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COMBUSTION AND LEACHING BEHAVIOR OF ELEMENTS
IN THE USGS COAL STANDARD CLB-1

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Combustion and Leaching Behavior of Elements in USGS
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Abstract

A series of heating and leaching experiments were conducted on a split taken from the coal used to create the U. S. Geological Survey's CLB-1 coal standard. The behaviors of approximately 30 elements were monitored by a variety of analytical procedures, including instrumental neutron activation analysis, inductively coupled plasma optical emission spectroscopy, atomic absorption spectroscopy, and ion chromatography. Br, Se, and Hg were the only elements to exhibit volatilization at temperatures up to 1000 C. At this temperature virtually all of these elements had been volatilized. Leaching of the coal with hot (80 C) distilled water generally removed no more than a few percent of the elements monitored. Hot water leaching of the low-temperature ash was more effective, removing from 10 to 50 percent of many elements. A split of the coal was subjected to sequential leaching by ammonium acetate, hydrochloric acid (HCl), hydrofluoric acid (HF), and nitric acid. Ammonium acetate, which was used to

remove ion-exchangeable cations, removed 40 to 50 percent of the Ca. HCl (1:3) was effective in removing 20 to 40 percent of the chalcophile elements (As, Sb, Zn, and Fe); elements that are generally acid soluble when associated with monosulfides. HF (48%) removed 20 to more than 80 percent of the elements (K, Na, Cs, Sc, Hf, U, and Mg) commonly associated with silicate and oxide minerals. Nitric acid (10%) was generally ineffective in leaching elements from this coal sample. The responses of the various elements in the Lower Bakerstown coal during the combustion and leaching experiments were similar to the behavior reported for the elements in other Appalachian basin coals.

Introduction

The U. S. Geological Survey has prepared a standard coal sample for the analysis of elements in coal. The standard, referred to as CLB-1, was prepared from a surface mine sample of the Lower Bakerstown coal, an Upper Pennsylvanian, medium-volatile bituminous coal from Maryland. Description of the collection procedure and chemical analysis of this coal will be the subjects of other papers.

We report on the behavior of about 30 elements upon combustion of this coal at different temperatures and upon leaching with various solvents. Much of the data in this paper has been taken from Finkelman and others (1990) who described the combustion and leaching behavior of the elements in the Lower Bakers-

town coal and in nine other coal samples.

The sample used in this study was extracted, prior to final grinding and homogenization, from the material collected for the preparation of the standard. Nevertheless, the information presented here should be useful in determining the modes of occurrence and, therefore, the physical and chemical responses of the elements in the CLB-1 coal standard.

Experimental Procedure

The Lower Bakerstown sample was ground to -100 mesh. A split was analyzed by instrumental neutron activation analysis (INAA) for 30 elements. Details of the analytical procedure are given in Palmer and Baedeker (1989). A split was ashed in a low-temperature (LT) ashing unit following the method of Miller (1984). The maximum temperature attained during LT ashing is believed to be less than 200 C. Other splits of the coal were ashed at 1) 550 C, the temperature generally used by the U. S. Geological Survey for ashing coal samples (Walthall and Fleming, 1989), 2) 750 C, the temperature recommended by the ASTM for ashing coal (ASTM, 1990), and 3) 1000 C. The residues from all the ashing procedures were analyzed by INAA and the analytical results calculated to an as-received coal basis and compared to the analytical data from the coal. All analytical results presented in this paper are on an as-received basis. Table 1 indicates the analytical procedures used to determine the concentration of each element.

A 10-g split of the Lower Bakerstown coal sample was subjected to sequential leaching, first with 50 mL of 1 N ammonium

acetate (NH_4OAc), followed by 50 mL of 1:3 hydrochloric acid (HCl), then with 50 mL of 48 percent hydrofluoric acid (HF), and finally with 50 mL of 10 percent nitric acid (HNO_3). The sample was agitated at room temperature (27 C) in each of the solvents for 18 to 24 h. After leaching with each solvent, the residue was rinsed two to three times with distilled water, air-dried, and weighed. Splits of the leached coal were then removed for INAA prior to leaching with the next solvent. Analytical results were calculated to an as-received basis, adjusting for material loss from leaching.

Other tests were performed, such as hot (80 C) water leaching the coal and the LT ash, and a separate HCl leaching experiment. Some leachates were analyzed by atomic absorption spectroscopy (AAS), inductively coupled plasma optical emission spectroscopy (ICP-OES), and ion chromatography (IC). Descriptions of the analytical procedures can be found in Golightly and Simon (1989). Semiquantitative mineralogy of the LT ash was determined by X-ray diffraction following the procedure of Hosterman and Dulong (1989). An analytical scanning electron microscope (SEM-EDX) was used to view and to identify the minerals in polished pellets of the coal.

