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Countercurrent Washing of Pittsburgh No. 8 Coal after Leaching with Molten Mixtures of Sodium and Potassium Hydroxides

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Abstract

Molten caustic leaching is an advanced chemical coal-cleaning process which results in the removal of over 90% of the sulfur and ash from coal. One of the steps in this process is the water washing of caustic-leached coals to remove unreacted caustic and impurities released by reactions with the molten caustic. A countercurrent procedure, designed for efficient washing with minimal water consumption, has been evaluated in the present work. A Pittsburgh No. 8 coal was leached with a one-to-one mixture of molten sodium and potassium hydroxides, and the resulting coal–caustic cake was washed using this countercurrent procedure. The countercurrent washing did result in recovery of caustic at predicted concentrations, and a relatively ash-free and sulfur-free coal was the final product. However, significant problems occurred during the countercurrent washing, all of which could be linked with the formation of a massive precipitate of carbonates from the alkaline process streams. The mass of the precipitate retained fluids and thus led to far lower than predicted recoveries of caustic solutions. The precipitate also caused a significant decrease in filtration rates.

INTRODUCTION

Molten caustic leaching (MCL), being developed at TRW in California, is an advanced chemical coal-cleaning process which removes more than 95% of the ash-forming minerals and 90% of the sulfur from coal (*1–4*). The basic steps in the MCL process are: 1) leaching of coal with molten caustic, 2) washing of the resulting coal–caustic mixture with water to remove most of the soluble salts formed during leaching, 3) washing of the water-washed coal cake with dilute acid to remove alkali metals ionically bound to the coal and to remove acid-soluble compounds such as iron hydroxides, and 4) finally water washing to remove residual acid.

The initial water-washing step results in recovery of unreacted caustic, as well as removal of acid-consuming impurities from the coal. Effective water washing is essential both for the separation and recovery of unspent caustic for use in processing additional coal samples and for the removal of alkaline species from coal which would otherwise consume acid in the subsequent acid-washing step.

TRW has proposed a countercurrent water-washing procedure which is intended to result in use of a minimal amount of water, to produce a relatively clean coal as feed to the acid-washing step, and to produce a concentrated caustic solution for reprocessing to anhydrous caustic (*5*). In this procedure the coal–caustic cake is initially washed with a 30% caustic solution to produce a 50% caustic solution which is then reprocessed. The coal cake is then washed with a more dilute caustic solution to produce a 30% caustic solution for use in the initial washing of the next coal sample. This process is repeated on the coal cake with progressively more dilute caustic solutions and finally with pure water. The designed net inputs to this procedure are water and coal–caustic mixtures. The designed outputs are 50% caustic solutions and coal samples essentially free of all alkaline impurities.

In previous work at the Ames Laboratory, laboratory-scale simulations of the TRW-developed countercurrent washing procedure were applied to samples of Kentucky No. 11 and Illinois No. 6 coals after they had been leached with molten caustic consisting only of sodium hydroxide (*6*). That work revealed some potentially serious problems with the procedure, all of which could be linked to the presence of high concentrations of sodium carbonate in the coal–caustic feed to the washing procedure. It was found that sodium carbonate, entering the process with the coal–caustic, precipitated and built up in the intermediate stages of countercurrent washing. This buildup reduced filtration rates and separation efficiencies, and the carbonate precipitate retained fluids which reduced the amount of concentrated caustic solution recovered. Eventually, the buildup of carbonate reached the final washing stages and exited with the cleaned coal, and this resulted in consumption of excessive amounts of acid in the subsequent acid-washing steps. The buildup of carbonate occurred because sodium carbonate has a very limited solubility in concentrated caustic (*7*). It thus precipitated on coal rather than exiting the process stream with the concentrated caustic solution, and the precipitate moved with the coal toward the final stages of the countercurrent washing process. In those final stages of the countercurrent procedure coal was washed with pure water, and the precipitated carbonate was dissolved from the coal and moved with the solutions back toward the initial stages of the countercurrent washing process. The net result of this back-and-forth movement was a sodium carbonate buildup in intermediate stages of countercurrent washing until it reached levels in the final stages exceeding its solubility in water. At that point a state was approached in which similar amounts of carbonate could exit the process with the washed coal as entered in coal–caustic cakes, but by this time the buildup of carbonate was so massive that very little caustic solution was recovered and filtration rates were unacceptably low.

