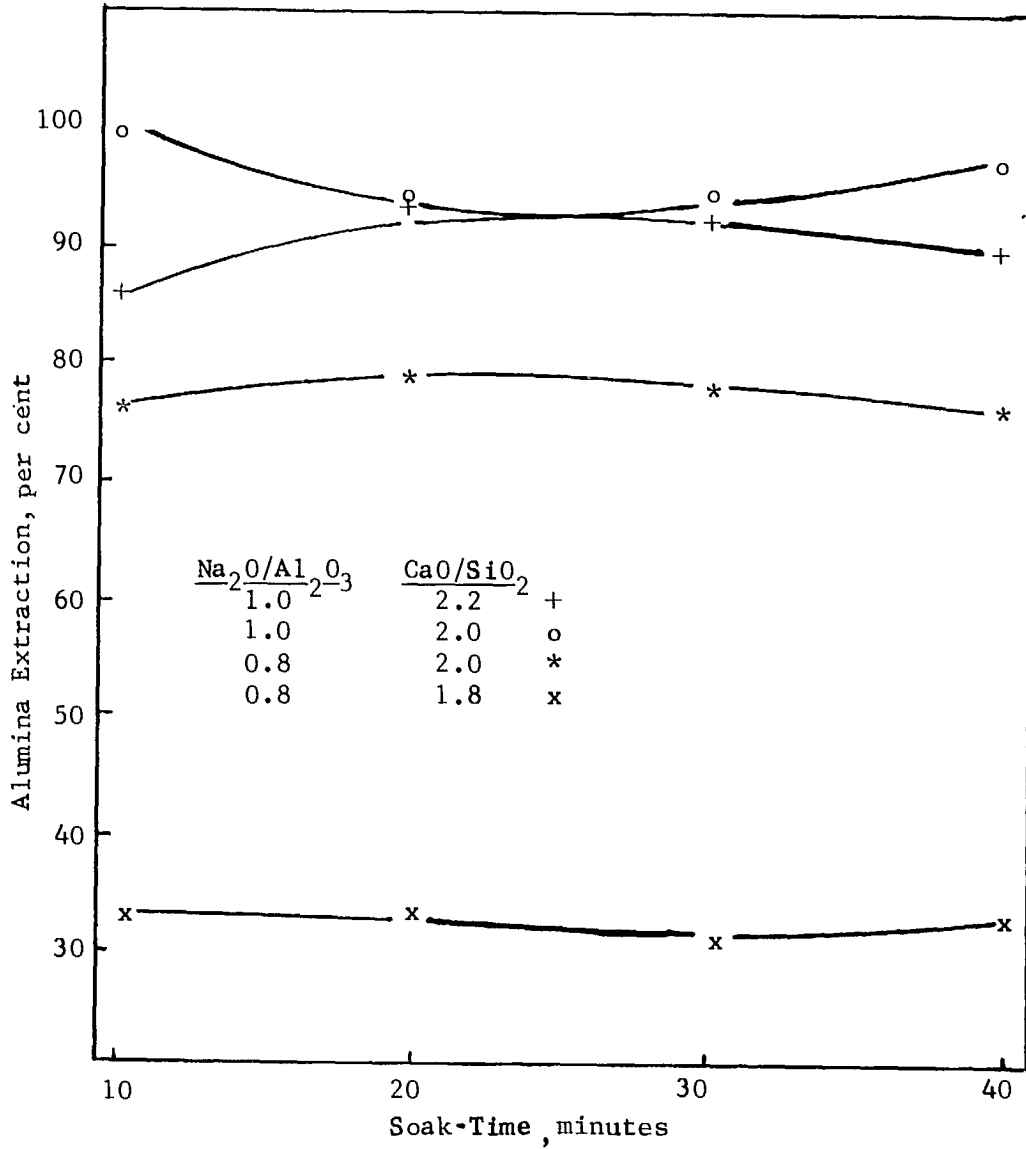


FIG. 5. Alumina Extraction from the Canadian Anorthosite as a Function of Composition and Soak-Time at 1275°C.



