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LEACHABILITY OF ALUMINA CALCINE PRODUCED IN
THE IDAHO WASTE CALCINING FACILITY

B. E. Paige

RELEASED FOR ANNOUNCEMENT
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By

B. E. Paige

A B S T R A C T

Leaching rates for cesium, strontium, cerium, and aluminum from amorphous alumina calcine produced in the Waste Calcining Facility of the Idaho Chemical Processing Plant have been determined. Each gram of the calcine contained 26 mCi of cerium-144, 10.5 mCi of cesium-137, 7.6 mCi of strontium-90, 1.4 mCi of ruthenium-106, and lesser amounts of other fission products. Rates were obtained for cesium, strontium, and aluminum with distilled water at 25°C and 80 to 90°C and with dilute nitric acid (0.25 to 0.5M) at 25°C. In distilled water, the leaching rates for cesium and strontium were two to three orders of magnitude higher than the rates for aluminum, while the leaching rates for cerium and ruthenium were apparently the same as the leaching rate for aluminum. The leaching rate for aluminum at 80 to 90°C was 2 to 3 times higher than the rate at 25°C, but the aluminum leached was still less than 0.03 percent in 7 weeks. The amount of calcine dissolved in water is relatively low; therefore, the high leaching rates of cesium and strontium makes recovery of these fission products from WCF calcine potentially feasible.

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LEACHABILITY OF ALUMINA CALCINE PRODUCED IN THE IDAHO WASTE CALCINING FACILITY

by

B. E. Paige

I. SUMMARY

Granular alumina calcine produced in the Waste Calcining Facility (WCF) at the Idaho Chemical Processing Plant (ICPP) was leached continuously in laboratory experiments with distilled water at 25°C and 80 to 90°C and in dilute (0.25 to 0.5M) nitric acid at 25°C. In a seven-week period, greater than 95 percent of the cesium and 33 percent of the strontium were leached from the amorphous WCF calcine with distilled water at 25°C. This leachability does not represent a hazard at the National Reactor Testing Station (NRTS) where the calcine is stored in stainless steel bins under dry climatic conditions. On the other hand, the high leaching rate for cesium and strontium with distilled water does make recovery of these fission products potentially feasible because less than 0.01 percent of the aluminum is leached in a seven-week period. Based on initial rates, the leaching rates for cerium and ruthenium are estimated to be the same as the rate for aluminum from the calcine. At 80 to 90°C, the initial leaching rate for aluminum was the same as that at 25°C, but the total amount of aluminum leached was two to three times higher at the higher temperature after the first two days. Nevertheless, the amount of aluminum leached is low enough that practical recovery of cesium and strontium appears feasible at the higher temperature.

During six weeks of leaching the calcine with 0.5M HNO_3 , the calcine disintegrated and more than 99 percent of the alumina dissolved; nearly 90 percent was dissolved in the first two days. Apparently, this reagent cannot be used for selective recovery of fission products from the amorphous calcine currently being produced in the WCF. Cesium, strontium, and cerium were leached at the same rate as aluminum, showing that these fission products were uniformly distributed in the calcine.

II. INTRODUCTION

The operation of the WCF at the ICPP has demonstrated that fluidized bed calcination can be used to convert a highly radioactive aqueous waste to a granular solid for storage^(1,2,3). The bulk of the highly radioactive waste at the ICPP was produced by processing aluminum alloy fuel and, therefore, contains high concentrations of aluminum nitrate. During the operation of the WCF, approximately 500,000 gallons of this aqueous waste containing aluminum nitrate have been successfully converted to alumina calcine. The leaching rate of this calcine is of interest for two reasons: (1) to compare the relative safety of storing calcine to other forms of solid waste, and (2) to determine the feasibility of recovering fission products at a future date. In this work, the leaching rate for WCF calcine was determined by leaching at 25°C and at 80 to 90°C with distilled water and at 25°C with HNO_3 .

Calcine is considered to be a safe form for permanent storage at the NRTS in Idaho. The WCF calcine is stored in stainless steel bins in buried concrete vaults. Should any liquid enter these vaults, an alarm on the liquid-level detector in the vault sump would sound, and the liquid would then be removed by means of a steam jet^(4,5). While the hydrology of the NRTS is extremely favorable for the storage of dry solid wastes⁽⁴⁾, in other locations shallow ground water tables or high annual rainfall may create potential storage hazards. Therefore, from a safety standpoint, the leaching rate is of interest in case water does inadvertently contact the calcine during long term storage.