Two potential solutions to the carbonate buildup problem have been investigated previously at the Ames Laboratory. One solution was to suppress the amount of carbonate formed during the molten caustic leaching step (*8*, *9*). Carbonate is a by-product of the leaching of coal with molten caustic. It has been found that the amount of this undesirable by-product can be reduced by about 70% by modifications in the leaching step. For example, a combination of pretreating coal before leaching by devolatilization at about 420°C and rigorous exclusion of air from the leaching reactor led to reductions in the amount of caustic converted to carbonate from about 35% to about 10%. A second potential solution to the carbonate buildup problem was to withdraw sidestreams of carbonate-containing solutions produced during countercurrent washing, remove the carbonate from these streams, and then reintroduce the carbonate-free streams back into the countercurrent washing procedure. It has been found that carbonate can be efficiently removed from NaOH-containing countercurrent washing streams by precipitation with lime (CaO) to form calcium carbonate which is then removed by filtration (*10*).

In the present work a third potential solution to the carbonate buildup problem was investigated. Potassium carbonate is more soluble than sodium carbonate in potassium hydroxide–sodium hydroxide mixtures (*11*), and there are also potential advantages to leaching coal with sodium hydroxide–potassium hydroxide mixtures rather than with sodium hydroxide alone (*1*). Thus, in the present work, coal was leached with a 1:1 mixture of molten sodium and potassium hydroxides, and the resultiing coal–caustic mixture was washed using a laboratory simulation of the TRW-designed countercurrent procedure (Fig. 1).

EXPERIMENTAL

Molten Caustic Leaching

Seven 100-g samples of Pittsburgh No. 8 coal were each leached with 200 g each of a 1:1 mixture of sodium hydroxide and potassium hydroxide at 390°C for 2 h under a nitrogen atmosphere. The leaching of a total of

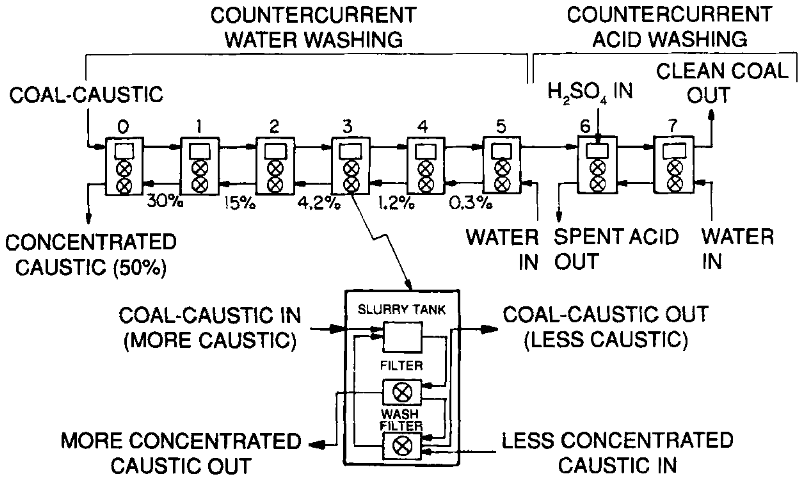

FIG. 1. Representation of the Ames Laboratory countercurrent washing procedure.

2100 g coal and caustic resulted in the recovery of 1950 g product. The remaining 150 g material were lost as water and other volatile compounds evolved during leaching. The leached products were composited and stored under a nitrogen atmosphere until countercurrent washing commenced. The composited sample was then ground and split into 14 identical 139-g samples.

Composition of Coal–Caustic Mixtures

One 139-g aliquot of coal–caustic was slurried with 500 mL water. The slurry was heated to 85°C for 5 min and was then vacuum-filtered through Whatman No. 41 filter paper using a Buchner funnel. The coal sample was next washed with an additional 1.5 L boiling water which was added in approximately 100-mL aliquots. The filtrate was diluted to exactly 2.0 L. The coal cake was slurried with 200 mL water, and the pH of the slurry adjusted to 1.0 with sulfuric acid. This slurry was then vacuum-filtered through Whatman No. 41 filter paper in a Buchner funnel and washed with an additional 1800 mL boiling water which was added to the filter cake in approximately 100-mL portions. The filtrate from the acid washing was also diluted to exactly 2.0 L. The product coal was dried at 110°C for 8 h.