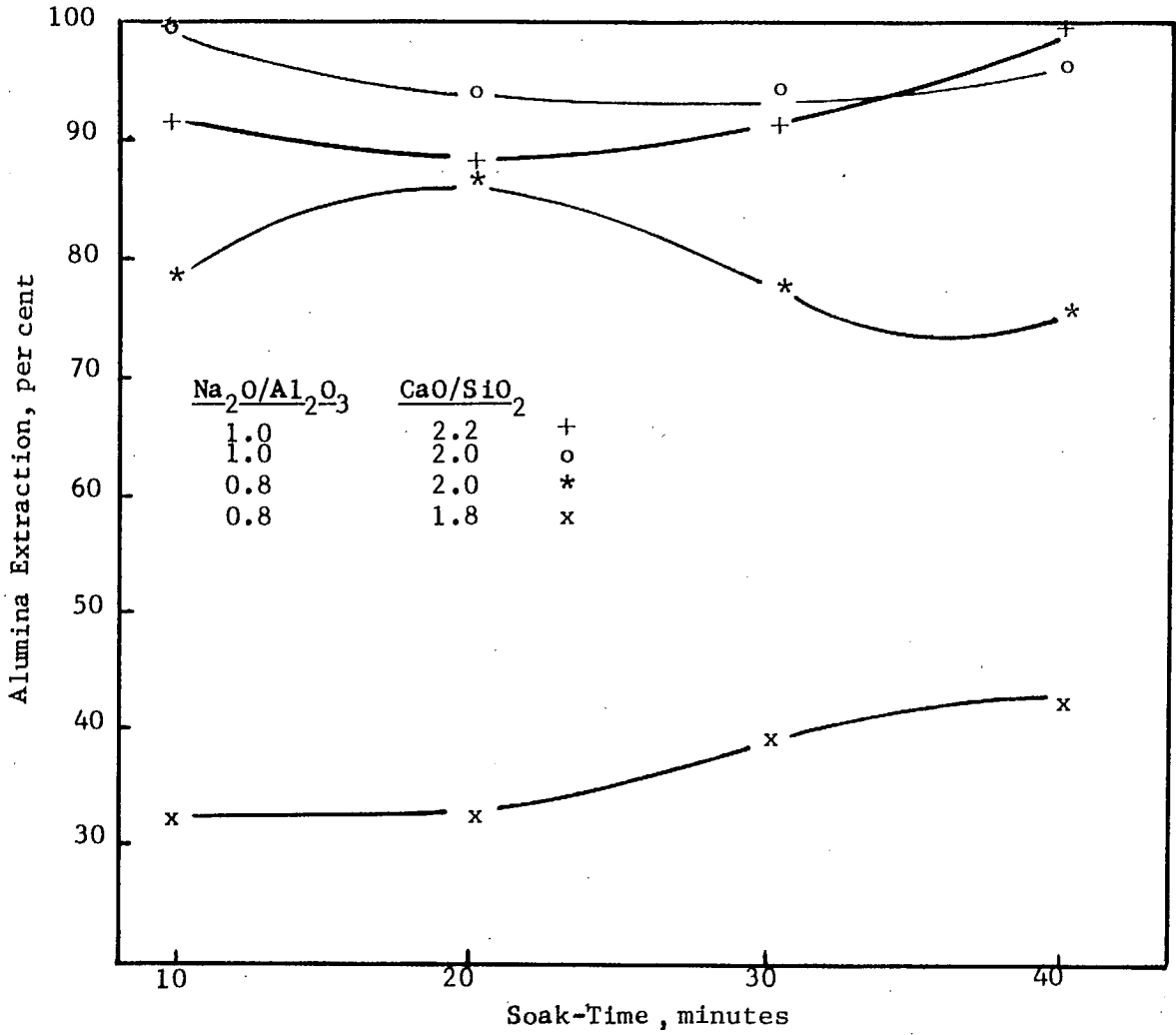


FIG. 6. Alumina Extraction from the Canadian Anorthosite as a Function of Composition and Soak-Time at 1300°C.