The analytical error for most elements is less than 10 percent of the amount present (Finkelman and others 1990). Because of propagation of errors and the uncertainty as to the efficiency of the leaching procedures, we consider leachabilities (Tables 3, 4, and 5) of 10 to 20 percent to be of questionable significance.

Results

Combustion Experiments

The purpose of the combustion tests was to determine the degree of volatility of the elements at various temperatures. Our criteria for establishing volatility were that at least 10 percent of the element was lost upon heating, and that this loss had to be sustained at all higher temperatures. The analytical error for most elements was less than 10 percent of the amount detected in the coal samples and in the ash samples.

Only three of the 30 elements monitored met these criteria; bromine, selenium, and mercury. Table 2 shows the percentage of each element volatilized during LT ashing and at 500 , 750 , and 1000 C. Each of these elements exhibited increasing volatilization with increasing temperature. By 1000 C virtually all of these elements had been volatilized. The behavior of these elements is consistent with the low boiling point of several of their compounds (Weast, 1980).

Atomic absorption spectroscopic analysis of the 550 C ash and the LT (<200 C) ash indicated that no volatilization of Cd, Cu, Li, Mg, Mn, Pb, and Zn occurred between these temperatures. There are, of course, other volatile elements in this coal, such as Cl, F, and S, but they were not included in our analytical scheme.

Leaching Experiments

Table 3 contains the results of the leaching experiments. The data have been modified from Finkelman and others (1990) by rounding their values to the nearest multiple of five. In general, less than 50 percent of each element was removed by the sequential leaching procedure. The exceptions were the transition metals (Fe, Co, Ni, and Zn), the alkali metals (Na, K, Cs), and Sb. The high proportion of water and/or ammonium acetate leachable Fe, Co, Ni, Zn, Ba, and Sr are probably due to contamination.

Hot Water Leaching of Coal - Aside from the questionable results for Fe, Co, Ni, Ba, and Sr, no element exhibited a substantial (greater than 20 percent) loss on hot water leaching. Ten to 20 percent of the rare-earth elements (REE), Th, and Sc were removed by hot (80 C) water leaching. ICP-OES analysis of the leachate indicated that the hot water did not extract Na, K, Fe, Ba, Ca, or Mg.

A separate split of the Lower Bakerstown coal was leached with hot water and the leachate analyzed by ICP-OES and IC (Table 4, column 1). No Fe, Co, Ni, Ba, or Sr was leached from the coal. These data confirm that the high values obtained for the elements from the hot water leach described above are due to contamination.

Hot Water Leaching of LT ash - 100 to 200 mg splits of the LT ash were leached with 30-40 ml hot (80 C) distilled water for 18 h in a hot water bath. After centrifugation and vacuum filtration

the leachates were analyzed by ICP-OES and IC. The results were calculated to a coal basis to determine the proportion of the element in the coal that had been extracted by this procedure (Table 4). At least 10 percent of every element monitored, except Ba, Li, Br, and N, was removed from the LT ash by the hot water. In contrast, virtually none of these elements were removed on hot water leaching of the coal.

This behavior is probably, in part, due to the formation of water soluble oxides and sulfates during the LT-ashing process. Specifically, water soluble oxides could form from the organically bound cations that are not volatilized (e. g., Mg, Na, Ni, S). Soluble oxides and sulfates could form from elements in sulfide minerals that are oxidized (e. g. Fe, Co, Ni, Zn, S). Acids (e. g. sulfuric acid) generated from the leaching process may attack carbonates releasing Fe, Mg, Mn, and Sr. The increased leachability of the LT ash may also be due to increased accessibility of the minerals to the solvent.

An interesting aspect is the low proportion of water-soluble Br, N, and S in the LT ash. These elements commonly form water-soluble salts. Finkelman and others (1990) found that about 90 percent of the Br in the Lower Bakerstown coal had been volatilized during LT ashing. This is consistent with Lyons and others (1989) who found that Br had the highest degree of organic association in the coals in their study, including a sample of the Lower Bakerstown coal collected from the same mine. It is likely that much of the N and S are also volatilized during LT ashing, accounting for the low proportion of water solubility for these

elements in the LT ash.