The cleaned coal samples resulting from this washing were analyzed for ash and heating value by ASTM procedures and for total sulfur by using a Fisher total sulfur analyzer. In the water filtrates, hydroxide and carbonate were determined by titration, aluminum and silicon by atomic absorption spectrophotometry, and iron by spectrophotometry using 1,10-

phenanthroline. Iron, silicon, and aluminum were also determined in the acid filtrates.

Countercurrent Washing

Eleven separate 139-g aliquots of the coal–caustic mixture were washed countercurrently using the procedure depicted in Fig. 1. At the end of the water-washing stages of the procedure, 1-g samples of coal cakes were withdrawn for analysis and the rest of the coal samples were washed countercurrently with dilute sulfuric acid, as also depicted in Fig. 1. The net result of this countercurrent washing was the recovery of 11 concentrated caustic samples, 11 cleaned coal samples, 11 water-washed coal samples, 11 spent acid samples, 10 samples of intermediate caustic streams in process at the time the 11th coal sample was processed, and 3 acid streams in process at the time the 11th coal sample was processed. Each of the caustic-containing samples was analyzed for carbonate, hydroxide, sodium, potassium, iron, aluminum, silicon, and total sulfur. Each of the acid-containing samples was analyzed for residual acid, iron, silicon, aluminum, sodium, and potassium. The 11 cleaned coal samples were analyzed for total sulfur, ash, and heating value. The 11 aliquots of water-washed coal samples were titrated to determine the total alkalinity remaining on the coal after water washing. The titration solutions were then filtered, and the filtrates were analyzed for sodium, potassium, iron, silicon, and aluminum that had been released from the coal samples into the acidic titration solutions; the coal samples were analyzed for ash and total sulfur content.

RESULTS AND DISCUSSION

Filter Cake Buildup

There was a buildup in the weight of filter cakes produced during the countercurrent washing of Pittsburgh No. 8 coal that had been leached with NaOH–KOH mixtures. As shown in Fig. 2, this buildup became more massive as more coal samples were washed by the countercurrent procedure. These data show that the filter cakes produced after 11 coal samples had been washed had weights about 12 times those of the dry coal contained in them. If the cakes contained only coal wetted with wash solutions, the filter cakes would have had approximately 3–4 times the weight of dry coal they contained. As was previously the case with coal samples leached with NaOH alone (*6*), this buildup of filter cake mass was the result of precipitation of carbonates.

There were differences between the precipitation of carbonate from the washing of coal cakes which had been leached with molten sodium hydroxide–potassium hydroxide mixtures and coal cakes leached with sodium