The optimum soak-time for compositions sintered at 1300°C, is 10 minutes for composition 1.0-2.0 (Fig. 6), whereas compositions 0.8-1.8 and 1.0-2.2 showed an increased alumina yield with increasing soak-time.

#### American Anorthosite

The effect of soak-time on the extractability of alumina from the American anorthosite was investigated under the conditions used to study the Canadian material. The results are shown in Figure 7, 8 and 9; these generally follow the pattern established for the Canadian material but optimum recoveries are significantly lower.

#### (c) Effect of Extended Soak-Time on the Alumina Yield

As previously mentioned, a reduction in the alumina yield was observed with the compositions 0.8-2.0 and 1.0-2.0 sintered at 1350°C for the Canadian material. The low yield had been attributed to the gelation or thickening of the leached solutions. A similar phenomenon was observed for these compositions when subjected to a soak-period of 40 minutes at temperatures of 1230°C and 1260°C using a slow heating rate of 50°C per hour. It appears that either a high sintering temperature or a slow heating rate were the principal causes of gel formation and the subsequent reduction in the yield of alumina. It is hypothesized that the gel was formed because of the volatilization of some constituent from the sintered reactants, and it is reasonable to assume that this constituent would be Na<sub>2</sub>O. The loss of Na<sub>2</sub>O from the sinter would allow the CaO to react with the available alumina to form calcium aluminates (CaO.Al<sub>2</sub>O<sub>3</sub> and 3CaO.Al<sub>2</sub>O<sub>3</sub>) and gehlenite