Ammonium acetate leach - Ammonium acetate was effective only in removing calcium (40 percent: Table 5) from the CLB-1 sample. Removal of Sr, Cs, Sb, Yb (Table 3), Mg, and Na (Table 5) is problematical. The absence of Ba and Fe in the ammonium acetate leachate (Table 5) supports the likelihood that the indication of ammonium acetate leachable Ba and Fe (Table 3) was due to contamination.

Hydrochloric acid leach - HCl was effective in removing As (40 percent), Sb (35 percent), and Fe [25 percent as determined from the analysis of the leached coal (Table 3) and 35 percent as determined from the analysis of the leachate (Table 5)]. HCl removed from 10 to 20 percent of Zn, Co, Sc, Br, Sm, Eu, Tb, and Ca.

A separate split of the Lower Bakerstown coal was leached directly with HCl. The unleached coal, the leached coal, and the leachate were analyzed by ICP-OES (Table 6). Mass balance was achieved for all elements and there was excellent agreement with the previous leaching experiments (Table 3).

Hydrofluoric acid leach - HF was the most effective acid for removing those elements commonly associated with silicate and oxide minerals (e. g. K, Na, Cs, Sc, Hf, U, and Mg). Other elements, of which more than 10 percent was removed by HF, include W, Sb, and Br. Results from the analysis of the leachates (Table 5) compare favorably with the results from the analysis of the leached coal (Table 3).

Nitric acid leach - From 10 to 20 percent of the REE were removed by HNO_3 . The reason for this is unclear. Cs, Na, and Mg were the only other elements that HNO_3 removed more than 10 percent. The HNO_3 leaching procedure used in this study was not as vigorous as the ASTM procedure (ASTM, 1990) accounting for the fact that Fe and other chalcophile elements were not extracted.

Mineralogy

Table 7 contains the results of the semiquantitative X-ray diffraction analysis. The mineralogy of this coal is similar to the mineralogy of most Appalachian coals and shows no distinctive or unusual characteristics (F. Dulong, personal commun., 1991). Minerals observed with the SEM-EDX system include: quartz, pyrite, illite, kaolinite, rutile, zircon, barite, monazite, galena, and iron oxides. These tentative mineralogical identifications are based on major element chemistry and morphology.

DISCUSSION

The combustion and leaching behavior of most elements in the sample of the Lower Bakerstown coal was similar to the behavior of the elements in the other Appalachian coal samples studied by Finkelman and others (1990). There was only small quantities of water soluble or ion-exchangeable cations (except for Ca), typical of Appalachian bituminous coals (Finkelman and others, 1990).

Most of the Na and K and much of the Sc and Cs were removed by HF. This is consistent with these elements occurring in sili-

cates. X-ray diffraction analysis of the LT ash indicates that illite, a K-bearing aluminosilicate, made up about 15 percent of the ash (Table 7). This is supported by the SEM-EDX analysis which showed that a potassium aluminum silicate, presumably illite, was a common mineral in this coal. The illite would account for about 1,000 ppm K, assuming no substitution of other alkali elements for K. This value compares favorably with the 700 ppm K in the CLB-1 standard. Some of the Ca is associated with carbonates and apatite (Table 7), but a substantial proportion is ion-exchangeable. The exchangeable Ca may be associated with clays and/or with organic functional groups. The SEM-EDX analysis indicated that a substantial amount of calcium was in the organic matrix. As this calcium was not associated with silicon or aluminum it appears that the calcium was chemically bound to the organics.

Semiquantitative X-ray diffraction analysis of the LT ash indicated approximately 15 weight percent pyrite. As the LT ash constitutes 8.5 percent of the coal this amount of pyrite is equivalent to about 6,500 ppm Fe in the coal. Sulfur-form analysis (13 determinations) indicates 0.36 weight percent pyritic sulfur. This amount of pyritic sulfur requires approximately 3,500 ppm Fe. The certified amount of Fe in CLB-1 is 7,800 ppm. Accepting the sulfur-form analysis value for pyritic sulfur as a more accurate value than the X-ray diffraction value for pyrite, we conclude that about 40 percent of the Fe occurs in pyrite. The leaching experiments indicated that 50 percent of the Fe was removed by ammonium acetate, HCl, and HF. This iron is likely to

be in exchangeable sites in clay, in carbonates (such as siderite), oxides, and silicates. The SEM-EDX analysis indicates the presence of iron oxide grains. The textural features of the grains indicated that they were not contamination but were indigenous to the coal. No siderite particles were observed with the SEM.

Removal of the chalcophile elements (Zn, Sb, As, and perhaps Co) by HCl is probably attributable to the presence of acid soluble monosulfides such as sphalerite (ZnS), a trace of which was detected by X-ray diffraction (Table 7). No leaching data could be obtained for Hg. The only monosulfide observed with the SEM-EDX was galena (PbS).