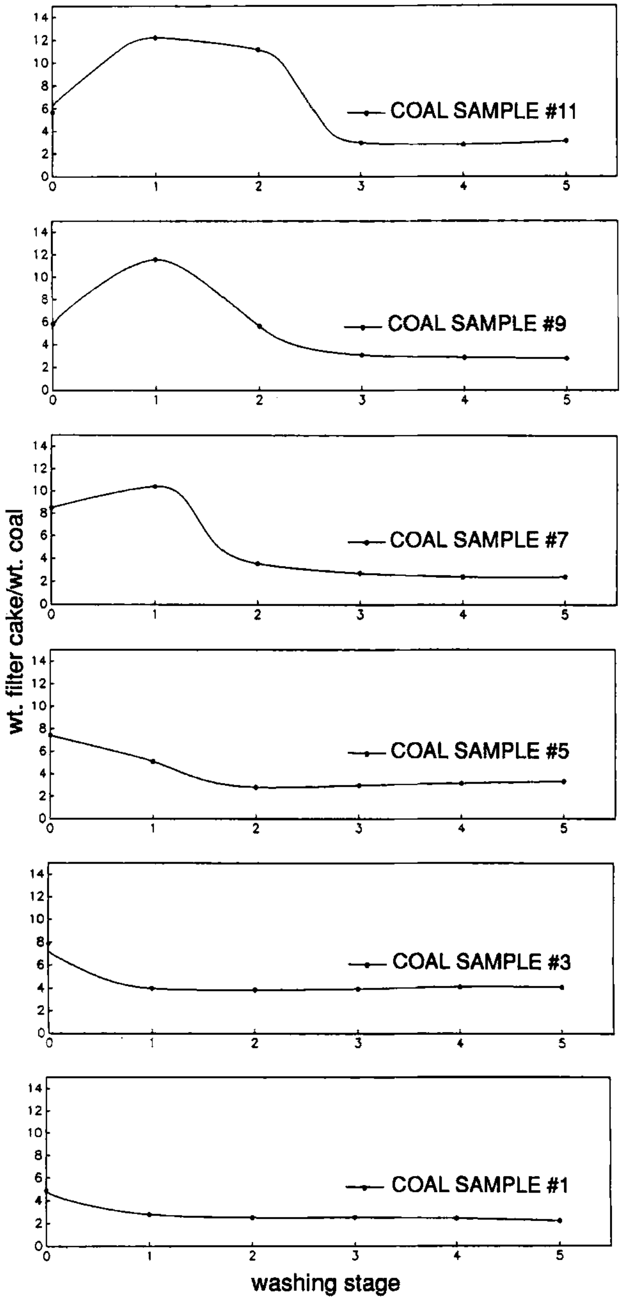

FIG. 2. Buildup of filter cake weights in intermediate washing stages as 11 successive coal samples were washed by the Ames Laboratory countercurrent washing procedure.

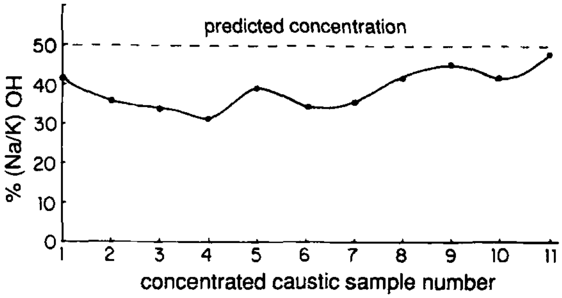

FIG. 3. Concentration of caustic in recovered concentrated caustic solutions at 11 successive coal samples were washed by the Ames Laboratory countercurrent washing procedure.

hydroxide only. As shown in Fig. 2, the present studies showed that sodium–potassium carbonate buildup persisted in the initial stages of countercurrent washing, whereas previous studies showed that sodium carbonate buildup progressed very rapidly to the final stages of countercurrent washing (*6*). However, it is anticipated that if more coal samples were washed countercurrently, the buildup would eventually reach the final stages even when NaOH–KOH was used.

Recovery of Concentrated Caustic Solutions

Countercurrent washing was predicted to produce a 50% caustic solution under the conditions used. As illustrated by the data in Fig. 3, the solutions produced did indeed approach 50% concentrations. However, as shown in Fig. 4, after the first few samples of coal–caustic were washed, the amounts of caustic solution produced declined. Less than half as much caustic solution was recovered during washing of the final 5 coal samples than was predicted. The reason for this decline in caustic production was

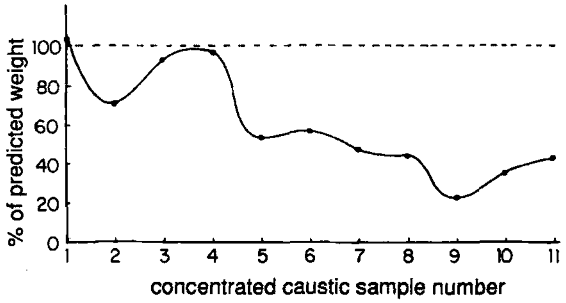

FIG. 4. Amount of caustic in recovered concentrated caustic solutions as 11 successive coal samples were washed by the Ames Laboratory countercurrent washing procedure.

TABLE 1
Fate of Caustic (NaOH–KOH) during Countercurrent Washing

	Predicted		Actual	
Stream	grams	%	grams	%
Concentrated caustic product	695.2	83.3	505.3	60.5
Caustic solutions in process	139.8	16.7	193.8	23.2
Caustic retained on apparatus	0.0	0.0	8.2	1.0
Converted to carbonate and humates during countercurrent washing	0.0	0.0	49.0	5.9
Neutralized during acid washing	0.0	0.0	58.8	7.0
Total	835.0	100.0	815.1	97.6

simply that the carbonate-caused buildup of filter cake mass led to a greater retention of fluids in intermediate stages of countercurrent washing. A summary of the fate of the caustic entering the countercurrent washing process is given in Table 1. These data reveal that only 61% of the caustic entering the procedure was recovered in concentrated caustic solutions as opposed to the 83% predicted.

