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Removal of Alkalies From Portland Cement Kiln Dust

By R. D. Wilson and W. E. Anable



UNITED STATES DEPARTMENT OF THE INTERIOR

Report of Investigations 9032

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

| | | | |
|----------|---------------------|--------|-----------------------|
| atm | atmosphere | kg | kilogram |
| °C | degree Celsius | μm | micrometer |
| cm | centimeter | min | minute |
| ft | foot | pct | percent |
| h | hour | psi | pound per square inch |
| in | inch | wt pct | weight percent |
| K | kelvin | yr | year |
| kcal/mol | kilocalorie per mol | | |

REMOVAL OF ALKALIES FROM PORTLAND CEMENT KILN DUST

By R. D. Wilson¹ and W. E. Anable²

ABSTRACT

Ten to twelve million tons of dust is produced by cement kilns annually in the United States, resulting in a serious waste disposal problem. The Bureau of Mines investigated two alkali volatilization methods for converting this dust into materials for making portland cement and a useful potassium product. Portland cement kiln dust was found to be composed of finely ground cement raw materials (CaCO_3 and SiO_2) having a layer of alkali sulfate (K_2SO_4 and Na_2SO_4). In this form, it cannot be directly recycled to cement kilns because concrete is weakened by a reaction between the alkali and the aggregate. Portland cements with 28-day strengths above 5,000 psi were produced from low-alkali residues using sintering and melting methods. For nearly complete removal of alkalies, primarily potassium sulfate, from cement kiln dust, a temperature above $1,300^\circ\text{C}$ and a reducing atmosphere were required.

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INTRODUCTION

From 10 to 12 million tons of cement kiln dust accumulate annually at domestic cement plants (1-3).³ This finely divided dust is emitted from cement kilns to prevent the buildup of excessive salts in the cement product. The alkali salts in the dust are derived from the clay raw materials, which include potassium and sodium feldspars. Some fractions of the accumulated dust can be recycled to the cement kiln, but the American Society for Testing and Materials (ASTM) specifies a limit of 0.6 pct for contained alkali (4). The alkali content must be kept low to prevent concrete decrepitation, which results from a reaction between the alkali and some concrete aggregates (5-8). The bulk of the kiln dust must be disposed of at waste disposal sites. The costs for storage, transportation, and disposal, although difficult to determine, are significant for the cement industry.

In keeping with its goal to minimize the undesirable environmental impacts in mineral processing operations and to conserve materials, the Bureau of Mines conducted research to remove the alkalies from cement kiln dust to render it acceptable for recycling. In a separate study, the Bureau's Avondale Research Center characterized waste from 102 cement plants across the Nation, and found that kiln dust contained hazardous elements, but at levels below the maximum established to protect public health and safety (9).

If separated into its valuable components, waste cement kiln dust could become a significant source of potassium and a raw material for making cement. In 1982, the United States imported almost 1 million tons of potassium as K_2O equivalent. If all the potassium from cement kiln dust could be reclaimed, potassium imports could be cut by 25 pct.

Moreover, the cement content of cement kiln dust is 13 pct of the total 1982 U.S. cement production (10-11).

Cement kiln dust has been used as a potassium-rich soil supplement to replace agricultural lime. The fertilizing value of kiln dust has been satisfactorily compared with the fertilizing value of commercial products containing calcium carbonate, and those containing potassium sulfate, on test plots of onions, mustard, potatoes, spring wheat, sunflowers, barley, and clover (12-13). Cement kiln dust fertilizer was particularly effective in potato production, yielding higher quality seed potatoes and tubers higher in starch (13). Other research was successful in making an improved superphosphate fertilizer by extracting phosphate from kiln dust with HNO_3 and H_3PO_4 (14). However, in these instances, no attention has been paid to recovering the cement values.

Several means have been demonstrated to separate alkali vapors from cement kiln dust. Alkalies have been hot-water leached to produce a potassium sulfate fertilizer and an insoluble fraction that is recyclable to the cement kiln (15), but water leaching is incompatible with modern dry cement-making processes.

In an alternative process, gases from cement kilns were passed through a series of cyclones and electrostatic precipitators, and the larger relatively alkaline-free particles were separated and recycled to the kiln, leaving an alkali-rich fine-particle-size product (16). Inherent material-handling problems have prevented adoption of this scheme.

This report describes the results of research at the Albany Research Center to evaluate alkali volatilization from cement kiln dust using sintering and melting techniques. ASTM C-109 strength tests (17) were conducted on the portland cement made from the treated cement kiln dust.