Se was unaffected by the solvents. It is generally assumed that Se is organically associated, substituting for S in the coal structure (Lakin and Davidson, 1973). The absence of any response by Se to the solvents used in this study is consistent with this assumption.

Br was largely unaffected by the solvents. This behavior could be explained if Br occurred as an ionic species primarily in inaccessible micropores, as has been postulated for Cl (Demir and others, 1990).

Twenty percent of the hafnium was removed by HF. This behavior is consistent with Hf occurring in zircons, some of which may be metamict and, therefore, vulnerable to HF attack. The SEM-EDX analysis indicated that fine-grained (<5 micrometers) zircons are present in this coal.

The combustion and leaching experiments did not offer any insights into the modes of occurrence of Ba, Sr, Ta, U, and Th.

This may be due, in part, to these elements occurring in fine-grained minerals encapsulated by the organic matrix, or to their occurring as insoluble minerals, such as barite (BaSO_4), or tantalite (FeTa_2O_6). Barite was observed in the Lower Bakerstown sample with the SEM-EDX.

Only a small amount (15 percent) of W was removed (by HF) in the leaching experiment. Eskenazy (1982) found that W was removed from Bulgarian coals only by HF and NaOH. She has postulated that W is present in coal as an organometallic complex.

Ten to 20 percent of the U was extracted by HF and HNO_3 . Much of the U in Appalachian coals occurs in zircons and in organic association (Finkelman, 1981). The leaching data do not allow us to differentiate between these modes of occurrence.

The proportion of rare-earth elements (REE) removed by HCl and HNO_3 in the Lower Bakerstown sample was the among the lowest of the 10 samples studied by Finkelman and others (1990). The light REE were more readily leached than the heavy REE. Finkelman and others (1990) concluded that the REE in the Lower Bakerstown and the nine other coal samples occurred in the phosphate minerals, monazite, and xenotime. Several probable monazite particles were detected with the SEM-EDX.

The proportion of many elements leached from the Lower Bakerstown sample were among the lowest of the 10 samples studied by Finkelman and others (1990). There are several possible explanations. A relatively large proportion of the elements in the Lower Bakerstown coal may be inorganically associated. The relatively high-rank Lower Bakerstown coal may have a lower porosity than

the other samples, thus reducing the accessibility of the acids to these minerals. Fine-grain size of the minerals may also account for the low proportion of leachable elements in the Lower Bakerstown coal sample. The SEM-EDX analysis indicated that the minerals in this coal are finely divided, generally less than 10 micrometers. These minerals are commonly embedded in a clay matrix or enmeshed in the organics. This would have the effect of reducing contact between the minerals and the solvents.

The combustion and leaching behavior of elements in the Lower Bakerstown coal did not reveal any unusual responses. This indicates that the elements in this coal are predominantly in the modes of occurrences typically found in Appalachian bituminous coals (Finkelman, 1981).

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Table 1. Analytical Methods

<u>Element</u>	INAA	ICP-OES	AAS	IC	<u>Other</u>
	<u>Coal</u>	<u>Leachate</u>	<u>Leachate</u>	<u>Leachate</u>	
Na	X	X	X		
K	X	X			
Sc	X	X			
Cr	X				
Fe	X	X			
Co	X	X			
Ni	X	X			
Zn	X	X			
As	X				
Se	X				
Br	X			X	
Rb	X				
Sr	X	X			
Sb	X				
Cs	X				
Ba	X	X			
La	X				
Ce	X				
Nd	X				
Sm	X				
Eu	X				
Tb	X				
Yb	X				

Lu	X				
Hf	X				
Ta	X				
W	X				
Hg	X				1
Th	X				
U	X				
Ti	X				
Al	X				
Ca		X			
P		X			
Mg		X	X		
Mn		X	X		
Li		X	X		
Cd		X	X		
Cr		X			
Cu				X	
Pb				X	
Zn				X	
F					X
N					X
S					X

1 - Cold-vapor AAS

2 - Ion-selective electrode

Table 2. Percentage of Element Volatilized at Different
Temperatures

<u>Temperature</u>	<u>Br</u>	<u>Se</u>	<u>Hg</u>
LTA (<200 C)	90	30	50
550 C	99	85	70
750 C	>99	92	>99
1000 C	>99	>99	>99

Table 3. Percentage of Element in the Lower Bakerstown Coal Sample Leached by Various Solvents

