Producing Clean Coal from Western Canadian Coalfields Using the Water-Based Roben Jig Process: Refining the Process

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Introduction

There are a number of coalfields in British Columbia (BC): several thermal coalfields and two major metallurgical coalfields, the Kootenay and Peace River (Figure 1). Metallurgical coals are destined mainly for use in commercial coke ovens to produce coke for use in blast furnaces in steelworks.

One of the main challenges after finding and identifying coal seams is evaluating the quality of the coal resource during the exploration stage. Understanding coal quality can be a complex process and is key to a sound economic evaluation of the resource. During the exploration phase of coal-mine development, evaluation of metallurgical-coal quality is often done using samples collected from drillcore. Although many coal seams outcrop, the bulk of the coal deposit is generally deep underground. Coal near the surface can be sampled using test pits or adits, but drilling is the method most often used to obtain representative coal-seam samples. During the feasibility stage of an exploration property, a bulk sample of more than a tonne is required for testing the coal in a pilot-scale coke oven to fully understand the coking potential of the coal. Sufficient sample can be obtained using several 6-inch drill-program cores, although this is costly, particularly for thinner seams.

Coal samples collected during exploration are prepared by screening and then lab-scale or pilot-scale washing that simulates the coal behaviour in commercial coking coal-wash plants. The coarser sized coal is processed using mixtures of organic liquids and the finer fraction is cleaned by a process called froth flotation. The quality of the coal produced by these smaller scale washing methods is critical to understand the market potential of the coal. These processes must produce the same quality coal as a commercial plant.

Figure 1. Location of coalfields in southeastern British Columbia from which the coal samples used in this study originated.

On the lab scale, the float-and-sink procedure (Figure 2) is used to separate coal from dirt, rock and mineral matter using a density separation, the same process used in commercial plants. The lower density solutions tend to float mainly the coal. During the float-and-sink process, the coal sample is separated at relative densities (specific gravities, sg) between roughly 1.40 and 1.80 using tanks of organic mixtures made from white spirit (1.40 sg), perchloroethylene (PCE; 1.60 sg) and methylene bromide (1.80 sg; ASTM

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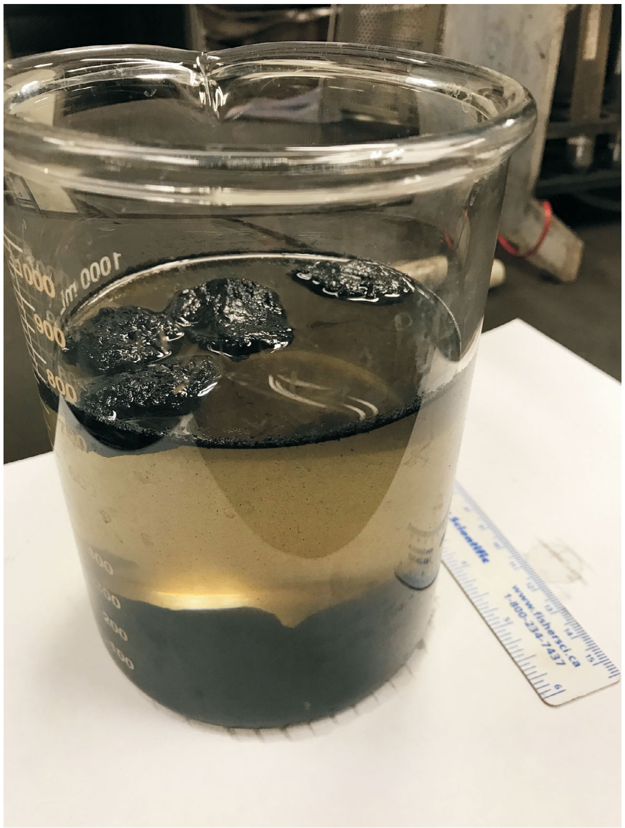

Figure 2. Coal particles floating in perchloroethylene.

D4371-06, 2012). This produces clean-coal samples at the target ash, sulphur and calorific content typical of what would be produced in a commercial coal-washing plant.

Commercial plants separate the coal into size fractions that are processed in equipment that separates the coal from waste (rock, dirt and minerals) using differences in density—coal being less dense than the waste. The equipment uses water-magnetite mixtures of controlled density in cyclones and baths, centrifugal force for coal-water mixtures in cyclones, and relative settling rates of the coal particles of differing densities in water to isolate/separate the 'clean' coal in jigs and settling tanks. The finest sizes are treated by water-based froth flotation, which can 'float' the coal from the waste. Exploration samples are treated/cleaned in a similar fashion.

Figure 3. Operator working with organic liquids in a specially designed fume hood.

Project economics are based on the results of the float-and-sink testing, which produces information on the yield of clean coal as well as the quality of the cleaned coal and resulting coke quality. The coking characteristics, in particular, for a metallurgical coal deposit are critical in evaluating project economics (i.e., expected price for the clean coal). It is important to ensure that coal/coking properties are correctly assessed from drillcore samples to properly evaluate project economics.