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

EXPERIMENTATION

MATERIALS

Three typical cement kiln dust samples were used for the experimental work. Sample A was a cyclone product from Arkansas, sample B was a baghouse product from California, and sample C was a baghouse from Missouri. Chemical and X-ray diffraction analyses are reported in tables 1 and 2. The particle size distribution was determined by dry sieving, from which the minus 400-mesh fraction was suspended in nonane and analyzed in an X-ray sedigraph for the smaller sizes. Composite data for cement kiln dust particle sizes are reported in figure 1.

Scanning electron microscope analyses showed the dust was composed of spherical particles with a core of calcium carbonate and clay and an alkali coating. Particle agglomeration takes place in the hot zone of the cement kiln to form a heterogeneous dust mixture.

In addition to the three kiln dust samples, four carbonaceous reducing agents used in some of the sintering studies were analyzed (table 3). All of the reducing agents were ground to pass a minus 100-mesh screen.

The kiln dust samples that were used in sintering experiments were prepared by

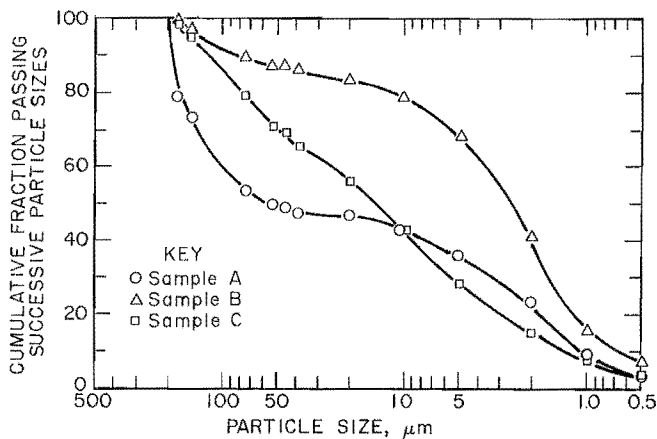


FIGURE 1.—Comparison of the particle size distribution of three cement kiln dust samples.

making 0.7-cm-diam pellets on a small 35-cm rotating disk pelletizer with either kiln dust alone or with a mixture of kiln dust and reductants. Since larger quantities of pellets were required for melting experiments, 1.3-cm pellets were prepared similarly on a 96-cm rotating disk pelletizer. No binders were needed, and all pellets were dried at 110° C prior to use.

EQUIPMENT AND PROCEDURE

Alkalies from cement kiln dust pellets were vaporized by sintering and melting techniques. Sintering was done on pelletized samples by heating in a muffle furnace from 1 to 2 h. The pellets

TABLE 1. — Analysis of cement kiln dusts, weight percent

| | Sample A | Sample B | Sample C |
|--------------------------------------|----------|----------|----------|
| Al ₂ O ₃ | 3.5 | 2.8 | 3.0 |
| CaO..... | 43.8 | 50.1 | 37.8 |
| Fe ₂ O ₃ | 2.3 | 1.9 | 1.9 |
| K ₂ O..... | 4.5 | 4.8 | 9.0 |
| MgO..... | .70 | 1.2 | 1.8 |
| Na ₂ O..... | .73 | .39 | .27 |
| SiO ₂ | 15.6 | 14.3 | 15.2 |
| Cl ⁻¹ | .05 | .13 | 2.8 |
| CO ₂ | 22.0 | 20.2 | 19.4 |
| F ⁻¹ | .46 | .53 | .36 |
| PO ₄ ⁻³ | .19 | .12 | .17 |
| SO ₄ ⁻² | 6.1 | 3.6 | 8.9 |

TABLE 2. — Mineralogical composition of three cement kiln dust samples¹

| | Sample A | Sample B | Sample C |
|-------------------------|----------|-----------|----------|
| Calcite..... | Primary. | Secondary | Primary. |
| CaO..... | Trace... | ...do.... | ND. |
| Alpha quartz. | ...do... | Trace.... | Trace. |
| CaSO ₄ | ND..... | ND..... | Do. |
| Dolomite..... | ND..... | ND..... | Do. |

ND Not detected.

¹Primary phase = 40 to 100 pct; secondary phase = 20 to 60 pct; trace = 1 to 10 pct.

TABLE 3. - Analysis of reducing agents (dry basis), weight percent

| Reductants | Volatile matter | Fixed carbon | Ash | Analyses of ash | | | |
|---------------------|-----------------|--------------|------|--------------------------------|------|------------------|------------------|
| | | | | Al ₂ O ₃ | CaO | MgO | SiO ₂ |
| Coal char..... | 14.0 | 72.5 | 13.5 | 1.48 | 1.14 | 0.30 | 3.19 |
| Coke breeze..... | 2.1 | 78.6 | 19.3 | 2.55 | .26 | .09 | 5.52 |
| Petroleum coke..... | 9.8 | 89.1 | 1.1 | .61 | .11 | .03 | .44 |
| Subbituminous coal. | 45.5 | 48.6 | 5.9 | 1.59 | .37 | .07 | 3.15 |
| Graphite fines..... | 2.5 | 97.4 | .10 | .04 | .02 | (¹) | (¹) |

¹Not determined.

were held in clay or graphite crucibles 5-cm ID by 10-cm high, partially fused at temperatures of 1,100°, 1,200°, and 1,300° C, cooled, and sampled for analysis.