Fate of Carbonate

In Fig. 5 the total alkalinity of intermediate process streams of the countercurrent washing procedure is shown to be much higher than the predicted amounts of NaOH–KOH. Data presented in Fig. 6 show that this increased alkalinity in the intermediate streams is due to carbonate. Of the carbonate present on coal samples entering the countercurrent procedure, 96% remained in the intermediate stages of the procedure after the 11th coal

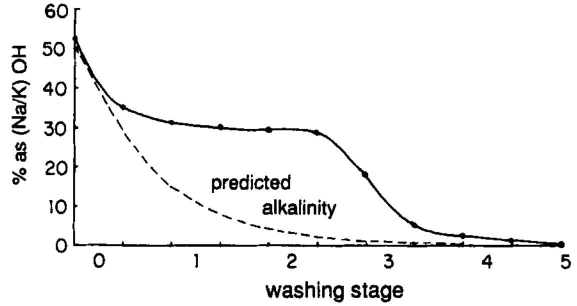

FIG. 5. Total alkalinity as (Na/K)OH in intermediate streams in process at the time the final coal sample was washed after 11 successive coal samples were washed by the Ames Laboratory countercurrent washing procedure.

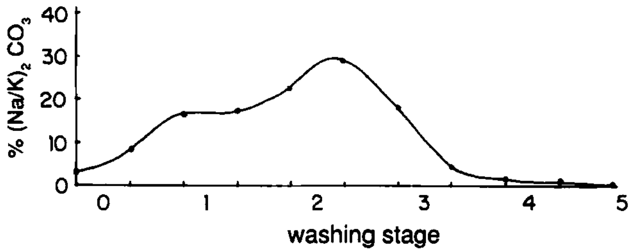

FIG. 6. Percent carbonate as (Na/K)CO$_3$ in intermediate streams at the time the final coal sample was washed after 11 successive coal samples were washed by the Ames Laboratory countercurrent washing procedure.

sample was washed, either in solution or precipitated upon the apparatus (Table 2). An additional 10% carbonate exited the process in concentrated caustic solutions. It is apparent that the amount of carbonate accounted for is greater than the amount entering the procedure. However, it has previously been found that prolonged contact between coal and aqueous caustic at elevated temperatures does lead to the formation of additional carbonate. Thus, it was not surprising that more carbonate was formed during countercurrent washing.

Recovered Clean Coal

The cleaned coal recovered from the countercurrent washing procedure had residual sulfur levels which showed an average sulfur removal of 73%. This compares with a sulfur removal of 86% obtained on an identical coal sample that was washed directly with extensive amounts of water. In addition, it was found that portions of coal removed from the process after water washing and then leached with hydrochloric acid had an average

TABLE 2
Fate of Carbonate during Countercurrent Washing

	Carbonate	
Stream	grams	%
Carbonate in coal-caustic input	418.0	100.0
Carbonate in concentrated caustic product	42.2	10.1
Carbonate in process streams	327.2	78.3
Carbonate retained on apparatus	76.0	18.2
Total	445.4	106.6

sulfur removal of 78%. These data indicate that 1) countercurrent water washing is less effective in removing soluble sulfur-containing compounds from leached coal than is extensive water washing, and 2) washing the water-washed coal samples with hydrochloric acid rather than sulfuric acid results in slightly greater reductions in residual sulfur levels. The apparent greater effectiveness of hydrochloric acid is quite reasonable because some sulfur is reincorporated into coal during washing with sulfuric acid (*12*).

In contrast with the results for sulfur removal, countercurrent washing of coal samples led to an almost identical removal of ash-forming minerals as did extensive water washing. In both cases, 91% of the ash-forming minerals were removed from the coal. However, aliquots which were washed with hydrochloric acid exhibited an ash removal of nearly 99%. This indicates the existence of some ash-forming species in the MCL-treated coal that is soluble in hydrochloric acid but not in sulfuric acid. Since the ash from coals has been found to contain predominantly iron, it is likely that the species is some iron-containing compound. One possibility is that insoluble jarosite-like double salts (*13*) form on the coal during acid washing with sulfuric acid.