Element	H2O	NH4OAc	HCl	HF	HNO3	Total*
Na	0	0	0	65	0	65
K	0	0	0	>80	-	>80
Sc	15	0	10	30	0	40
Cr	0	0	0	10	10	20
Fe	(25) ⁺	15	25	10	0	50
Co	(55)	(60)	10	0	0	(70)
Ni	(25)	(55)	0	0	0	(55)
Zn	0	(65)	20	0	0	(85)
As	0	0	40	0	0	40
Se	0	0	0	0	0	0
Br	0	0	20	15	0	35
Rb	-	-	-	-	-	-
Sr	(20)	15	0	10	10	35
Sb	0	10	35	20	0	65
Cs	0	10	-	35	15	60
Ba	(>35)	(25)	-	-	-	>25
La	0	0	0	0	20	20
Ce	10	0	0	0	20	20
Nd	-	-	-	-	-	-
Sm	10	0	10	0	15	25
Eu	10	0	15	0	15	30
Tb	15	0	15	0	15	30
Yb	10	20	-	-	10	>30
Lu	-	-	-	-	-	-
Hf	0	0	0	20	0	20
Ta	0	0	0	0	0	0
W	0	0	0	15	0	15
Hg	-	-	-	-	-	-
Th	15	0	0	0	10	10
U	0	0	0	20	10	30

* Excluding water leach

Dash(-)= no data

+ values in parenthesis are believed to be due to contamination

Table 4. Hot Water Leachability of Selected Elements in the Lower Bakerstown coal and LT ash. Analysis by ICP-OES and IC.

(Values are in percent of the elemental concentration in coal)

Element	Coal	LT ash*
Fe	0	10
Mg	0	25
Mn	0	35
Ba	0	0
Co	0	30
Cr	0	10
Ni	0	40
Sc	0	<30
Sr	0	20
Zn	0	50
Na	0	10
Li	0	10
Al	0	10
F	10	<65
Br	0	0
N #	0	0
S #	0	10-20

* Average of two to three separate leaching experiments or range of values.

Determined as nitrate and sulfate, respectively.

Table 5. AAS Analysis of Leachates

(Average of two analyses. Values are in percent of the elemental concentration in coal except the second column which is in ppm)

<u>Element</u>	<u>Solvents</u>				
	<u>CLB-1*</u> (ppm)	<u>NH₄OAc</u> (%)	<u>HCl</u> (%)	<u>HF</u> (%)	<u>HNO₃</u> (%)
Ca	1610	40	10	0	10
Mg	284	10	0	20	20
Na	185	15	0	65	15
K	700	0	0	60	0
Ba	63	0	0	0	10
Fe	7800	0	30	0	0

* Data from J. Kane, U. S. Geological Survey, Reston, VA.

Table 6. ICP-OES Results of Direct HCl Leach.
 (Values are in ppm, except as noted)

Element	Raw Coal	Leached Coal	Diff. (%)	Leachate	NH ₄ +HCl*
Fe	8500	6400	-2100(25)	2900	2900
Mg	260	260	0 (0)	<2000	38
Ca	1500	360	-1140(75)	700	830
Ti	500	610	+110	<10	ND
Mn	10	3	7(70)	5	ND
Ba	40	40	0(0)	3	<1
Sr	80	50	30(65)	23	ND
Na	170	140	30(20)	30	30
P	360	60	300(85)	260	ND
Al	8300	8200	100(0)	ND	ND

* Sum of columns 3 and 4, Table 3

Table 7. Semiquantitative X-ray Mineralogy of the CLB coal Compared to the Mineralogy of Pocahontas Basin Coals (values are in weight percent of low-temperature ash)

	<u>QTZ</u>	<u>KAOL</u>	<u>ILL</u>	<u>MX-LYR</u>	<u>PYR</u>	<u>CAL</u>	<u>BASS</u>	<u>OTHERS</u>
CLB	15	45	15	trace	15	trace	trace	minor*
LP-5 ⁺	20	40	10	-	25	<5	-	minor ⁺
Poc.#	30	41	16	trace	4	3	1	minor

QTZ=quartz, KAOL=kaolinite, ILL=illite, MX-LYR=mixed layer clays, PYR=pyrite, CAL=calcite, BASS=bassinite, trace=<5 percent, minor=>5 and <10 percent. Dash (-) = no data.

* includes possible traces of siderite, apatite, sphalerite, barite, feldspar, chlorite, and rutile.

average of 60 analyses of Pocahontas coal LT ash (Renton, 1978).

+ data from Lyons and others (1989) for coal samples from the same mine. Other minerals include chlorite, feldspar, siderite.