A water-cooled electric arc furnace (fig. 2) was used for the melting studies. The furnace shell was 45 cm high and 33 cm in diameter, and the hearth consisted of a 10-cm layer of magnesite.

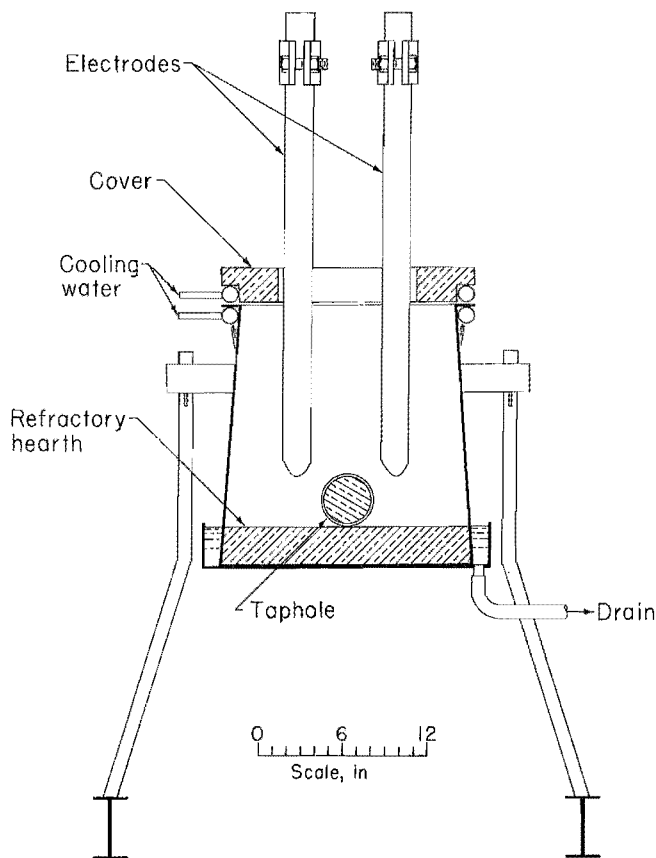


FIGURE 2.—Electric furnace for melting 25-kg samples of pelletized cement kiln dust.

The furnace was equipped with two 5-cm-diam graphite electrodes.

Kiln dust pellets were melted and held in the furnace at 1,600° to 1,700° C for 30 min. Condensed vapor samples for chemical analysis were collected on 6-in filter-paper disks placed 2 ft above the liquid bath surface. The molten furnace material was poured through the taphole into a conical cast iron crucible, and the solidified product was ground to pass a 100-mesh screen.

Strength tests were performed on mortar cubes that were made from both sintered and melted materials according to ASTM specifications C-109 (17). The sintered material was made by adding CaO and SiO₂ to as-received kiln dust to adjust the chemical composition to ASTM specifications (18). The mixture was ground, moist-rolled to obtain 0.7-cm-diam pellets, and dried. The pellets were heated in a graphite crucible at 1,500° C for 3 h and partially fused. The fused product was rapidly cooled and ground to 3.8- to 4.7- μ m (19) and cast. The cast mortar cubes were stored for 1, 3, 7, and 28 days in lime water prior to compression testing.

Melted material for strength tests also was adjusted to meet ASTM specifications by adding CaO and SiO₂. Pellets were made similarly to the sintered material pellets; i.e., heated at 1,500° C for 3 h, rapidly cooled, ground, cast, stored, and tested. The compositions of the portland cements made from sintered material (sample 1) and melted material (sample 2), as well as a typical commercial portland cement and the ASTM standard specification, are given in table 4.

TABLE 4. - Analysis of portland cements, weight percent

| Constituent | Sample 1 | Sample 2 | Standard portland cement | ASTM standard | Constituent | Sample 1 | Sample 2 | Standard portland cement | ASTM standard |
|------------------------------------|----------|----------|--------------------------|------------------|-----------------------|----------|----------|--------------------------|------------------|
| Al ₂ O ₃ ... | 4.4 | 4.0 | 5.2 | 4- 7 | Na ₂ O.... | 0.04 | 0.04 | ND | (¹) |
| CaO..... | 62.2 | 66.8 | 64.0 | 63-67 | SiO ₂ | 25.9 | 20.9 | 20.9 | 21-24 |
| Fe ₂ O ₃ ... | 3.7 | 3.7 | 2.3 | 2- 4 | C..... | .44 | .68 | ND | <1.0 |
| K ₂ O..... | .04 | .07 | ND | (¹) | S..... | 2.0 | .74 | ND | 1- 2 |
| MgO..... | 1.1 | 3.8 | 2.8 | <5.0 | LOI..... | <.01 | .01 | 1.0 | <3.0 |

LOI Loss on ignition. ND Not detected.