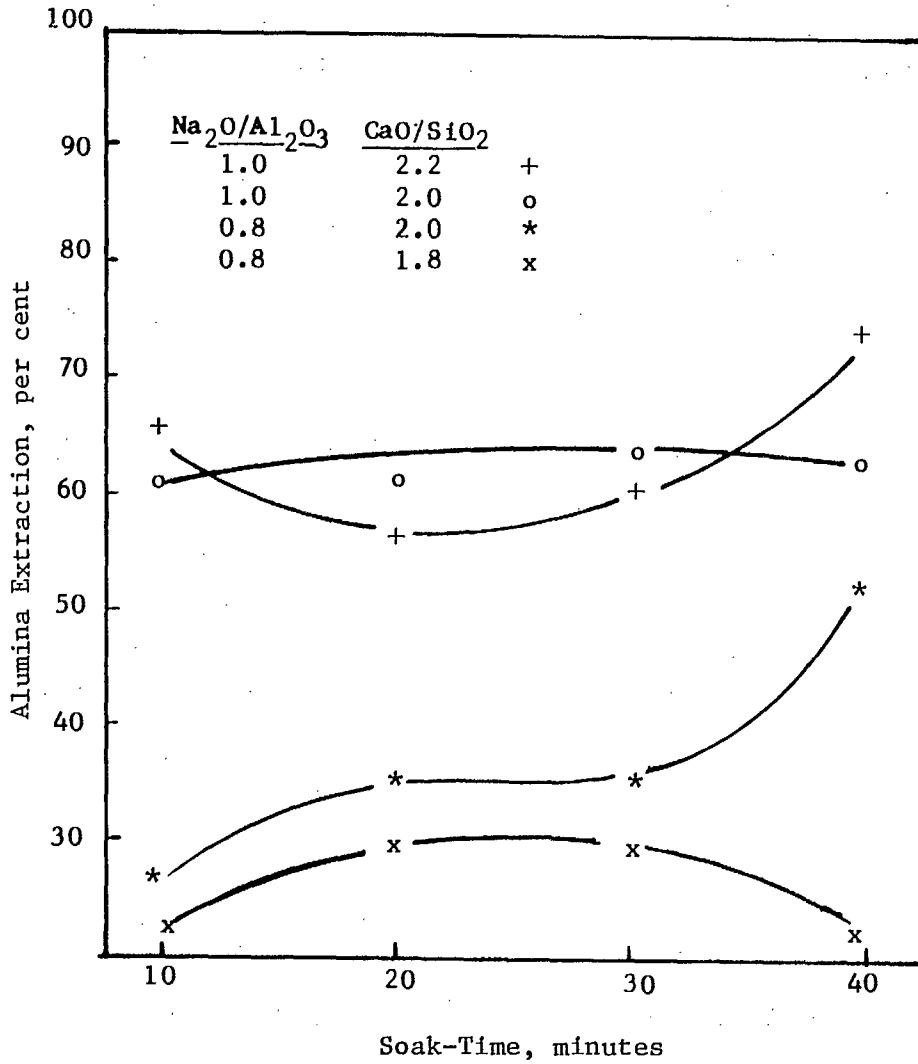


FIG. 7. Alumina Extraction from the American Anorthosite as a Function of Composition and Soak-Time at 1250°C.

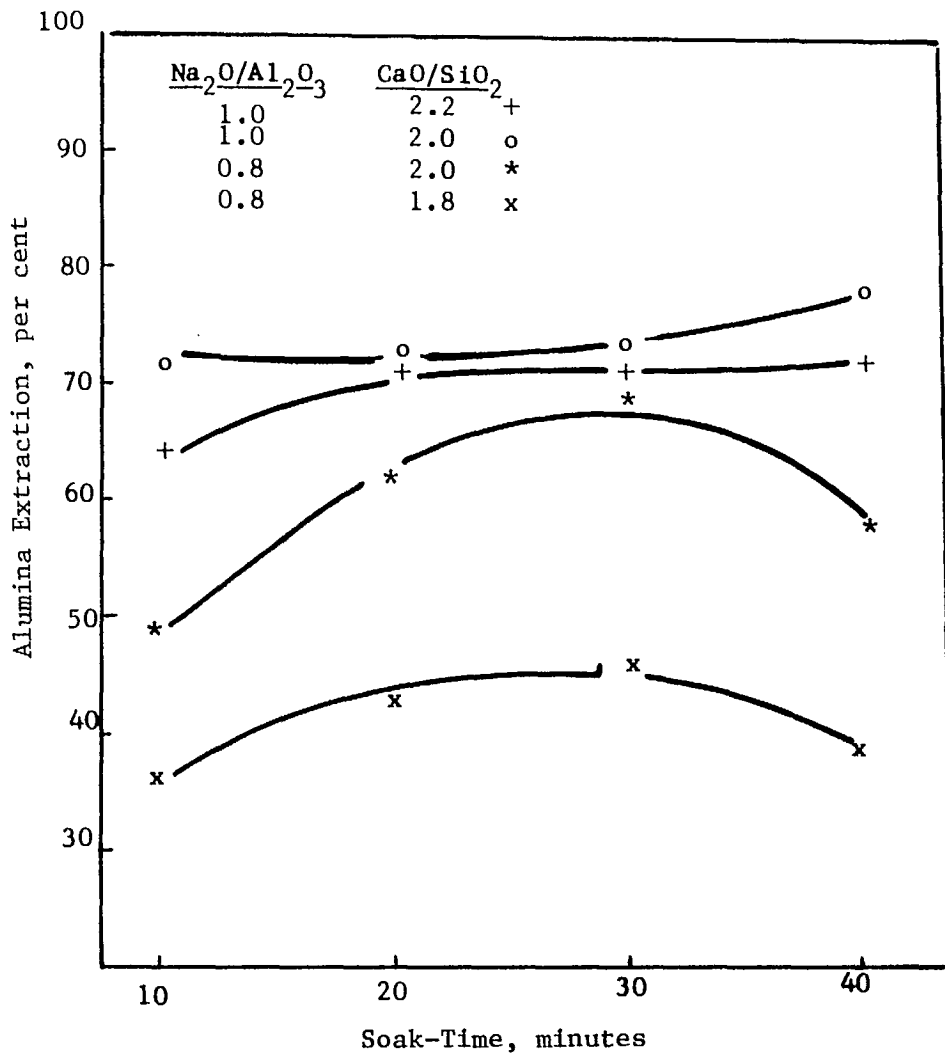


FIG. 8. Alumina Extraction from the American Anorthosite as a Function of Composition and Soak-Time at 1275°C.