The leaching of fission products from calcine also has been considered as a means of either removing the fission products for future use⁽⁶⁾ or of removing heat producing fission products or long-lived fission products from the bulk of the waste solids⁽⁷⁾. The latter was studied in earlier work using calcine containing tracer amounts of fission products and 0.5M HNO_3 in a batch contact technique. Considerable work has also been done on the development of a pulsed, multiple-stage, countercurrent column for leaching fission products from granular calcine^(8,9,10). In the current work, leaching was performed in an apparatus which was designed to pass liquid through the calcine continuously and, at the same time, minimize attrition of the solids.

III. EXPERIMENTAL

1. Method

A Pyrex glass apparatus was specially designed to minimize attrition during continuous leaching of the alumina calcine. Twenty grams of granular solid produced in the WCF were placed on a coarse frit filter in the apparatus, and 500 ml of leachate were circulated through it at approximately 100 ml per minute. At the end of the leaching period, the leachate was completely removed and replaced with fresh liquid. The leachate was adjusted to a known volume, and aluminum was determined by emission spectroscopy, cesium by gamma spectrometry, and strontium and cerium by radiochemical separation techniques. The total surface area for the calcine could not be determined, therefore the leaching rate was determined as percent of material leached per day, which is not a true rate and does not include the surface area leached. Details of the apparatus and additional details of the technique are given in Appendix A.

2. Material

The calcine used in these experiments was produced in the WCF from reprocessing waste which had aged approximately 6.5 years after discharge from the reactor. A batch of calcine was collected in a remote sampler, transferred to a stainless steel container, and mixed thoroughly to assure homogeneity of the calcine for experimental testing. The calcine is primarily amorphous alumina, but it also contains some fission product oxides or nitrates, undecomposed aluminum and sodium nitrates, small quantities of water, and oxides of mercury and boron. Details of chemical composition and physical characteristics are reported elsewhere⁽²⁾ for calcine produced in the WCF. Twenty grams of this calcine were used for each leaching test. At the start of the leaching tests, each gram of calcine contained 26 mCi of cerium-144, 10.5 mCi of cesium-137, 7.6 mCi of strontium-90, 1.4 mCi of ruthenium-106, and lesser amounts of other fission products.

IV. RESULTS

1. Leaching of WCF Calcine with Distilled Water at 25°C

The leaching rates obtained for cesium and strontium in radioactive WCF calcine using distilled water at 25°C are presented in Figure 1. Detailed data are given in Appendix B. The material balances, based on the original content of the calcine, varied widely; therefore, the percent leached per unit time was calculated on the basis of the content of the leachate and the residual calcine, ie, 100% material balance, so that the comparison of the rate and total amount of these materials leached was valid. The rate for cesium was more than twice as high as the rate for strontium during the first seven weeks, but this proportion was decreasing at the end of the 14-week test period. After 100 days of leaching, the rate for cesium was about 0.02 percent per day and was still decreasing fairly rapidly as it became depleted in the calcine. The rate for strontium was about the same as the rate for cesium after 100 days, but it was leveling off. The rates for both cesium and strontium were considerably higher at all times than the leaching rates for alumina-phosphate glass prepared from the alumina calcine(3).

The leaching rate for aluminum, ie, the dissolution of the alumina, was obtained by separate experiments using non-radioactive WCF calcine because the activity level was too high in the radioactive leachates to determine aluminum by emission spectroscopic methods. The leaching rate for aluminum, reported as percent of the original material, is also shown in Figure 1. The rate for the leaching of aluminum is about 1.5×10^{-4} percent per day after 7 weeks, which is about two orders of magnitude lower than the rates for cesium and strontium and shows that the fission products were being preferentially removed.

Since the leaching rates in this work are expressed as percent leached per day, the high initial rate for the aluminum is probably due to the presence of fines and to the particle surface irregularities which contribute considerably to the total surface area of the WCF calcine. In two to five days, the fines have been dissolved and the surface area has decreased, producing a corresponding decrease in the rate of leaching. This factor of surface area also contributes to the high initial leaching rate for cesium and strontium, but the general shapes of the curves indicate that the leaching rate for these fission products may be diffusion dependent.

The initial leaching rates for cerium and ruthenium are compared to the leaching rate for the aluminum in Figure 2. The leaching rates for cerium and ruthenium were calculated as percent of the isotope in the original calcine because the fraction leached was very small and because the concentration of ruthenium was difficult to determine accurately in the residual calcine (see Appendix A). In this plot, only the first three days of the leaching test are shown because the concentrations of cerium and ruthenium in the ensuing leachates were too low for analysis in the presence of the high concentrations of cesium and strontium.

Figure 1. Relative Leachability of Aluminum, Cesium, and Strontium from WCF Calcine with Distilled Water at 25°C.