¹Total Na₂O + 0.625 K₂O should equal 0.6 or less.

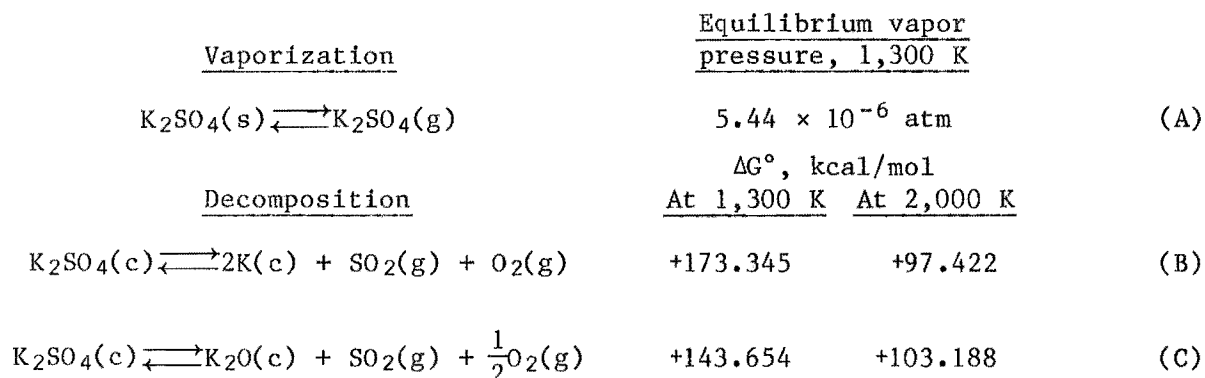
RESULTS AND DISCUSSION

OXIDATIVE AND REDUCTION VAPORIZATION MECHANISMS

Heating of cement kiln dust pellets in clay crucibles for up to 2 h at temperatures up to 1,300° C removed less than one-half of the potassium sulfate (K₂SO₄). However, in graphite crucibles, the evaporation of K₂SO₄ was significantly greater, as shown in figure 3. The probable chemical reaction mechanisms

involved help to explain the difference in the quantities of K₂SO₄ that were evaporated from each type of crucible. In the clay crucibles, the mechanism was oxidative, but in the graphite crucibles, the reaction was reductive.

In clay crucibles, the sequence of reactions for the transport of K₂SO₄ can be expressed in terms of the vaporization and decomposition reactions A, B, and C (20-24).



This discussion will focus on reactions involving potassium because it is the major alkali present (see table 5). However, thermodynamics for reactions involving sodium are similar to thermodynamics for reactions involving potassium. Data at typical sintering temperature of 1,300 K and a temperature above the fusion point (2,000 K) are presented here.

Since there are no appropriate data for the reactions depicted in reactions A and B in air, the data in an inert atmosphere

were used as a first approximation to provide a basis for the present research. Any potassium produced as suggested in reaction B would immediately oxidize and form K₂O. Reactions B and C have a surprisingly high positive Gibbs free energy (ΔG°), favoring the stability of K₂SO₄ rather than the decomposition products. Nevertheless, because escape of the gaseous products keeps the system from reaching equilibrium, the experimental results showed some alkali removal.

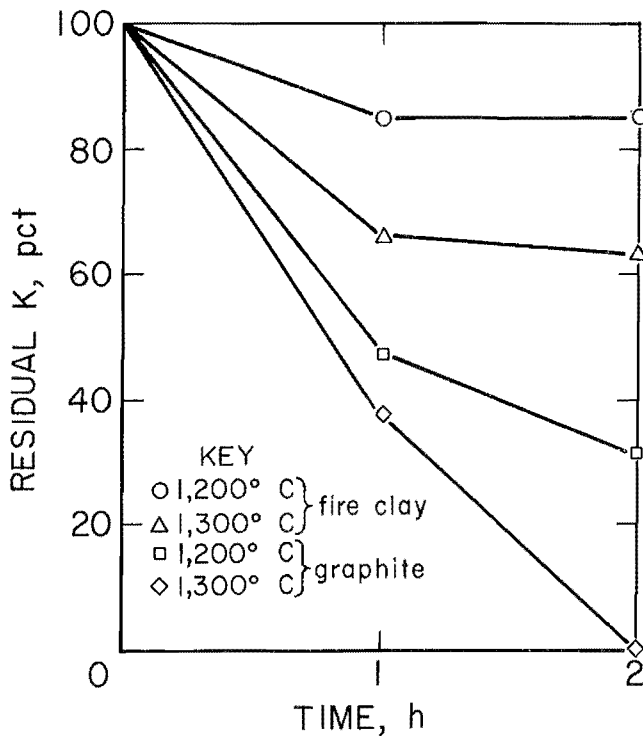


FIGURE 3.—Potassium in cement kiln dust pellets heated to 1,200° and 1,300° C as a function of time.

