

A Note on Difficult to Wash Coals from Southeast British Columbia (82G/7)

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KEYWORDS: *Coal washing, ash chemistry, carbonate contents, vitrinite reflectance, SEM on coal, petrography.*

TABLE 1 WASHABILITY DATA FOR THREE IN PIT SAMPLES

INTRODUCTION

Some coal seams in southeast BC are characterized by fine size consist and a moderate amount of inherent ash. Mining strategies and wash plants are designed to handle these coals but sometimes seams are mined, that are unusually difficult to clean. Samples of a number of such coals were acquired and studied. Samples come from a mine and an adjacent property (samples GC in tables) near Sparwood in southeast British Columbia. Two in-pit samples of coal were collected the mine from areas where the seam was known to wash easily and one sample from an area where it was known to be difficult to wash. A bulk sample of coal from a property near then mine was test washed at the mine wash plant and found to be difficult to wash and a sample of this coal was studied. Occasionally fine coal in the froth floatation circuits in the mine wash plant is difficult to wash. Raw, clean and reject samples of this material were collected. All samples were subjected to a range of chemical analyses, petrographic analysis and in some cases scanning electron microscope (SEM) analysis.

DATA

The three samples collected from the mine pit were crushed and screened at 10×0.6 , 0.6×0.15 and minus 0.15 millimetres (Table 1). The coarser size consists were washed using four SG increments. Oxide analyses (Table 2) were performed on some of the samples to provide an indication of how mineralogy varies between the easy and more difficult to wash samples. Sample 1980-69 was collected from an area where the coal was known to be difficult to wash. Samples 1968-east1 and 1968-east2 were collected from areas where the coal has normal washing characteristics.

The coal from the property is located in the hanging wall of a fault. The seam is thickened and crushed by the fault. A bulk sample of the coal was mined and test washed in the mine wash plant. Washing results and indications of potential coking quality were disappointing, so a sample was collected for further analysis. The sample was screened at 0.6 millimetres and the 0.6 x 0.0 milli

size mm	SG	wt	adm	VM	Ash	FC	Cash	Cwt
		Samp	le 196	8-east	1			
10x0.6		81.9	0.6	20.7	30.4	58.3	30.4	81.9
0.6x0.15		13.3	0.5	20.7	26.7	52.1	29.9	95.2
< 0.15		4.8	0.5	20.5	33.4	45.6	30.1	100
10x0.6	1.4F	41.5	0.1	22.5	6.8	70.6	6.8	41.5
10x0.6	1.4x1.6	15.9	0.2	21.1	20.3	58.4	10.5	57.4
10x0.6	1.6x1.8	6.6	0.3	19.7	40.8	39.3	13.7	64.0
10x0.6	1.8S	36.0	0.4	12.7	77.1	9.9	36.5	100
0.6x0.15	1.4F	54.8	1.0	24.3	4.9	69.8	4.9	54.8
0.6x0.15	1.4x1.6	15.5	0.6	21.0	17.4	61.0	7.7	70.4
0.6x0.15	1.6x1.8	4.9	0.8	19.8	36.9	42.6	9.6	75.3
0.6x0.15	1.8S	24.7	0.7	13.2	75.2	10.9	25.8	100
		Samp	le 196	8-east	2			
10x0.6		80.5	0.6	20.1	21.4	57.9	21.4	80.5
0.6x0.15		13.2	0.7	20.6	14.3	64.4	20.4	93.7
< 0.15		6.3	0.7	20.4	17.1	61.7	20.2	100
10x0.6	1.4F	55.6	0.2	21.6	6.5	71.7	6.5	55.6
10x0.6	1.4x1.6	26.3	0.2	20.4	18.2	61.2	10.3	81.9
10x0.6	1.6x1.8	5.4	0.3	20.2	37.3	42.3	11.9	87.3
10x0.6	1.8S	12.7	0.7	14.9	77.9	6.4	20.3	100
0.6x0.15	1.4F	61.1	1.1	23.1	5.0	70.8	5.0	61.1
0.6x0.15	1.4x1.6	27.3	1.2	19.7	15.4	63.7	8.2	88.4
0.6x0.15	1.6x1.8	4.2	0.8	19.7	34.3	45.3	9.4	92.5
0.6x0.15	1.8S	7.5	0.5	17.2	69.7	12.6	13.9	100
		Samp	le 198	0-69				
10x0.6		67.9	0.5	17.6	44.9	37.0	44.9	67.9
0.6x0.15		20.8	0.6	20.6	27.9	51.0	40.9	88.7
< 0.15		11.3	0.7	20.3	27.1	51.9	39.3	100
10x0.6	1.4F	26.5	0.2	25.8	7.0	67.1	7.0	26.5
10x0.6	1.4x1.6	11.2	0.3	21.6	21.9	56.2	11.4	37.7
10x0.6	1.6x1.8	18.1	0.3	18.9	41.0	39.8	21.0	55.8
10x0.6	1.8S	44.2	0.3	11.0	74.3	14.4	44.6	100
0.6x0.15	1.4F	44.8	1.3	26.3	6.2	66.2	6.2	44.8
0.6x0.15	1.4x1.6	22.7	1.0	22.0	18.0	59.0	10.1	67.5
0.6x0.15	1.6x1.8	10.0	1.3	19.3	38.1	41.4	13.8	77.5
0.6x0.15	1.8S	22.5	1.4	12.8	68.7	17.1	26.1	100
Cash=cun	nulative a	sh%	Cwt=	cumu	lative	weigh	t %	