Background

For years, a major concern in the handling and use of organic liquids such as perchloroethylene (PCE) was the safety risks associated with human exposure. Perchloroethylene is a known carcinogen and poses a safety hazard for laboratory operators, so it must be handled carefully. Figure 3 shows a laboratory technician working in a specially designed fume hood wearing personal protective equipment, including a respirator mask.

In addition to the health issues, there are increasing concerns about whether the solvents impact the quality of coking coal. Many Canadian geologists have found that cleaned drillcore coal samples often had lower caking/coking properties than bulk or production coal samples, an observation that goes back many decades. A number of investigations looked at how PCE and other organic solvents may impact the coking quality of coal samples, including Australian and American work (DuBroff et al., 1985; Campbell, 2010; Iveson and Galvin, 2010, 2012). These studies found that there were different impacts depending on the quality characteristics of the coal being assessed. Coals similar to the western Canadian coking coals (higher inert, lower thermal rheological coals) appeared to have been negatively impacted.

Based on these observations, the Canadian Carbonization Research Association (CCRA) undertook a preliminary program to investigate the impact of the organic solvents used in float-and-sink procedures on the coal and coke properties of a higher inert, low-fluidity western Canadian coal sample (Holuszko et al., 2017). This study looked at the effects of perchloroethylene on coal rheology and coke quality. It was found that an 80% decrease (relative to the control sample) in Gieseler maximum fluidity occurred in the perchloroethylene-treated coal immediately following treatment. The coke resulting from the treated sample showed a 16-point decrease in coke strength after reaction (CSR) when compared to the control sample. These two coal- and coke-quality parameters are key when evaluating coal resources and reserves. The ramifications of using the wrong numbers for these parameters when determining the characteristics of product for sale are severe and could result in unwarranted project abandonment or false overvaluing of the property.

After the initial study outlined above, the CCRA also completed an exploratory study that examined an alternative to organic liquids by washing coal samples in a jig. A lab-scale Roben Jig (Figures 4, 5) was used to clean several coals using only water, and the resulting quality characteristics of the clean coal and its coke were compared to those of coal that was processed using the traditional process of washing with organic chemicals.

It was found that it was possible to produce a clean-coal product with quality properties very similar to those obtained using the organic liquids. The Roben Jig–cleaned coals had the same/similar results for coal-quality parameters and better results for coal-rheology parameters. These findings are important because they demonstrate that the Roben Jig can be used to produce clean-coal composites similar to those obtained from traditional float-and-sink methods.

Objectives

The objective of this project was to revise the existing operating methodology for the Roben Jig in order to minimize misplaced material (Mackay et al., 2018). Another goal was to answer the following questions:

- At what apparent relative density is misplacement of coal particles occurring?
- What are the characteristics of the misplaced coal particles?
- What is the preferred method of operating the Roben Jig?
- How does the Roben Jig compare to an industrial processing plant?
- How many coal types need to be tested so that results are statistically significant?
- How is the perchloroethylene interacting and affecting the coal chemistry to cause a reduction in rheology and an increase in Hardgrove Grindability Index?

If successful, this project would benefit the coal industry by eliminating of use of PCEs and other organic liquids in the production of small-mass exploration samples for the determination of coal- and coke-quality parameters, and reduce the exposure of lab technicians/operators to carcinogenic organic liquids.

Figure 4. Roben Jig equipment used in this study.

Figure 5. Inverted Roben Jig with coal slice to be removed.

Experimental Washing Methodology

The research group devised two Roben Jig methodologies that could yield products with lower ash content while minimizing misplaced coal and rock particles. These methodologies were compared to the original coal-washing methodologies from the Phase 1 research (Mackay et al., 2018). The clean coals from all processes were then compared to the product from an industrial coal-washing plant.

The coarse coal particles in each sample (greater than 0.50 mm) were washed during this study in several different ways:

- Raw coal was washed in an industrial coal-washing plant.
- Raw coal was segregated into one coarse fraction (12.5 × 0.5 mm) and washed in organic liquids using the float-and-sink method and following the ASTM D4371 standard (Phase 1 Method: Float-and-Sink, One Coarse Fraction)
- Raw coal was segregated into one coarse fraction (12.5 × 0.5 mm) and washed in the Roben Jig (Phase 1 Method: Roben Jig, One Coarse Fraction)
- Clean coal resulting from the jigging of one coarse fraction was then rejigged (New Method A: Re-Jigging).
- Raw coal was segregated into two coarse fractions (12.5 × 2 mm and 2 × 0.5 mm) and washed in organic liquids using the float-and-sink method and following the ASTM D4371 standard (New Method B: Float-and-Sink, Two Coarse Fractions).
- Raw coal was segregated into two coarse fractions (12.5 × 2 mm and 2 × 0.5 mm) and washed using the Roben Jig (New Method C: Roben Jig, Two Coarse Fractions).