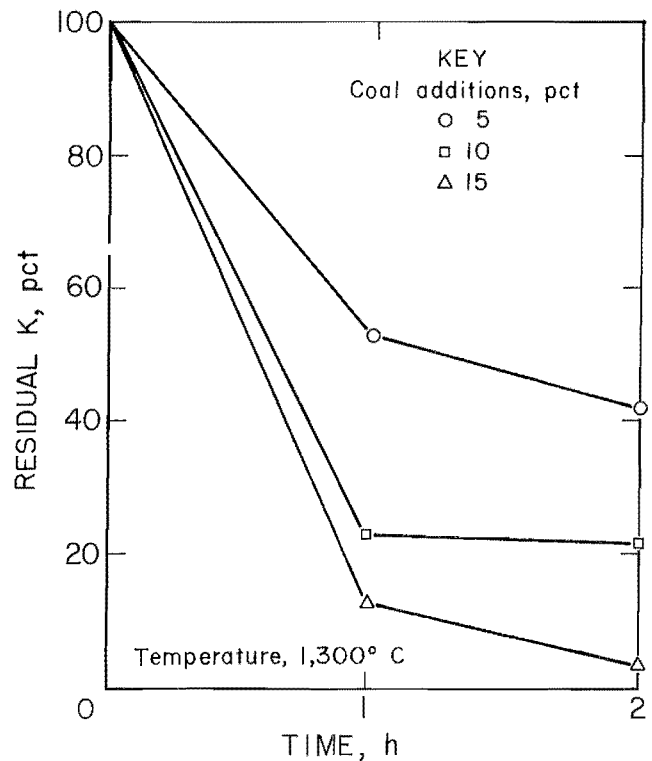


FIGURE 4.—Potassium in cement kiln dust pellets heated to 1,300° C in fireclay crucibles as a function of time and single reductant addition.

TABLE 5. - Analysis of condensed vapor from molten cement kiln dust, percent

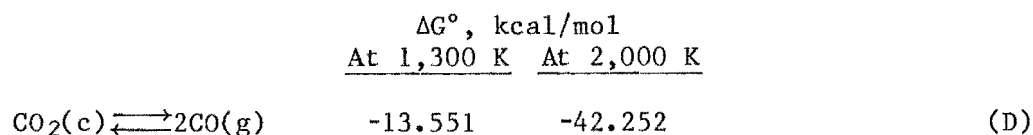
| Vapor from dust sample | Al ₂ O ₃ | C | CaO | Fe ₂ O ₃ | K | Na | MgO | SiO ₂ |
|------------------------|--------------------------------|------|------|--------------------------------|------|------|------|------------------|
| A..... | 0.64 | 0.71 | 6.4 | 1.1 | 33.0 | 4.2 | 2.3 | 3.1 |
| | .55 | .65 | 5.7 | .61 | 33.7 | 5.8 | .70 | 2.6 |
| B..... | .83 | 4.4 | 17.5 | 2.47 | 16.8 | 1.5 | 6.66 | 6.09 |
| | .74 | 1.59 | 21.0 | 2.2 | 23.0 | 1.2 | 2.8 | 3.6 |
| C..... | .36 | .25 | 3.3 | 1.75 | 37.0 | .94 | 2.35 | 9.30 |
| | .98 | .52 | 6.8 | 1.1 | 35.0 | 1.06 | 1.6 | 7.1 |