TABLE 2 OXIDE DATA FOR INCREMENTAL WASHABILITY SAMPLES

millimetr	es	Ash	SiO_2	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K_2O	P_2O_5	Ва
1980-69												
10x0.6	1.8sink	74.3	68.8	1.3	23.2	1.04	0.5	0.3	0.1	1.9	0.1	0.1
10x0.6	1.8-1.6	41.0	64.8	1.7	28.5	0.83	0.3	0.2	0.2	0.6	0.1	0.1
10x0.6	1.6-1.4	21.9	65.5	1.7	28.7	0.57	0.3	0.1	0.2	0.9	0.1	0.1
10x0.6	1.4float	7.0	58.2	3.0	28.4	1.67	0.4	0.7	0.6	0.7	0.7	0.6
0.6x0.15	1.8sink	68.7	65.7	1.3	26.4	1.57	0.4	0.2	0.2	1.5	0.1	0.1
0.6x0.15	1.8-1.6	38.1	64.2	1.6	28.6	0.81	0.3	0.2	0.3	1.1	0.1	0.1
0.6x0.15	1.6-1.4	18.0	62.5	1.8	29.2	0.97	0.3	0.3	0.3	0.8	0.2	0.2
0.6x0.15	1.4float	6.2	57.9	3.8	28.8	1.48	0.4	0.7	0.6	0.9	0.8	0.6
1968-east	2											
10x0.6	1.8 sink	77.9	54.8	1.8	38.6	0.57	0.4	1.3	0.1	0.3	0	0.1
10x0.6	1.8-1.6	37.3	55.1	3.0	26.5	2.27	2.4	5.6	0.2	0.2	0.1	0.1
10x0.6	1.6-1.4	18.2	56.1	2.2	27.3	3.33	1.7	3.7	0.2	0.2	0.2	0.2
10x0.6	1.4float	6.5	52.1	2.5	26.2	6.19	1.4	3.1	0.7	0.3	0.9	0.7
0.6x0.15	1.6-1.4	15.4	59.5	2.6	27.3	2.78	0.9	2.2	0.3	0.3	0.3	0.3
0.6x0.15	1.4float	5.0	54.7	4.3	26.9	3.35	0.9	2.3	0.6	0.4	0.9	0.7

TABLE 3 ANALYTICAL DATA FOR SCREENED FRESH AND ACID LEACHED SAMPLES, PROPERTY SAMPLES

						daf			
			arm	adm	ΜΛ	VM 6	Ash	FC	FSI
GC1		fresh	4.9	0.7	23.8	31.7	25.0	50.5	4.0
		leached		0.3	23.4	30.2	22.7	53.6	2.0
sample	size mm		wt						
GC2	>0.6	fresh	46.5	0.6	20.6	31.7	34.9	43.9	2.5
GC3	$0.6 \ge 0.0$	fresh	53.5	0.7	25.4	30.7	17.2	56.8	5.0
GC2	>0.6	leached		0.3	20.1	31.3	35.8	43.8	1.5
GC3	$0.6 \ge 0.0$	leached		0.3	24.4	29.5	17.4	57.9	2.5
screene	d samples	fresh							
GC4	$0.6 \ge 0.0$	1.4F	64.8	1.2	27.9	29.5	5.3	65.6	5.5
GC5	$0.6 \ge 0.0$	1.4x1.6	14.1	1.4	25.8	29.7	13.2	59.6	1.0
GC6	$0.6 \ge 0.0$	1.6x1.8	7.7	1.4	24.0	34.5	30.7	44.0	0.5
GC7	$0.6 \ge 0.0$	1.8S	13.4	0.8	16.8	56.0	70.0	12.4	0.0
screene	d samples	leached							
GC4	0.6 x 0.0	1.4F		0.7	30.9	32.4	4.7	63.8	4.5
GC5	$0.6 \ge 0.0$	1.4x1.6		1.2	26.4	30.0	11.9	60.6	1.0
GC6	0.6 x 0.0	1.6x1.8		1.0	23.6	33.0	28.6	46.8	0.5
GC7	0.6 x 0.0	1.8S		0.6	13.7	47.0	70.8	14.9	0.0

arm = as-received moisture adm = air-dried moisture

F=float S=sink leached=leached in 2 molar HCl to remove carbonate

metre material washed using four SG increments (Table 3). Oxide analyses were performed on the various splits (Table 4) and X ray diffraction analysis was used to identify the major minerals present in the ash. The samples have moderately high CaO contents and X ray diffraction indicates that this is present mainly in calcite. To see if the calcite effected the rheological properties of the samples they were leached in 2 warm molar HCl and then re analyzed (Table 4).