Figure 2. Relative Initial Leaching Rates for Aluminum, Cerium, and Ruthenium from WCF Calcine with Distilled Water at 25°C.

Frequent sampling was not feasible at the beginning of the non-radioactive leaching tests because of the low concentration of the aluminum in the leachate. The initial rates shown in Figure 2 indicate that both cerium and ruthenium are leached at the same rate as the aluminum from the calcine showing that there is no preferential leaching of these fission products.

The total percent of cesium and strontium leached from WCF calcine is shown in Figure 3. About 95 percent of the cesium and 33 percent of the strontium were leached in a seven-week period, while only 0.01 percent of the aluminum was leached in a similar period. The total fission product content of the leachate and residual calcine was compared to the amount of fission product in the original calcine to determine the material balance. The material balance was 86 percent for cesium-137 and 96 percent for cerium-144. The material balance for strontium was 121 percent; the values for strontium have been high throughout the work using this calcine⁽³⁾, indicating that the strontium value in the original calcine may have been low. This work shows that a large fraction of the cesium and strontium can be recovered by circulating water through the calcine at 25°C without destroying the bulk of the alumina calcine.

The residual radioactive calcine was examined at the end of the leaching test to determine if any physical change had occurred during the leaching. Although the calcine agglomerated while it was wet, it became granular and free-flowing after it was air dried. X-ray diffraction analysis showed that no crystalline species formed during either 7 or 14 weeks of leaching with water at 25°C.

2. Leaching of WCF Calcine with Distilled Water at 80 to 90°C

Leaching rates were obtained for aluminum from WCF calcine using distilled water at 80 to 90°C. The leaching rates for cesium and strontium from radioactive calcine were very high at 25°C, so that an increase in the leachability of these fission products at higher temperature would have very little practical significance. Therefore, non-radioactive calcine was used since direct measurements of the aluminum concentration are much simpler. From the aluminum data, the leaching rate of other fission products such as cerium and ruthenium can be estimated.

The apparatus described for the leaching experiments at 25°C was modified to a closed system, and a condenser was added. Gradual plugging of the coarse-frit filter during the leaching decreased the circulating rate to as low as 10 ml per minute, and an inadequate heating in the circulating system caused a temperature variation between 80 and 90°C throughout the testing period. The cumulative percent of the aluminum leached in this temperature range is compared in Figure 4 to the cumulative percent leached at 25°C. The data points represent the average value for duplicate runs. Although the initial leaching rate was similar, the rate at 80 - 90°C increased, and the total aluminum leached was two to three times higher after the first two days of testing. This is a considerably smaller effect than is produced by similar temperature increases

Figure 3. Total Percent of Cesium and Strontium Leached from WCF Calcine with Distilled Water at 25°C.

Figure 4. Effect of Temperature upon Amount of Aluminum Leached from WCF Calcine with Distilled Water.

for most dissolutions, including the dissolution of alumina-phosphate glass prepared from WCF calcine⁽¹¹⁾.

Some undissolved solids were present in the leachate. Centrifugation and subsequent analysis showed that these solids represented an insignificant amount of alumina in the leachate both at 25 and at 80 to 90°C.

3. Leaching of WCF Calcine with Dilute Nitric Acid at 25°C

Acid treatment of alumina calcine has been proposed as a possible method to recover fission products for future use⁽⁶⁾ or to remove the heat producing fission products prior to storage of the bulk waste solids⁽⁷⁾. Data shown in Figure 5 were obtained by circulating 500 ml of 0.5M HNO_3 through 20 grams of calcine at 25°C. The present work was performed with full-level radioactive product from the WCF which is primarily amorphous alumina⁽¹⁾. The calcine used by earlier workers^(7,8,9,10), which was produced in pilot plant equipment, was "spiked" with fission products and contained 60 to 70% α -alumina. During the six weeks of the current test, the WCF calcine disintegrated and more than 99 percent of the alumina dissolved. This would not have occurred with α -alumina since it is not soluble in nitric acid.

At the end of each leaching period, the leachate was removed and replaced with fresh acid. The acidity decreased to as low as 0.25M during the initial one-hour and two-hour leaching periods. During the 16-hour period which completed a total of 22 hours of testing the acidity decreased to 0.03M, but in succeeding leachates the acidity did not decrease below 0.25M. The leachate generated during this period of rapid dissolution contained some undissolved alumina. Analyses showed that the solids represented less than 0.1 percent of the total aluminum in the leachate. The decrease in dissolution of alumina caused by the low acidity in this one leaching period had little effect on the overall dissolution curve for 1000 hours.