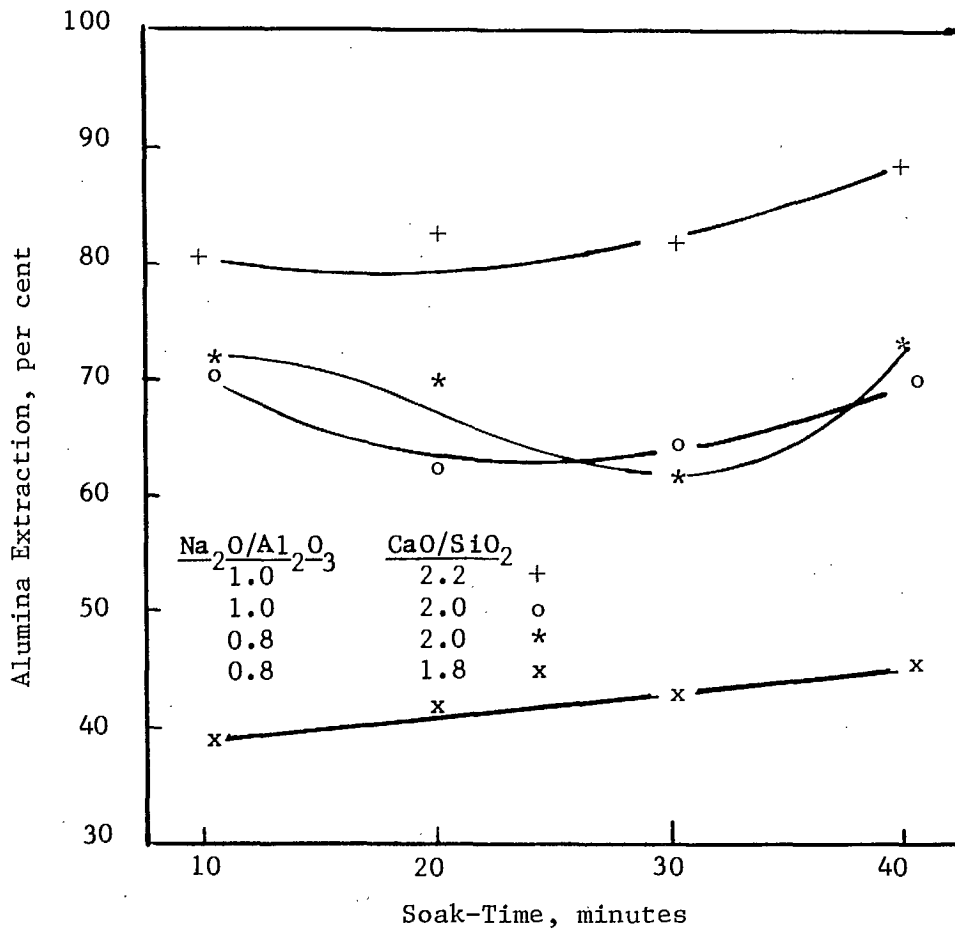


FIG. 9. Alumina Extraction from the American Anorthosite as a Function of Composition and Soak-Time at 1300°C.

( $2\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$ ). This is analogous to the situation which occurs in high-alumina cement wherein, upon hydration, a gel is formed as well as the hydrated compounds,  $\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 10\text{H}_2\text{O}$ ,  $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 6\text{H}_2\text{O}$  and  $2\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2\cdot 8\text{H}_2\text{O}$ . Gelation of the leached sinter in solution is therefore not surprising. In addition to the calcium aluminates, there are other compounds in this sinter such as di- and tricalcium silicates (which are also constituents in portland cement) which may contribute to the rapid setting of the leached sinter in solution.