Samples of froth floatation coal were collected to see if there was any obvious reason why it is sometimes diffi-

TABLE 4 OXIDE ANALYSIS FOR PROPERTY DATA, FRESH AND LEACHED SAMPLES

			Ash dl	o SiO ₂	2TiO2	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K_2O	P_2O_5	Ва
raw sam	ples												
GC2	>0.6mm	fresh	35.2	66.9	0.86	19.6	3.18	1.52	4.24	0.01	2.30	0.19	0.15
		leached	35.9	71.5	0.90	21.4	1.89	0.59	0.21	0.03	2.40	0.18	0.17
GC3	<0.6mm	fresh	17.3	58.5	1.16	24.6	2.36	0.90	7.04	0.01	1.66	0.39	0.29
		leached	17.4	66.5	1.26	26.8	1.63	0.51	0.32	0.15	1.89	0.38	0.33
washed	<0.6mm	samples											
GC4	1.4F	fresh	5.4	52.2	2.45	30.5	2.95	0.92	5.96	0.18	1.16	1.17	0.83
		leached	4.7	55.9	2.63	34.0	2.05	0.34	0.71	0.18	1.28	1.14	0.87
GC5	1.4-1.6	fresh	13.3	54.6	1.40	30.6	2.63	1.00	5.74	0.08	1.16	0.88	0.55
		leached	12.0	59.1	1.53	33.3	1.53	0.41	0.38	0.20	1.27	0.89	0.63
GC6	1.6-1.8	fresh	31.1	57.7	1.05	27.5	2.23	0.92	6.86	0.01	1.46	0.36	0.24
		leached	28.9	63.4	1.14	30.7	1.31	0.44	0.21	0.07	1.60	0.39	0.29
GC7	1.8S	fresh	70.6	62.5	0.74	21.6	2.21	0.85	7.83	0.01	1.96	0.10	0.10
		leached	71.2	69.3	0.82	24.4	1.79	0.53	0.32	0.07	2.14	0.09	0.12

and leached samples have 1.5-2.5% loss.

oxides that increase in % indicate no loss. They increase because there is a loss of other oxides oxides that decrease in % have been removed by leaching

TABLE 5 ANALYTICAL DATA FOR FROTH FLOTTION (FF) SAMPLES

						_				
sample	yield	adm	VM	Ash	FC					
ff feed	100	0.57	22.11	17.15	59.65					
ff clean 75.5 0.89 22.59 12.9 63.62										
ff reject	24.5	0.52	20.91	30.25	48.32					
Y=yield ca	lculated t	rom								
100*17.15	=(100-Y)	*30.25+	Y*12.9	Y=75.5%	, D					
carbon reco	overy=CF	calcula	ted from							
CR=(1-12.	9/100*1.2	2)/(1-17.	15/100*1	.2)*75.5	= 80%					
weight mir	neral matt	er/ash=1	1.2 assum	led						

cult to produce a low-ash clean product from this material. Clean, feed and reject samples were analyzed for proximate values (Table 5) and subsequently studied using a Scanning Electron Microscope.

The reflectance of a number of samples was measured. The equipment at the Geological Survey Branch has not been routinely used for some time so a number of duplicate measurements were made. Reflectance standards were checked for internal consistency and then five samples previously analyzed at another lab were analyzed (Table 6). Reflectance analyses were performed on two different microscopes (Leitz ortho plan pol and Leitz MPV3). The measurement procedure involves measuring the maximum reflectance on three standards then on 10 tellinite or telocollinite grains of the sample. The procedure is repeated 5 times to provide 50 measurement of sample grains and 18 measurements of standards. Individual reflectance measurements are corrected for time drift and scaling using the 6 sets of three standard measurements. The 1 sigma SD of the mean for all samples is less than 0.01.

The petrographic composition of some samples was estimated using a counting stage to count 300 grains. The continuous variation from telinite or telocollinite and

TABLE 6
REFLECTANCE MEASUREMENTS, MINE AND
PROPERTY AND INTERNAL AND EXTERNAL
DUPLICATES

Sample	Rmmax	bif	mic
1968-east1 1.4float	1.14	0.05	k
1968-east1 1.4float dup	1.14	0.07	g
1968-east1 1.4-1.6	1.14	0.11	k
1968-east1 1.4-1.6 dup	1.14	0.08	g
1968-east2 1.4 float	1.17	0.07	g
1968-east2 1.4-1.6	1.19	0.18	k
1968-east2 1.4-1.6 dup	1.18	0.09	g
1980-69 1.4 float	1.07	0.07	g
198- 69 1.4-1.6	1.12	0.10	g
1980-69 1.6-1.8	1.19	0.08	g
ff clean	1.01	0.07	g
GC 1.4 float	1.08	0.07	g
lab	GSB	Other	
sample 1	1.6	1.65	
2	1.59	1.58	
3	1.09	1.09	
4	1.04	1.00	
5	1.04	1.03	
dup=duplicate ff=froth f	loatation	bif=biref	lectance
mic=microscope			
g=Leitz orthoplan pd	k=Leitz	MPV3	

from semifusinite to macrinite makes it difficult to produce consistent results in terms of maceral composition. The process, despite the best efforts of the various classification schemes is very subjective. Attempts were made to differentiate between inert plus semi inert macerals and reactive macerals and to differentiate between macerals retaining some original structure (semi fusinite and tellinite) and those retaining no original structure (macrinite and collinite).