REDUCTION VAPORIZATION

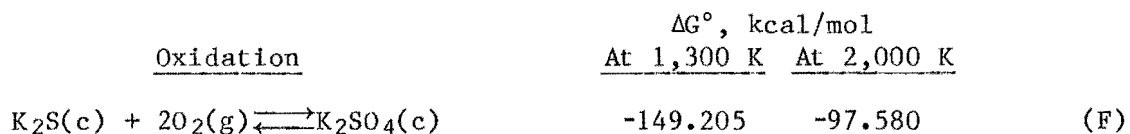
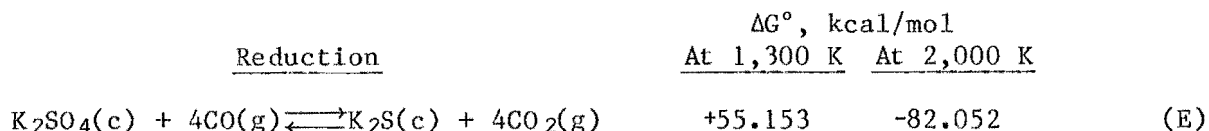
In graphite crucibles, carbon promoted up to 100 pct evaporation of alkalis from cement kiln dust when heated at 1,300° C and nearly 70 pct evaporation at 1,200° C. Although the kiln dust pellets heated in graphite crucibles were not in intimate contact with the graphite, a carbon monoxide atmosphere produced from the oxidizing crucible was responsible for the alkali evaporation. Similarly, figure 4 shows that 15 pct coal additions

that produced carbon monoxide upon heating reduced the residual potassium to nearly zero in fireclay crucibles. Figure 5 shows that 15 pct graphite fines were nearly as effective.

The reaction sequence was the same for melting experiments in the electric arc furnace, where carbon monoxide was produced from the oxidation of the graphite electrodes. As shown by reaction D, carbon monoxide readily forms by a reaction of carbon dioxide with carbon above 1,300° C.

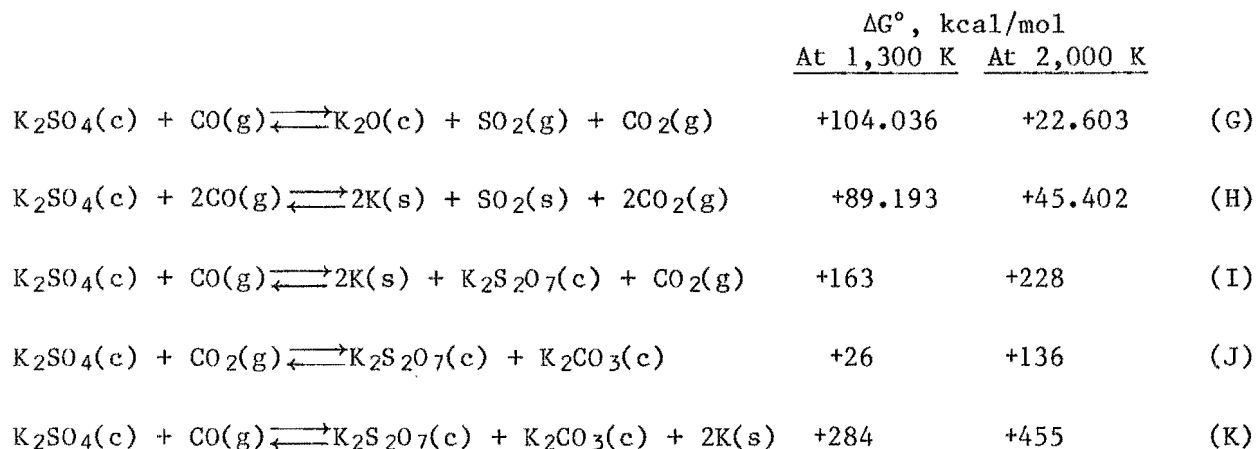


The reduction-evaporation mechanisms for alkali removal are not straightforward. However, by removal of oxygen during the formation of CO and K_2SO_4 , the indicated reactions E and F, are most likely (24).



These reactions have a negative Gibbs free energy at the temperatures used for sintering and melting cement kiln dust.

Reactions G through K are other possible reactions but these have positive free energies (24).



Furthermore, no evidence of significant quantities of SO_2 was ever detected, thus reactions G and H can be eliminated as major contributors to the overall process. The mole ratio of potassium to sulfur to carbon in the condensed vapor was found to be 2.2:1:0.1. This fact eliminates the likelihood that potassium carbonate was formed. Finally, X-ray analysis showed that the major component of condensed vapor was $\text{K}_3\text{Na}(\text{SO}_4)_2$ with no evidence of $\text{K}_2\text{S}_2\text{O}_7$. Because carbon strongly promotes the removal of alkali and because K_2SO_4 does not appear to be the vaporizing species, the most likely mechanism of potassium removal by carbonaceous materials is that potassium is transported as K_2S and reoxidized and condensed in the oxidative atmosphere outside the furnace to K_2SO_4 . Additional research, as in a kinetic study of reactions E and F, could prove the reaction mechanism.