Compositions 0.8-1.8, 0.8-2.0, 1.0-2.0 and 1.0-2.2 were sintered at  $1275^\circ\text{C}$  for a period of 48 hours to determine the extent of the  $\text{Na}_2\text{O}$  loss and its effect on the alumina yield. It was noticed upon completion of the sintering period that the compositions 1.0-2.0 and 1.0-2.2 differed markedly in appearance from any previously sintered samples of the same composition. These samples either partially or completely disintegrated during cooling from the sintering temperature and this phenomenon, known as 'dusting', has been previously reported by others (10,11) for lime-anorthosite sinters. The effect of dusting results from the disruptive volume-change associated with the  $\beta$  to  $\gamma$  phase-inversion of dicalcium silicate, which occurs on cooling this material below approximately  $700^\circ\text{C}$ . The occurrence of  $\gamma\text{Ca}_2\text{SiO}_4$ , however, was not reported in lime-soda-sinters where the  $\text{Ca}_2\text{SiO}_4$  is generally in the form of the  $\beta$  modification, as this form is stabilized by the presence of sodium in the structure. The lime-soda-sinter, 1.0-2.2, which dusted most severely, was subjected to X-ray diffraction, which indicated only  $\gamma\text{CaSiO}_4$ . It was

assumed that because  $\text{Na}_2\text{O}$  was lost during the long sintering process, insufficient  $\text{Na}_2\text{O}$  was present to stabilize the  $\beta$   $\text{Ca}_2\text{SiO}_4$ , which was therefore converted to the  $\gamma$  form. Thus it appears that the composition shifted from a lime-soda-sinter to closer to a lime-sinter. This shift has been observed to occur with both long sintering time and high sintering temperatures ( $1350^\circ\text{C}$ ) and may explain the absence or presence of the gel.

(d) Effect of Heating Rate on Alumina Extraction for the Canadian Anorthosite

Selected rates of heating were used to sinter the Canadian anorthosite, viz., 3.6, 42, and  $72^\circ\text{C}/\text{minute}$ , in order to establish the relationship between the heating rate and the extractability of alumina. Samples with compositions, 0.8-1.8, 0.8-2.0, 1.0-2.0 and 1.0-2.2 were heated to  $1275^\circ\text{C}$  and soaked for a period of either 10 or 30 minutes. The fast heating rates were obtained by using a gas-fired muffle furnace and the slow heating rate was achieved in an electrically-heated Globar furnace.

The effect of the different heating rates on the alumina extraction is given in Table 4, which shows that the alumina recovery is lowered as the rate of heating the initial mixture is increased. This is understandable because the faster heating schedules allow insufficient time for the optimum amount of reaction to occur. On the other hand, with a slower heating rate, a longer time is available for the solid-state reactions to reach equilibrium and/or to be completed. The reactions may have been completed prior to reaching the sintering temperature at the slowest heating rate.

TABLE 4

Effect of Heating Rate and Soak Time on the Alumina Yield from Anorthosite Sintered to 1275°C

Heating Rate, C°/min	Soak-Time, min	Percentage of Alumina Extracted			
		0.8-1.8	0.8-2.0	1.0-2.0	1.0-2.2
3.6	10	31	75	100	86
42.0	10	19	63	84	73
3.6	30	31	78	94	92
72.0	30	28	70	93	80

(e) Effect of Na<sub>2</sub>O Addition on the Alumina Extraction

Experiments were conducted to study the effect of increasing the Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> ratio on the amount of alumina extracted from both the Canadian and American anorthosites. The compositions prepared for this study are given in Table 3 and consisted of a constant mole ratio of CaO/SiO<sub>2</sub> equal to 2.0 and varying Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> mole ratios of 0.8, 1.0, 1.2 and 1.4. These mixtures were sintered at 1275°C with a soak-time of 40 minutes. The yield of alumina for the various compositions is shown in Figure 10, and indicates that the optimum recovery occurred when the mole ratio of Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> was equal to 1.0 for the Canadian anorthosite and 1.2 for the American anorthosite.