DISCUSSION

Coals may be difficult to clean for a number of reasons, but first it is important not to confuse washing difficulty with problems caused by size consist. Coals difficult to clean in wash plants have large amounts of near gravity material in a particular size range. This material can easily be miss placed with higher ash content grains ending up in the clean coal stream and lower ash content grains ending up in the reject stream. The result is a decrease in yield, an increase in wash ash content and an increase of carbon in the reject stream. Also when evaluating laboratory wash data it should be appreciated that the average ash content of particles in the specific gravity (SG) split can vary by about 10% for a 0.1 change in SG and therefore in an SG bracket from 1.4 to 1.5, the ash content of particles can range from about 18% to 28%.

Difficult to wash coals contain a large number of particles with SG values in the range of 1.4 to 1.5 and the ash content of these particles is maximized. There have been a number of papers suggesting ways of measuring washing difficulty prior to cleaning coals in a wash plant (Sanders and Brookes, 1986, and Ryan, 1992). They generally emphasize the importance of measuring the amount and ash content of near gravity material and attempt to reduce washing difficulty to a single number. This is a bit simplistic but it does provide a useful empirical scale for comparing coals.

The method introduced by Ryan (1992) uses ash analyses on head and wash samples and is therefore a simple and rapid way of estimating relative washing difficulty of samples. Data for the three in-pit samples are in Figure 1, which contains iso washing difficulty lines. The Figure clearly separates the 3 samples (1980-69, 1968-east1 and 1968-east2) based on their washing difficulty in agreement with the way they washed in the wash plant. It also indicates that finer material is more difficult to wash than coarse material. Sample 1980-69 is more difficult to wash in both size ranges than the other two samples and it contains more minus 0.6 millimetre material than the other two samples. Plant recovery will be lower when washing 1980-69 type coal because of the finer size consist and increased washing difficulty. There is a rough correlation of raw ash with washing difficulty. The difference in washing difficulty does not appear to be caused by oxidation as the three samples 1968-east1, 1968-east2 and 1980-69 all have percent transmittance values of 99%.

The oxide data for samples 1968-east2 and 1980-69 (Figure 2) can be used to illustrate possible mineral dif-



Figure 1. Washing difficulty for samples. X axis is wash ash at 1.6 SG. Y axis is calculated carbon recovery.



Figure 2. Al_2O_3/SiO_2 ratio versus ash % for samples 1968-east2 and 1980-69.

ferences between the two samples. The Al2O3/SiO2 ratios probably indicate the proportion of clays to free quartz in the sample. The ratio tends to increase at lower ash concentrations because of a tendency for vitrinite to contain more dispersed kaolinite than inertinite. Often washing difficulty is related to clay content. In this case the difficult to wash sample 1968-69 appears to show more of a tendency for the ratio to increase as ash decreases than the easier to wash sample 1980-east2 (Figure 2) but there is not a marked difference. Varying clay contents probably do not fully explain the difference in washing difficulty.

Calcium oxide concentrations in the property samples are quite high and are substantially reduced by acid leaching (Table 4). This indicates the presence of a carbonate, which was confirmed using XRD (Table 7). It is possible to estimate the amount of carbonate in total or in the ash component of samples by assuming, either that all the Ca, Fe and Mg is in carbonate (maximum estimate) or that only the Ca is in carbonate (minimum estimate). This is done by calculating the elemental content of Ca, Mg and Fe and then recalculating the molar content of these elements as X,CO3 either in the total sample or in the ash (Table 8, 9 and 10). Obviously this assumes that all the Ca is in carbonate when in fact some is combined with phosphorus in apatite and also that all the Fe is in carbonates where as a small proportion is combined with sulphur in pyrite. The carbonate and calcite concentrations for the three samples (property sample, 1968-east2 and 1980-69)

TABLE 7 X-RAY DIFFRACTION RESULTS FOR 2 PROPERTY SAMPLES

sample		Ash	Minerals detected
GC-2	>0.6 mm	37.7	abundant quartz, moderate kaolinite
			minor mixed clays moderate calcite and dolomite
GC-7	<0.6mm 1.8S	75.6	abundant quartz, moderate kaolinite significant calcite some gypsum

TABLE 8 CUMULATIVE OXIDE AND CARBONATE CALCULATIONS FOR THE PROPERTY

	plus0.6mm	minus0.6mm	float1.4	1.4x1.6	1.6x1.8	1.8sink
ash	35.15	17.26	5.33	9.62	17.54	33.43
SiO ₂	66.90	58.50	52.20	52.63	53.08	54.34
Al ₂ O ₃	19.60	24.60	30.50	30.52	30.25	29.09
Fe ₂ O ₃	3.18	2.36	2.95	2.89	2.83	2.75
TiO ₂	0.86	1.16	2.45	2.26	2.15	1.97
P_2O_5	0.19	0.39	1.17	1.12	1.05	0.92
CaO	4.24	7.04	5.96	5.92	6.00	6.25
MgO	1.52	0.90	0.92	0.93	0.93	0.92
MnO	0.01	0.01	0.01	0.01	0.01	0.01
Na ₂ O	0.01	0.01	0.18	0.16	0.15	0.13
K ₂ O	2.30	1.66	1.16	1.16	1.19	1.29
BaO	0.15	0.29	0.83	0.78	0.73	0.65
LOI	5.03	5.28	8.06	7.01	5.86	5.80
B/A	0.17	0.21	0.23	0.23	0.22	0.22
alkalinity	0.06	0.04	0.01	0.02	0.04	0.07
wt% CB TS	5.23	3.03	0.89	1.59	2.89	5.52
wt% C TS	2.66	2.17	0.57	1.02	1.88	3.73
CO2 addition	2.37	1.35	0.39	0.70	1.28	2.48
mm/ash	1.07	1.08	1.07	1.07	1.07	1.07
wt% C in ash	7.57	12.56	10.64	10.57	10.72	11.15
wt% CB in ash	15.36	17.87	16.84	16.72	16.78	17.07