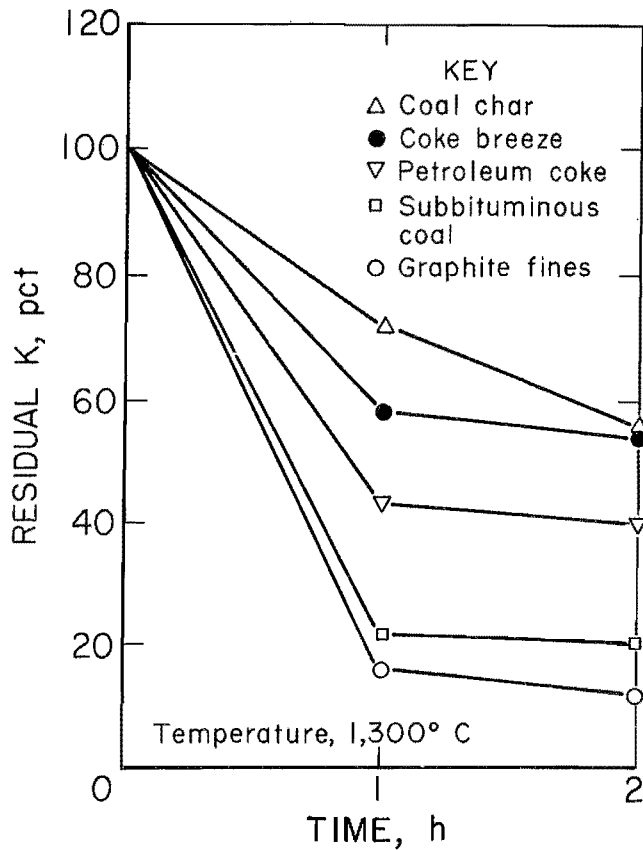


FIGURE 5.—Potassium in cement kiln dust pellets heated to 1,300° C in fireclay crucibles as a function of time and five reductant additions.

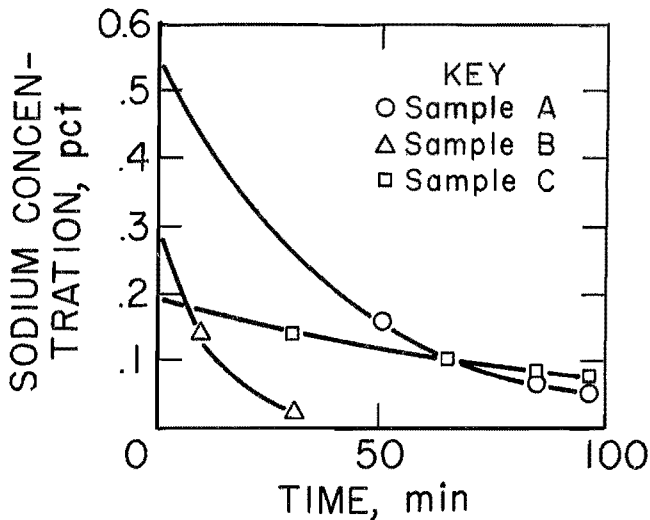


FIGURE 6.—Sodium in molten cement kiln dust as a function of time for samples A, B, and C (table 1). The constant (k) from $C = C_0 e^{-kT}$ was determined for each sample: sample A, $k = 0.024$; sample B, $k = 0.082$; sample C, $k = 0.009$.

ALKALI EVAPORATION CURVES FOR MELTED KILN DUSTS

The concentration of alkalis in molten cement kiln dust decreased over a period of time, suggesting a first-order decomposition. Figures 6 and 7 show the data obtained from samples A through C as a function of time, along with a least-squares regression curve for each set of data using the first-order rate equation.

$$C = C_0 e^{-kT}, \quad (1)$$

where C_0 is the initial alkali percent, k is a constant characteristic of the kiln dust arc furnace system derived from the data, and C is the percent alkali in the bath at time T . The differences observed in the data in figures 6 and 7 are attributable to the differences in the initial alkali concentration of cement kiln dust. A correlation coefficient, (r^2) = 0.88, was found for the application of this equation to the data. An