The material balance for coal during the countercurrent washing process was excellent. As shown in Table 3, essentially quantitative accounting of coal was obtained.

Acid Wash Streams

In order to determine consumption during the acid-washing step, the amount of sulfuric acid recovered in the spent acid streams resulting from acid washing was compared to the amount of sulfuric acid added. In addition, the amount of sodium and potassium released during acid washing was determined and compared with the equivalents of acid consumed.

TABLE 3
Fate of Coal during Countercurrent Washing

	Recovery (MAF basis)	
Stream	grams	%
Input coal	373.5	100.0
Recovered clean coal	329.4	87.4
Coal retained on apparatus	40.8	10.9
Coal withdrawn for analysis	4.7	1.2
Coal remaining in solutions	5.9	1.6
Total	377.9	101.2

Also, the equivalents of hydrochloric acid consumed by the aliquots of water-washed coal were determined and compared with these values.

The equivalents of sulfuric acid consumed during acid washing should have been the same as those of hydrochloric acid required to titrate aliquots of the same coal samples, and the equivalents of sodium plus potassium released should have been very close to these. As shown in Table 4, the amount of hydrochloric acid consumed and the average amounts of sodium plus potassium released were indeed very comparable, but about 50% more sulfuric acid was consumed. Some of this sulfuric acid undoubtedly was reincorporated into the coal, and this accounts for the lower sulfur removal observed with sulfuric acid washed cleaned coal samples. The fact that hydrochloric acid washing resulted in greater removal of ash-forming minerals than did the washing with sulfuric acid makes it very unlikely that the "missing" sulfuric acid was consumed by other coal impurities.

Distribution of Sodium and Potassium

A total of 316 g sodium and 383 g potassium were contained in the caustic used for leaching the coal samples washed by the countercurrent procedure, and 308 g sodium and 378 g potassium were found in process streams (Table 5). This was considered an excellent material balance for these species. In the caustic used for leaching the coal samples, sodium and potassium were present in a ratio of 0.83 to 1. However, as is also

TABLE 4
Acid Consumption and Na/K Release by Water-Washed Coal Samples

	Milliequivalents/gram of coal		
Coal sample	HCl	Na + K	H_2SO_4
1	2.8	1.9	7.1
2	2.6	1.5	2.2
3	2.5	2.9	5.6
4	2.3	2.7	4.1
5	2.5	2.9	3.0
6	2.5	2.6	3.1
7	2.3	2.0	2.5
8	2.2	2.6	3.9
9	2.3	2.2	4.6
10	2.4	2.9	4.1
11	2.4	2.7	2.7
Average	2.4	2.4	3.8

TABLE 5
Fates of Sodium and Potassium during Countercurrent Washing

Stream	Sodium grams	Sodium %	Potassium grams	Potassium %	Sodium/potassium ratio
Input caustic	316.3	100.0	383.0	100.0	0.83
Concentrated caustic	120.6	38.1	209.3	54.6	0.58
In process	155.7	49.2	139.7	36.5	1.11
On apparatus	19.3	6.1	17.2	4.5	1.12
On cleaned coal	12.1	3.8	11.9	3.1	1.02
Total recovered	307.7	97.3	378.1	98.7	0.81

shown in Table 5, sodium was depleted from the recovered caustic. Conversely, sodium was enriched in the intermediate streams in process at the termination of the washing, in the material retained on the apparatus, and on the cleaned coal. These data indicate, as would be expected based upon solubilities, that sodium carbonate is preferentially precipitating on coal during countercurrent washing.

As depicted in Fig. 7, the depletion of sodium was already significant in the first concentrated caustic sample recovered and decreased further to a low level by the 5th sample. As also shown in Fig. 7, sodium was slightly depleted from the initial acid wash streams but was enhanced in streams arising from the acid washing of the 5th and all subsequent coal samples. The enrichment of sodium relative to potassium in intermediate streams in process at the end of the run paralleled the concentrations of carbonate in these same streams. These data are all consistent with the preferential precipitation of sodium carbonate from concentrated NaOH–KOH caustic streams.