LOI = loss on ignition B/A = base acid ratio Aller limits = D/A actis = acid %

Alkalinity = B/A ratio x ash%

CB = total carbonates C = calcite TS = total sample

are estimated using the oxide data calculated for cumulative rather than incremental wash samples. The first two samples (property sample and 1968-east2), which wash easily have high carbonate contents where as 1980-69, which does not wash easily has a low content.

Carbonate in the property and 1968-east2 samples appears to be concentrated in the ash (Figure 3). Contents calculated on a total sample basis increase as ash increases and contents calculated on an ash base are relatively constant at about 15% carbonate in the ash. Under the microscope there does not appear to be much calcite associated with the coal and most found occurs as discrete grains (Photo 1). In general it is difficult to locate the amount of carbonate indicated in the oxide analysis and it must be finely dispersed in the ash. In sample 1980-69 carbonate contents are low and when calculated on an ash base tend to increase as ash decreases indicating in part a coal association for the carbonate material (Figure 3). Carbonate in this sample occurs as small blebs in desmocollinite (Photo 2).

TABLE 9CALCULATED CUMULATIVE OXIDE DATA AND
CARBONATE CONTENTS FOR 1968-EAST 2

	10x0.6 mm 1.8 sink	10x.6 1.8-1.6	10x0.6mm 1.6-1.4	10x0.6mm 1.4float	0.6x0.15mm 1.6-1.4	0.6x0.15mm 1.4 float
ash	20.3	11.9	10.3	6.5	8.2	5.0
SiO ₂	53.7	53.5	53.4	52.1	56.2	54.7
Al ₂ O ₃	28.1	26.5	26.5	26.2	27.0	26.9
Fe ₂ O ₃	4.51	5.09	5.27	6.19	3.17	3.35
TiO ₂	2.38	2.47	2.44	2.53	3.80	4.32
P ₂ O ₅	0.59	0.67	0.71	0.93	0.68	0.87
CaO	3.16	3.43	3.29	3.10	2.30	2.34
MgO	1.40	1.55	1.49	1.41	0.89	0.88
MnO	0.01	0.01	0.01	0.01	0.01	0.01
Na ₂ O	0.44	0.49	0.51	0.67	0.54	0.64
K ₂ O	0.23	0.23	0.23	0.25	0.34	0.37
BaO	0.45	0.50	0.53	0.68	0.57	0.72
LOI	5.03	5.28	8.06	7.01	5.86	5.80
B/A	0.19	0.17	0.21	0.24	0.14	0.15
alkalinity	0.039	0.021	0.022	0.015	0.011	0.008
wt% CB TS	3.02	1.98	1.69	1.13	0.86	0.54
wt% calcite TS	1.14	0.73	0.60	0.36	0.34	0.21
CO ₂ addition	1.32	0.86	0.73	0.48	0.37	0.23
mm/ash	1.06	1.07	1.07	1.07	1.05	1.05
ash	20.3	11.9	10.3	6.5	8.2	5.0
wt% C in ash	5.60	6.09	5.85	5.52	4.09	4.17
wt% CB in ash	15.13	16.74	16.64	17.46	10.57	10.88

TABLE 10 CALCULATED CUMULATIVE OXIDE DATA AND CARBONATE CONTENTS FOR 1980-69

	10x0.6mm 1.8sink	10x0.6mm 1.8-1.6	10x0.6mm 1.6-1.4	10x0.6mm 1.4 float	0.6x0.15mm 1.8 sink	0.6x0.15mm 1.8-1.6	0.6x0.15mm 1.6-1.4	0.6x0.1mm 1.4 float
ash	44.6	21.0	11.4	7.0	26.1	13.8	10.1	6.2
SiO ₂	64.9	61.8	60.3	58.2	61.3	60.1	59.5	57.9
Al ₂ O ₃	26.1	28.5	25.3	28.4	28.3	28.9	28.9	28.8
Fe ₂ O ₃	1.12	1.18	0.92	1.67	1.32	1.24	1.31	1.48
TiO ₂	1.83	2.29	1.42	2.96	2.53	2.89	3.08	3.75
P_2O_5	0.26	0.38	0.09	0.72	0.41	0.50	0.57	0.76
CaO	0.36	0.42	0.23	0.70	0.45	0.53	0.58	0.73
MgO	0.42	0.34	0.41	0.43	0.36	0.36	0.37	0.41
MnO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Na ₂ O	0.27	0.41	0.14	0.64	0.43	0.50	0.53	0.63
K_2O	1.24	0.73	1.42	0.74	1.06	0.93	0.90	0.93
BaO	0.22	0.34	0.08	0.60	0.33	0.40	0.45	0.58
LOI	5.03	5.28	8.060	7.010	5.860	5.80	5.03	5.03
B/A	0.06	0.04	0.053	0.082	0.065	0.07	0.07	0.08
alkalinity	0.02	0.01	0.006	0.006	0.017	0.01	0.01	0.01
wt% CB TS	1.38	0.66	0.296	0.318	0.901	0.48	0.37	0.44
wt% C TS	0.28	0.16	0.047	0.087	0.211	0.13	0.10	0.13
CO ₂ addition	0.60	0.28	0.130	0.135	0.385	0.20	0.16	0.19
mm/ash	1.01	1.01	1.011	1.019	1.015	1.01	1.02	1.03
ash	44.6	21.0	11.4	7.0	26.1	13.8	10.1	6.2
wt% C in ash	0.64	0.74	0.416	1.249	0.809	0.94	1.03	2.14
wt% CB in ash	3.13	3.16	2.608	4.571	3.471	3.49	3.70	7.07