(f) Effect of CaO Addition on the Alumina Extraction

The effect of CaO additions on the amount of alumina extracted was also investigated. The compositions used for this investigation were prepared according to Table 3 and consisted of a constant mole ratio of Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> equal to 1.0, and a variable



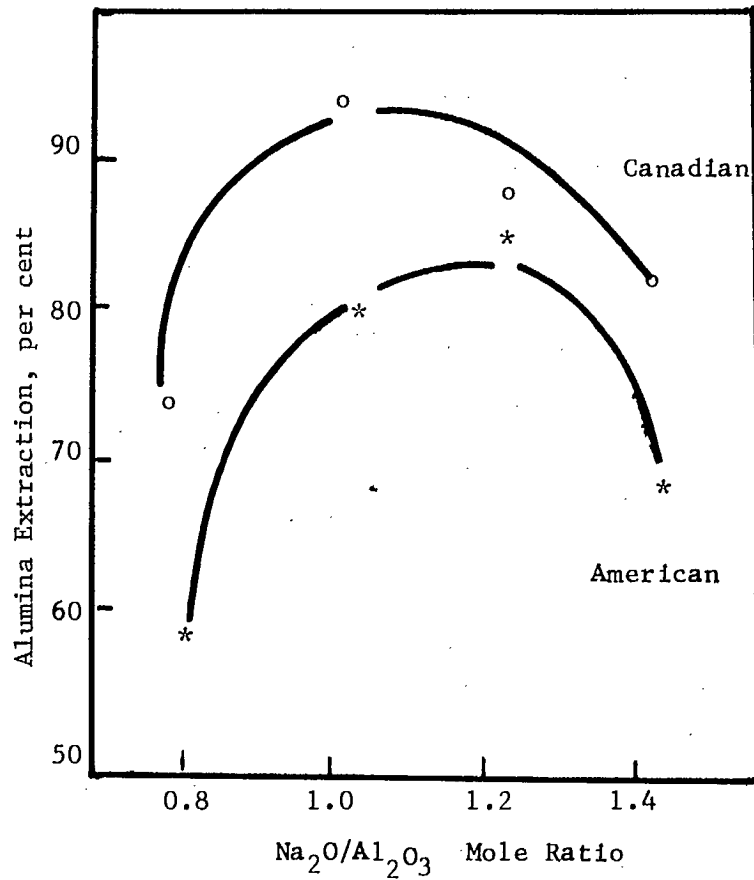


Fig. 10. Effect of Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> Mole Ratio on Alumina Recovery with Mole Ratio of CaO/SiO<sub>2</sub>=2.0.

CaO/SiO<sub>2</sub> mole ratio equal to 1.8, 2.0, 2.2 and 2.6. These mixtures were also sintered at 1275°C for a soak-period of 40 minutes. The results, (Figure 11), indicate that the maximum alumina yield occurred in a composition having a mole ratio of CaO/SiO<sub>2</sub> equal to 2, for both Canadian and American anorthosites.

(g) Silica Content of the Leach Solution

The leachates of both the Canadian and American materials were analyzed for their silica content. The results showed that the silica content of the leach solution is higher for reactant composition 1.0-2.0 and lower for the composition 1.0-2.2. Compositions 0.8-1.8 and 0.8-2.0 have a silica content intermediate between the other two compositions. The silica concentration varies slightly with increasing sintering temperatures and soak-times. A general trend appears to be that the silica content decreases as the sintering time increases for sintering carried out at 1250°C. The 1275 and 1300°C sinters also showed slight increases in silica solubility as the soak-time increased. There is a tendency for the silica content to increase at high sintering temperature (1350°C), the exception being composition 1.0-2.2 where the silica content decreases.

The variation in silica content is related to the reaction time and temperature, which allowed the constituents to react, in particular, CaO and SiO<sub>2</sub>, to form compounds such as the calcium silicates. At lower sintering temperatures and shorter soak-times, the low silica content of the composition 1.0-2.2 (less than 1.5 g of SiO<sub>2</sub> per 100 g of alumina) is in contrast to that of the composition 1.0-2.0, which has a silica content of