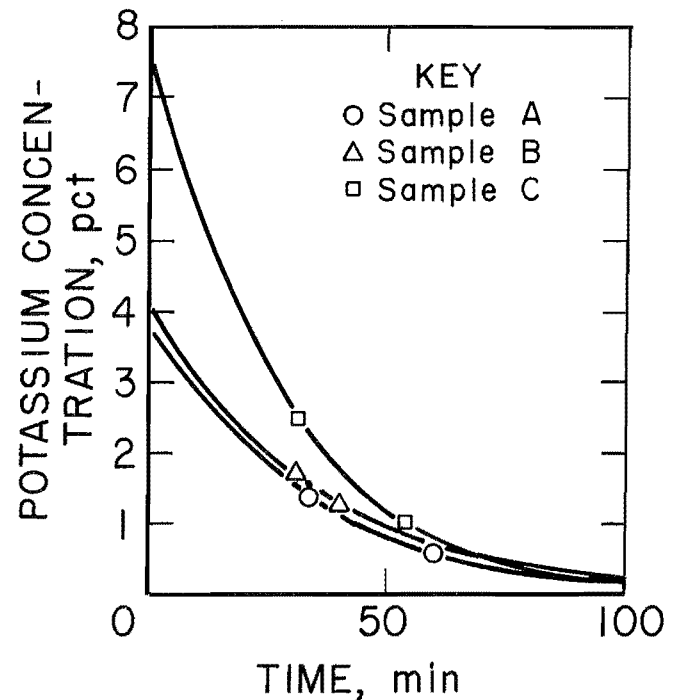


FIGURE 7.—Potassium in molten cement kiln dust as a function of time for samples A, B, and C (table 1). The constant (k) from $C = C_0 e^{-kT}$ was determined for each sample: sample A, $k = 0.063$; sample B, $k = 0.029$; sample C, $k = 0.040$.

improved equation could be made by collecting more samples, melting larger quantities of dust, and stirring the molten bath to produce a uniform composition. The derived equations match the actual rate of alkali evaporation and can be used to predict the alkali concentration in small arc furnace melts at any time up to 100 min.

STRENGTH OF PORTLAND CEMENT MADE FROM SINTERED AND MELTED KILN DUST MATERIAL

X-ray diffraction and compression tests showed that satisfactory cement could be made from cement kiln dust. ASTM standard C-150 (25) specifies that total alkali content of cement calculated as weight percent $\text{Na}_2\text{O} + 0.625 \times \text{K}_2\text{O}$ must be below 0.6 pct. All melted and sintered cement kiln dust cement had an alkali content of 0.094 pct or less, almost an order of magnitude below the ASTM standard.

Figure 8 shows that portland cement made from melted kiln (sample 1) and kiln dust sintered in carbon crucibles (sample 2) exceeded the ASTM C-109 specification for strength except at a curing time of 1 day for the melted sample. At 7 and 28 days, both kiln-dust-containing cements were stronger than a typical commercial portland cement, which was used as a

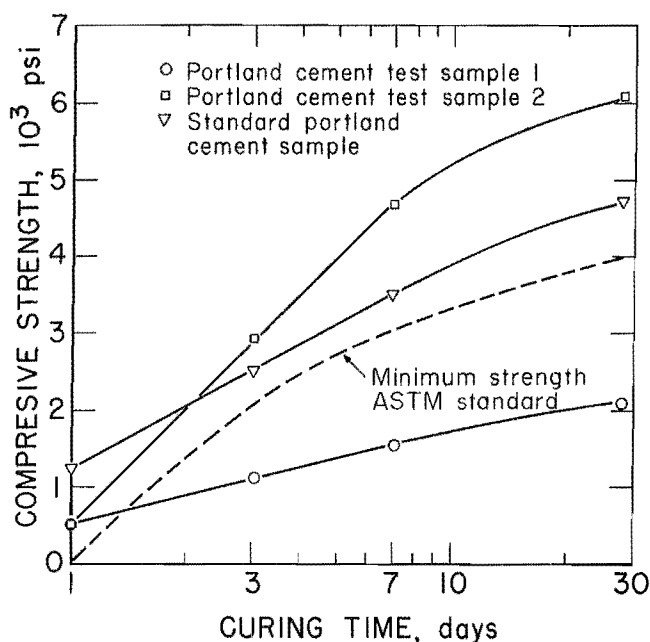


FIGURE 8.—Compressive strengths of cast cement cubes made from cement kiln dust after curing for 1, 3, 7, and 28 days. Sample 1 was made from sintered kiln dust, and sample 2 was made from melted and sintered kiln dust to which CaO and SiO₂ additions to ASTM specifications had been made.

standard. Samples B and C were adjusted to ASTM specifications by additions of CaO and SiO₂. Figure 8 also shows that the strength of a sample of sintered material that was not adjusted for CaO and SiO₂ was below the C-109 specifications.

CONCLUSIONS

Pellets of cement kiln dust high in alkali salts were sintered or melted with several reducing agents and at several temperatures. The results showed several degrees of alkali removal. Portland cements made from either sintered or melted kiln dusts exhibited strength equal to or greater than ASTM standards. Conclusions that can be made concerning the removal of alkalis from cement kiln dust are as follows:

1. The presence of carbon at a temperature of 1,300° C or more is sufficient to eliminate alkalis from cement kiln dust.
2. In a reducing atmosphere, the formation of volatile potassium and sodium is the most likely mechanism for alkali removal.
3. Vaporization of alkalis follows a simple, first-order reaction.

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