Photo 1. Calcite in property sample; field of view is about 0.25 millimetres.

Figure 3. Carbonate (CB) versus ash % for mine samples 1980-east2, 1968-69 and property sample. Data presented on an ash basis and on a total sample (TS) basis.



Photo 2. Calcite in 1980-69 occurring as small blebs in desmiocollinite; field of view 0.25 mm.

If the carbonate material is associated with the ash, then this will tend to increase the SG of ash and make it easier to remove. When the carbonate material is associated with the coal, for example occurring as blebs in desmocollinite or on cleats, it will tend to be washed out attached to coal fragments in intermediate SG increments. This will cause loss of coal and deteriorate washing characteristics. Part of the reason for the increased washing difficulty of sample 180-69 may be small amounts of carbonate (calcite) associated with the coal.

The mode of occurrence of carbonate minerals in samples has important implications for coke quality as influenced by base/acid ratio and alkalinity (base/acid ratio times ash content). When carbonate is in the ash base/acid ratios remain constant but alkalinity decreases as ash is removed from the sample. This means that properties such as CSR (coke strength after reaction) improve markedly as a coal is cleaned. On the other hand if the carbonate is associated with the coal part of the sample either as cleat filling or impregnating one of the macerals then as ash is removed carbonate content increases and both



Figure 4. Alkalinity versus ash % for mine samples 1968-east2, 1980-69. and property sample.

TABLE 11 LEACHING RESULTS (WARM 2 M HCI), PROPERTY SAMPLES

	fresh	leach	loss	fresh	leach	loss	fresh	leach	loss
	Fe ₂ O ₃	Fe ₂ O ₃	%	MgO	MgO	%	CaO	CaO	%
>0.6mm	3.18	1.89	40.5	1.52	0.59	61.1	4.24	0.21	95.0
<0.6mm	2.36	1.63	31.1	0.90	0.51	43.3	7.04	0.32	95.5
1.4 Float	2.95	2.05	30.4	0.92	0.34	63.4	5.96	0.71	88.1
1.4-1.6	2.63	1.53	42.1	1.00	0.41	59.3	5.74	0.38	93.4
1.6-1.8	2.23	1.31	41.3	0.92	0.44	52.7	6.86	0.21	96.9
1.8 Sink	2.21	1.79	19.0	0.85	0.53	37.8	7.83	0.32	96.0
Average	%		34.1			52.9			94.2

base/acid ratio and alkalinity increase. This causes CSR either to deteriorate or not improve as much as expected based on reducing the ash concentration. For the three samples discussed here, alkalinity decreases markedly at low ash for the property and 1968-east2 samples but is only moderately effected by decreasing the ash content in sample 1980-69 (Figure 4).

The presence of carbonate minerals in the property sample was also confirmed by leaching samples in warm 2 molar HCl. The oxide analyses before and after the leaching (Tables 4 and 11) indicate removal of 95% of CaO, 55% of MgO and 35% of Fe2O3. The carbonate contains some iron and magnesium though leaching does not remove all the Fe and Mg indicating the presence of non carbonate hosts for these elements. About 5% of the Ca remains in the samples probably associated with apatite.

The property coal has unexpected variations in quality that do not appear to be related to oxidation. The FSI values for the fresh and leached samples were measured to see if high carbonate content effects FSI (Table 3). In most cases, leaching out the carbonate reduced the FSI (Figure 5). The leaching was performed using dilute warm HCl and it is assumed that this alone did not deteriorate FSI. If this is the case, then possibly the CO2 emitted when the FSI sample is heated to a temperature of 820(C and the carbonate breaks down, helps maintain the FSI



Figure 5. FSI versus ash % adb for property samples before and after leaching in warm dilute HCl.

button. Certainly in most cases the volatile matter content decreased after leaching and removal of the carbonate. If the FSI values are artificially high, then this could lead to an initial deceptively good estimation of coking quality.

Despite the intense shearing, the property sample appears to wash very well (Figure 1) and the main problem with the bulk sample is one of size consist. A very high proportion of the coal is minus 0.6 millimetres. The apparent low FSI of two of the wash samples may be caused by clay coating fine coal particles. An effect apparently seen in the SEM work and discussed later.