During the first hour of leaching, the cesium leached faster than the calcine dissolved. Strontium and cerium, however, were leached at the same rate as the aluminum, as was the cesium after the initially higher rate. The high alpha alumina content of the calcine studied previously and the low ratio of acid-to-calcine account for the lesser amount of alumina dissolved in the earlier work. In view of the complete disintegration of the calcine, the partial removal or recovery of the specific fission products from amorphous calcine produced in the WCF with dilute nitric acid is not practical. This work does show that amorphous calcined alumina now being produced in the WCF can be easily dissolved in dilute nitric acid if the need should arise. However, α -alumina can be produced in the WCF by changing the boron and sodium concentrations in the aqueous feed to the calciner⁽¹⁾ or by heating the amorphous alumina now being stored^(12,13) thus drastically changing the solubility of the calcine in dilute nitric acid. This current work also verified that the fission products were uniformly distributed throughout the calcine.

Figure 5. Comparison of Leachability of Fission Products to Dissolution of WCF Calcine with Dilute Nitric Acid.

V. CONCLUSIONS

Distilled water at 25°C leached more than 95 percent of the cesium and 33 percent of the strontium from the WCF calcine in seven weeks; most of the leaching occurred during the first two or three days. Only 0.01 percent of the aluminum was leached in a similar period, and, apparently, the cerium and ruthenium were leached at the same rate as the aluminum. After 14 weeks, the cesium was essentially all removed from the calcine, and the leaching rate was decreasing rapidly; however, the leaching rate for strontium had leveled off at about 0.02 percent per day. Since other fission products and aluminum have low leaching rates, most of the cesium and some of the strontium can be recovered by circulating distilled water through the calcine at 25°C. The recovery can probably also be accomplished at higher temperatures since the leaching rate of aluminum at 80 to 90°C was not a great deal higher than at 25°C. During six weeks of leaching with dilute HNO_3 (0.25 to 0.5M) at 25°C, the WCF calcine disintegrated, and more than 99 percent of the alumina dissolved. The fission products were found to be uniformly distributed in the calcine. Dilute nitric acid will dissolve the amorphous alumina calcine now being produced in the WCF if the need arises, but it cannot be used to preferentially remove specific fission products from the calcine unless the calcine is converted to α -alumina.

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APPENDIX A

TECHNIQUE FOR LEACHING ALUMINA CALCINE

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TECHNIQUE FOR LEACHING ALUMINA CALCINE

The apparatus, which was specially designed to minimize attrition of the granular calcine, is shown in Figure A-1. Twenty grams of calcine produced in the WCF were placed on the coarse frit filter and 500 ml of leaching liquid were placed in the bottom reservoir. The air-lift circulated the leachate at about 100 ml per minute. The disengager prevents excessive motion of the leachate in the calcine. At the end of each leaching period, the leachate was removed through the side access lip, transferred to a calibrated vessel, and made to a known volume. In the radioactive leachates, the cesium was analyzed with a gamma spectrometer using a spectrum stripping technique, and strontium was separated by radiochemical techniques for analysis. Cerium and ruthenium were also determined by radiochemical separations because the quantities of these fission products, relative to cesium, were too low to determine them accurately by the spectrum stripping techniques. A portion of the residual calcine was air dried, weighed, dissolved, and analyzed for aluminum, cesium, and strontium. Ruthenium was not determined because the dissolution technique used for this highly radioactive residue permitted loss of the ruthenium during dissolution. The fission product content of the damp residual calcine was converted to a dry basis by using the aluminum concentration.

All leaching studies using radioactive calcine were performed remotely in the 34-inch wide cave shown in Figure A-2. The cave easily accommodated four separate apparatus. The installation was economical to install and no difficulty was encountered during the operation of several tests, each of which were of seven weeks duration. In studies using non-radioactive calcine, the experiments were performed on the bench top. In experiments at 25°C, where aluminum concentrations were very low, the leachate was concentrated by evaporation prior to analysis by emission spectroscopy.

CONTINUOUS LEACHING APPARATUS

CPP-S-3356

Figure A-1. Apparatus for Leaching of WCF Calcine at 25°C.

Figure A-2. Remote Cell for Leaching WCF Calcine.

APPENDIX B

LEACHING DATA FOR WCF CALCINE

APPENDIX B

LEACHING DATA FOR WCF CALCINE

The data obtained by leaching WCF calcine at 25°C with distilled water is presented in Table B-I. The discussion of the data is included in the text.