The formation of carbonate precipitates during countercurrent washing is favorable to the overall process only in that sodium carbonate appears to precipitate preferentially to potassium carbonate. Sodium hydroxide, which is far less expensive than potassium hydroxide, can thus be used as make up for caustic that has been consumed. However, one process being considered for treatment of spent acid streams involves the formation of double salts such as jarosite (*13*). These salts form more readily with potassium than with sodium, and thus there is no advantage in the preferential retention of sodium rather than potassium on coal for the subsequent treatment of the spent acid streams.

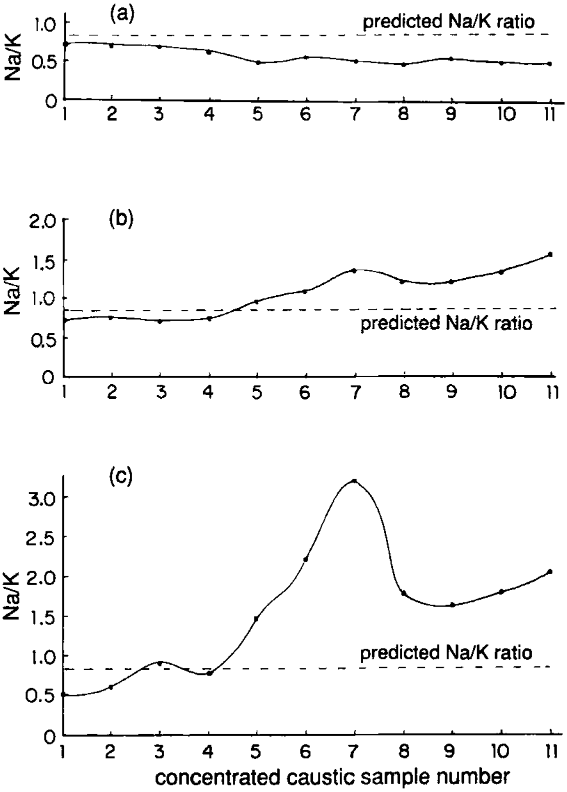

FIG. 7. Na/K ratio in (a) concentrated caustic products, (b) acid wash streams, and (c) intermediate streams in process when the final coal sample was washed after 11 successive coal samples were washed by the Ames Laboratory countercurrent washing procedure.

Fate of Silicon, Aluminum, and Iron during Countercurrent Washing

Silicon, aluminum, and iron are the major components of ash-forming minerals in the Pittsburgh No. 8 coal studied. These elements are believed to be converted to silicates, aluminates, and ferrates during molten caustic leaching (*14*, *15*). These salts are soluble, but during the washing of coal–caustic cakes containing these salts, chemical transformations can occur. As shown in Table 6, much of the recovered silicon was present in the concentrated caustic product stream and essentially none in the acid wash streams. In contrast, essentially equivalent amounts of aluminum were recovered in the caustic products and the spent acid streams, and most of

TABLE 6
Fate of Silicon, Aluminum, and Iron during Countercurrent Washing, in grams

Species	In concentrated caustic	In spent acid	In process
Silicon	3.3	<0.5	1.7–2.4
Aluminum	0.4	0.4	0.6
Iron	0.7	6.0	0.9

the iron was recovered in the acid wash streams and very little in the caustic product.

There was a buildup of silicon, aluminum, and iron in the intermediate streams in process when the final coal sample was washed, but the total amount of these species was less than 3 g total compared with the 330 g carbonate in the same streams. Thus, the observed buildup of iron, silicon, and aluminum in intermediate process streams probably only reflects entrainment of these compounds in a much larger carbonate precipitate.

CONCLUSIONS

Countercurrent washing of coal samples leached with a molten sodium–potassium hydroxide mixture resulted in similar problems of carbonate precipitation and buildup as were encountered previously with coal samples leached with molten sodium hydroxide alone. The carbonate precipitates formed appear to be significantly enriched in sodium and depleted in potassium. Control of carbonate precipitation either by significantly reducing the amount of carbonate entering the countercurrent washing step or by removing it during the step will be required in order for washing to proceed efficiently for more than a few coal samples.

Acknowledgments

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