Bustin (1982) investigated the effect of shearing on coal washability. In part he concluded that the main effect was to increase the raw ash content of a seam by mixing hanging and foot wall material into the seam and generally making the ash more difficult to remove. Data for the sheared and non sheared samples of the two seams studied by Bustin are plotted in Figure 1. In one case shearing has increased washing difficulty and in the other it appears to have had little effect. Shearing has had the effect of increasing the ash content of the material in each SG increment. This may be because shearing has decreased coal density by introducing micro fractures and therefore allowed more ash to be attached to each coal particle within an SG increment. Alternatively shearing may have skewed the distribution of particles in each SG increment towards the high SG end. In general the main effects of shearing are to incorporate extra ash into the seam and decrease the over all size consist.

SCANNING ELECTRON MICROSCOPE STUDIES AND PETROGRAPHY OF SAMPLES

Feed, clean and reject froth floatation samples were studied using a petrographic microscope and a scanning electron microscope (SEM). Generally the froth floatation feed (0.15×0.0 mm material) can be washed to less than 10% ash in froth floatation circuits. Some mine froth floatation feed washes to a significantly higher ash, in this case about 13% (Table 5). Based on the feed, wash and reject ash contents the yield from the circuit is estimated to be about 75.5% with about an 80% carbon recovery. The circuit is not loosing a significant amount of coal but the coal floating is still associated with a moderate amount of ash.

Generally there is very little well preserved semifusinite seen in the samples when studied using a reflecting microscope. The semifusinite that does occur has very flattened cell structure nearly always mineralized with kaolinite. A lot of the mineral matter occurs as fine grains of quartz or kaolinite coating coal grains. The total reactives content (vitrinite macerals only) (Table 12) on a mineral matter free basis is slightly higher for the difficult to wash sample 1980-69. This is mainly because of an increase in detrovitrinite in the high ash vitrain lithotype. The total reactives contents for these samples range from 52% to 77%. The property samples have higher reactive maceral contents than the mine samples.

TABLE 12 ESTIMATED PETROGRAPHY OF SOME SAMPLES. MINERAL MATTER FREE BASIS

sample	liptinite	telinite	telo collinite	desmo collinite	detro virinite	semi fusinite	fusinite	macrinite	micrinite	inerto detrinite	total reactives	total inerts
ff feed	3	9	24	24	4	10	0	17	0	9	64	36
ff clean	3	7	23	31	7	6	1	16	0	6	70	30
ff reject	3	8	17	35	5	12	1	10	0	9	68	32
1968-east1 1.4-F	6	7	16	21	2	18	0	23	0	7	52	48
1968-east 1 1.4-1.6	8	7	10	24	4	21	0	18	0	8	53	47
1968-east 2 1.4-F	2	15	13	33	0	15	0	16	0	6	63	37
1968-east 2 1.4-1.6	6	11	7	31	2	12	0	24	0	6	58	42
1980-69 10x.6 1.4-F	4	7	21	26	0	19	0	16	0	6	59	41
1980-69 1.4-1.6	7	7	13	25	25	10	0	6	0	7	77	23
GC grab	4	0	28	39	5	8	0	11	0	5	76	24
GC 1.4-F	4	11	29	30	1	9	0	10	1	4	76	24

The fine grain size and continuous variation between vitrinite and semifusinite makes it difficult to get reproducible petrographic data for the samples. There is considerable subjective judgment in separating semifusinite from telinite and macrinite from semifusinite. Some small vitrinite grains were observed in the petrographic microscope and SEM to have high reflecting or high atomic density rims characteristic of high temperature oxidation (Photo 3). It is unlikely that the rims were formed by the pellet making process because the rims were also found in a pellet was made at lower temperature. The grains were seen in feed and wash samples so the effect is not related to froth floatation. Most of the grains were small and contained complete high reflecting rims indicating that either only small grains were effected or the effect post dates tectonic shearing or crushing at the



Photo 3. High reflecting rim to vitrinite grain; field of view 0.25 mm.



Photo 4. Apparent vesicle in altered vitrinite grain; field of view 0.25 mm.



Photo 5. Fine kaolinite coating to coal grains; scanning electron microscope image.

plant. Some of the grains appeared to be partially coked and to contain vesicles (Photo 4). The origin of these grains is not clear; they may have been altered by fires associated with underground mining that occurred prior to 1950. If the coal contained a lot of these grains it would increase the inert content of the coal and probably decrease its coking ability.

In the SEM it appears that many coal grains are coated by very fine kaolinite flakes (Photo 5). Brecciation of semifusinite, which contains kaolinite cell filling, appears to release fine kaolinite, that coats coal fragments making them hard to separate in froth floatation and increasing their ash contents. This also makes it difficult to pelletize the samples because kaolinite also coats the transoptic grains and they do not fuse very well. Semifusinite is more brittle than the reactive macerals and more likely to respond to shearing by brecciation rather than flow. There is no indication that the coal has a high ash content. It appears that shearing has dispersed the kaolinite. This means that whereas before shearing a few large fragments rich in kaolinite would be removed from the clean coal, after shearing kaolinite is finely dispersed partially coating coal grains that may remain with the clean coal. If kaolinite completely coats coal grains, then the effect would be to decrease yield and have marginal effect on clean coal ash. If kaolinite partially coats coal grains, then they may still be recovered with clean coal and the effect is to increase clean coal ash and have marginal effect on yield. The dispersal of the kaolinite associated with tectonic shearing obviously predates the coal entering the wash plant. The coating of the grains with kaolinite may occur before or during crushing, screening and washing the coal.