TABLE B-I

FRACTION OF COMPONENT LEACHED PER HOUR FROM WCF CALCINE AT 25°C WITH DISTILLED WATER

Time From Start of Test	Aluminum		Cesium				Strontium				Cerium	Ruthenium
	Experiment Number		Experiment Number				Experiment Number				Experiment Number	Experiment Number
	5	6	1	2	3	4	1	2	3	4	1	2
1	--	--	1.07×10^{-1}	1.06×10^{-1}	1.08×10^{-1}	1.06×10^{-1}	6.4×10^{-3}	6.4×10^{-3}	6.5×10^{-3}	6.4×10^{-3}	1.1×10^{-5}	3.8×10^{-5}
2	2.2×10^{-1}	6.7×10^{-4}	2.1×10^{-2}	2.2×10^{-2}	2.4×10^{-2}	2.2×10^{-2}	3.2×10^{-3}	3.1×10^{-3}	3.5×10^{-3}	3.0×10^{-3}	5×10^{-6}	4×10^{-6}
4	--	--	9.6×10^{-3}	9.9×10^{-3}	1.04×10^{-2}	1.09×10^{-2}	2.1×10^{-3}	2.1×10^{-3}	2.1×10^{-3}	2.0×10^{-3}	5×10^{-6}	3×10^{-6}
24	5.0×10^{-5}	--	1.5×10^{-2}	1.5×10^{-2}	1.7×10^{-2}	1.7×10^{-2}	2.1×10^{-3}	2.1×10^{-3}	2.6×10^{-3}	2.5×10^{-3}	5×10^{-8}	$< 10^{-6}$
46	--	--	1.2×10^{-2}	1.3×10^{-2}	1.2×10^{-2}	1.2×10^{-2}	5.2×10^{-3}	5.4×10^{-3}	5.1×10^{-3}	5.1×10^{-3}	$< 10^{-8}$	--
70	--	6.3×10^{-6}	4.3×10^{-3}	4.3×10^{-3}	3.2×10^{-3}	3.2×10^{-3}	2.8×10^{-3}	2.7×10^{-3}	2.1×10^{-3}	2.4×10^{-3}	--	--
142	--	7.1×10^{-6}	6.6×10^{-4}	6.8×10^{-4}	7.9×10^{-4}	9.7×10^{-4}	4.5×10^{-4}	5.4×10^{-4}	3.4×10^{-4}	3.1×10^{-4}	--	--
166	9.2×10^{-6}	--	9.0×10^{-4}	7.9×10^{-4}	6.7×10^{-4}	6.0×10^{-4}	3.5×10^{-4}	3.5×10^{-4}	2.6×10^{-4}	2.7×10^{-4}	--	--
232	3.8×10^{-6}	2.6×10^{-6}	2.2×10^{-4}	2.1×10^{-4}	1.9×10^{-4}	2.0×10^{-4}	1.7×10^{-4}	1.5×10^{-4}	1.4×10^{-4}	1.4×10^{-4}	--	--
328	1.1×10^{-5}	5.8×10^{-6}	1.16×10^{-4}	1.10×10^{-4}	1.02×10^{-4}	1.08×10^{-4}	1.2×10^{-4}	1.2×10^{-4}	1.2×10^{-4}	1.2×10^{-4}	--	--
436	2.0×10^{-5}	7.1×10^{-6}	6.7×10^{-5}	6.6×10^{-5}	6.4×10^{-5}	6.7×10^{-5}	8.1×10^{-5}	7.1×10^{-5}	6.2×10^{-5}	5.5×10^{-5}	--	--
664	9.6×10^{-6}	4.0×10^{-6}	4.3×10^{-5}	4.3×10^{-5}	3.9×10^{-5}	4.4×10^{-5}	2.9×10^{-5}	2.5×10^{-5}	2.4×10^{-5}	2.5×10^{-5}	--	--
832	5.0×10^{-6}	3.2×10^{-6}	2.7×10^{-5}	2.7×10^{-5}	2.6×10^{-5}	2.3×10^{-5}	1.8×10^{-5}	1.7×10^{-5}	1.7×10^{-5}	1.7×10^{-5}	--	--
1168	--	3.4×10^{-6}	2.1×10^{-5}	1.9×10^{-5}	1.5×10^{-5}	1.7×10^{-5}	1.2×10^{-5}	1.2×10^{-5}	1.1×10^{-5}	1.2×10^{-5}	--	--
2008	--	--	--	--	1.2×10^{-5}	1.2×10^{-5}	--	--	9.9×10^{-6}	1.1×10^{-5}	--	--
2344	--	--	--	--	8.8×10^{-6}	7.8×10^{-6}	--	--	1.1×10^{-5}	1.1×10^{-5}	--	--