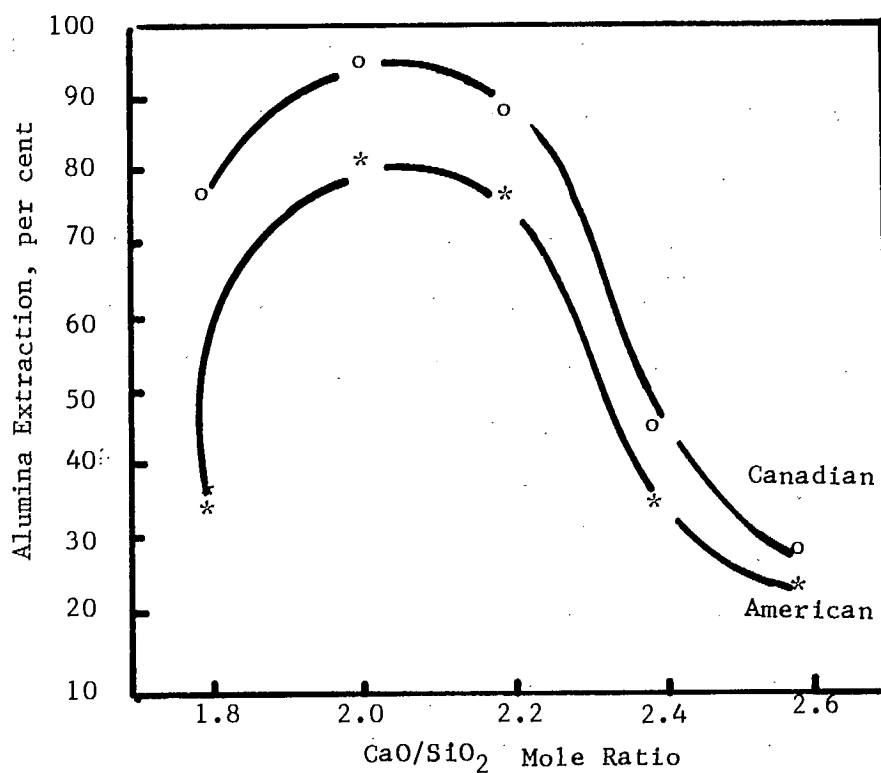


FIG. 11. Effect of CaO/SiO<sub>2</sub> Mole Ratio on Alumina Recovery with Mole Ratio Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub>=1.0.

3 g of  $\text{SiO}_2$  per 100 g of  $\text{Al}_2\text{O}_3$ . This is attributed to the high CaO content of the 1.0-2.2 sample, which allows the free silica to react more readily to form calcium silicates.

(h) Effect of Sample Preparation Techniques on the Alumina Recovery

The effect of different sample preparation techniques on the alumina extraction was investigated using the Canadian materials having a reactant composition of 1.0-2.2. Pellets and pressed discs of this composition were sintered at  $1275^\circ\text{C}$  for a period of 40 minutes. Chemical analyses indicated that the alumina recovery from the pelletized sample was only 77% in contrast to the 89% recovered from the pressed-disc sample. The higher alumina recovery of the pressed-disc sample is presumably due to the use of a much more closely compacted material that allowed the reaction to proceed readily. This finding is in agreement with the results obtained by Ampian<sup>10</sup>.

### CONCLUSIONS

The lime-soda-sinter process for extracting alumina from anorthosite has been investigated. The maximum alumina extracted from the Canadian and the American materials was 98% and 90% respectively. The results show that the maximum alumina yield is critically dependent on the reactant compositions, sintering temperature and soak-time. These studies also show that the stoichiometric composition ( $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3 = 1.0$ ,  $\text{CaO}/\text{SiO}_2 = 2.0$ ), or those with excess  $\text{Na}_2\text{O}$  and  $\text{CaO}$  above the stoichiometric requirements, increase alumina recovery. The optimum condition

of sintering has been established at 1275°C to 1300°C for a period up to 30 minutes. Extremes of high or low sintering temperature and long sintering time show a decrease in alumina yield.

Gelation is not a problem in the regular sinters, however, this phenomenon was observed in sinters subjected to high temperature sintering or a long sintering time. The principal cause of gel formation appears to relate to the loss of Na<sub>2</sub>O through volatilization.

The feasibility of extracting alumina from the anorthosite by the lime-soda sinter process has been established. This work will form a basis for economic evaluation with other processes such as lime sinter and acid extraction from melted and quenched anorthosite which are being conducted at CANMET for the Minerals Research Program.

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