Generally the mineral matter in semifusinite is predominately kaolinite. When a seam rich in inertinite is deformed there is an increased release of fine kaolinite which is subsequently very difficult to wash out because it partially coats coal particles and ends up in with the clean coal. The average raw ash content of the seam does not change. The liberation of kaolinite, from mineralized semifusinite, that would otherwise be removed in the washing process appears to increase the amount of difficult to remove ash and to increase the wash ash content of the froth product by about 3%.

WASHING DIFFICULTY AND PETROGRAPHY

Washing difficulty increases the more the coal macerals are intermixed with mineral matter. There are a number of ways mineral matter can be included in macerals, but they all relate to processes that occurred in the coal swamp or during diagenesis. The formation of semifusinite and fusinite by fire leaves the cells intact and empty. They are often later filled by mineral matter during diagenesis. In fact, if the cells in semifusinite are not filled, then they will probably be collapsed later when the coal seam is deformed. Fusinite is probably stronger and more likely to survive with or without mineralized cells. The presence of semifusinite or fusinite therefore implies a certain amount of inherent ash, usually kaolinite. Massive vitrinite (telinite or telocollinite) generally does not contain much mineral matter, though sometimes cells in telinite are mineralized with kaolinite. Desmocollinite indicates a moderate amount of "soft sediment deformation" in the coal seam and as it forms, fragments of inert macerals and mineral matter may be included in a vitrinite ground mass. This is apparent in sample 1980-69, which has a lot of heavily mineralized desmocollinite, that also contains a lot of detrovitrinite. Fine mudstone bands may also be incorporated into the macerals. The amount of clay material entering the swamp and the amount of soft sediment deformation during diagenesis influences the amount of near gravity material formed (Ryan, 1996).

Early deformation in coal seams is apparent from the rounding and compaction around macrinite grains embedded in vitrinite (Photo 6). The cell structure in some semifusinite grains is deformed and appears to have experienced simple shear (Photo 7). Higher reflecting



Photo 6. Rounding and compaction around a macrinite grain; field of view 0.25 mm.



Photo 7. Deformation of semifusinite; field of view 0.25 mm.

semifusinite generally deforms brittlely breaking into shards that may be dispersed as inertodetrinite in desmocollinite.

Tectonic shearing produces brittle deformation and therefore reduces the size consist but does not produce middlings material. However it does appear to be selective in how it breaks fragments in the coal seam producing more very fine telinite particles and possibly releasing kaolinite from semifusinite. Shearing and dispersing fine clay bands through the coal seam can increase the washing difficulty and wash ash content with out increasing the amount of ash in the seam.

VITRINITE REFLECTANCE

The Rmmax values of the washed splits of the three mine pit samples and the froth floatation samples were measured (Table 6). The reflectance increases with washing ease for the three pit samples but there is no obvious reason why rank should effect washing difficulty. The rank and bireflectance also tend to increase as the SG of the wash increment increases. At higher SG values the vitrinite appears more likely to contain finely dispersed micrinite, which increases its SG and reflectance. This effect is probably restricted to coals of intermediate rank, in which micrinite is just starting to form.

CONCLUSIONS

Many washing problems are caused by excessive amount of fines. Though mining methods can reduce fines generation a highly sheared coal will generate fines how ever it is handled. It is probably possible to estimate fines potential by looking at coal faces in the pit or to design a simple in pit attrition test. The as-received water content of samples may also be an indication of the friability of the coal. In that the more fractured the coal the more surface area available for wetting.

Coals are difficult to wash in any size consist when there is an excessive amount of near gravity material. This can originate from processes in the coal swamp and during diagenesis of the coal or from shearing. Turbulence and introduction of clay into the coal swamp can result in a lot of mineralized desmocollinite as in sample 1980-69. A combination of fires to produce semifusinite and fusinite in the coal swamp and diagenetic in filling of cells with kaolinite can increase the mineral matter content of the inert macerals, which tend to concentrate in the intermediate SG increments.

Shearing can increase washing difficulty. It appears to do it by generating fine kaolinite probably in part by breaking mineralized semifusinite grains and releasing the kaolinite contained in the cells. This material then coats vitrinite grains making them difficult to float and increasing their ash content. Shearing can also increase the raw ash content of a seam by incorporating hangingwall or footwall rock into the seam.

The association and amount of carbonate in coals can effect washing difficulty. If the carbonate is associated with ash it will probably help with ash removal. If it is associated with the coal it will cause loss of coal in intermediate SG increments and in crease the ash and Ca content of the clean coal.

It is possible using ash chemistry data to estimate the amount and location of carbonate minerals in the coal. In the easy to wash samples from the property and 1968-east2, the carbonate mineral is calcite or dolomite and is associated with the ash making up about 16% of the ash. In that the carbonate correlates with the ash and not the total sample it is probably not related to fracturing associated with faulting. Its amount may relate to environmental conditions in the coal swamp, which also influence the amount of inherent ash.

The difficult to wash sample (1980-69) contains less carbonate material, which is partially associated with the coal and makes up about 3% of the sample. It might be that for these coals there is a correlation between the amount of carbonate and ease of washing in which case a simple acid test for the presence of carbonate in the coal may indicate whether the coal will be easy or difficult to wash.

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