Common to all methodologies, the fine coal (particle sizes of less than 0.5 mm) was washed using the froth flotation method (ASTM D5114-90(2010)). The clean coal resulting from this method was recombined with the coarser coal (greater than 0.5 mm) when creating clean-coal composite samples.

Industrial Coal-Washing Plant Method

This research project had a unique opportunity to piggyback on a single-seam run at an operating industrial-processing plant at a mine in southeastern BC. Mine operations seldom schedule ‘single-seam’ runs unless it is part of the natural release of coal from the mine plan; it is more usual to see many seams being processed through the plant together. The ability to be able to compare laboratory-washed coal to that cleaned in an industrial-sized wash plant is considered the gold standard in validating a laboratory method for washing coal. As much as possible, companies want to be able to predict the actual clean-coal product that will be delivered from a mine’s washing plant. As the single seam was being run through the plant, raw coal was collected from the ‘feed’ side of the plant. This coal was used in the laboratory processing by organic liquids and the Roben Jig. Clean coal was also collected from the plant and analyzed for clean-coal quality and coke quality. Industrial-sized processing plants do not achieve perfect separation, and material is misplaced. A simple float-and-sink using organic liquids was also done to determine where the misplaced particles originated.

Phase 1 Method: Float-and-Sink, One Coarse Fraction

The specific gravity of a coal particle is dependent on mineral-matter content and maceral composition. Coal particles containing the lowest amount of mineral matter will float when separated in a 1.30 sg liquid, whereas those with the highest mineral-matter content are separated at 1.80 sg.

The float-and-sink method (ASTM D4371-06, 2012) was used in this project. This technique fractionates coal and mineral-matter particles based on particle density by allowing particles to settle in organic-liquid mixtures with known specific gravities. Mixtures of white spirits, perchloroethylene and methylene bromide are used to produce media densities ranging from 1.30 sg to 1.80 sg.

Phase 1 Method: Roben Jig, One Coarse Fraction

The Roben Jig is a device that enables the sorting of coal particles based on density to occur as the coal is jigged up and down in a column of water. Although no published standard (ASTM, ISO or Australian) exists for the use of the Roben Jig, the following procedure was developed by the inventor. Approximately 15 kg of 12.5 mm × 0.25 mm coal and tracers (glass marbles) of a known specific gravity (2.70) were added to the jig tube with a 0.25 mm screen at the bottom. This mesh base allowed water to enter during the jig downstroke and particles to be sorted during the jig upstroke. This tube, with coal added, was gently lowered into the jig vessel. Water level was adjusted so that it was approximately 100 mm above the level of the coal. The jig tube was attached to the pneumatic jigging mechanism. Once turned on, this mechanism moved the jig tube up and down. The down stroke was rapid to suspend particles individually, the upstroke was slower to allow the particles to sort according to density. The jigging time was 15 minutes. When the jig cycle was complete, the coal sample was presumed to have been sorted into a density continuum column, heaviest material (discard) at the bottom grading to lightest (best) coal at the top.

After jigging was completed, the jigging tube was lifted from the jig vessel, allowing the water to drain from the coal. A sample pusher was inserted in the jig tube and pressed to allow more water to drain. The entire tube was then inverted to allow the coal to be pushed upward. Once the jig tube was inverted, and the screen removed, the marbles were visible, as they had the highest specific gravity; this was evidence that the jigging was successful. A tray was attached to the top of the tube and the sample pusher was rotated, causing the coal to be pushed above the jig tube and allowing the operator to scrape off the layer. The layer was then carefully scraped into the apparent relative density (ARD) basket. Note that, because the jig tube was inverted after jigging, the first fraction collected was the highest density (heaviest or highest ash content). The thickness of the layers was dictated by the particle-size distribution of the coal and by how many fractions one expected to remove from the sorted column. Since the wet ARDs were calculated immediately, the depth of the layers could be increased or decreased to obtain a range of ARDs and subsequent range of ash contents.

Each wet coal layer was weighed and air dried, and a dry ARD calculated. Samples were then prepped for laboratory testing. Similar ARDs were added together before prepping or tested first to confirm ash results. The calculated ARD is an average for that layer.

New Method A: Re-Jigging

In this method, the clean coal from the coarse size fraction was re-jigged to see if it was possible to further segregate the particles based on density. First, a slice was taken that had an average ARD of 1.29 and an ash value of 10.20%. The slice was added back into the Roben Jig and processed. Through this ‘re-jigging’ action, it was possible to further clean the coal by removing 158 g of a higher ash (22.77%), higher ARD (1.39) coal; this allowed for a selection of a lower ash product <7%) compared to the starting value of 10.20%. This method showed promise for being effective at cleaning coal to lower ash cut points. In this study, the clean coal resulting from jigging the coarse fraction (12.5 × 0.5 mm) was added back into the empty jig and re-jigged.

New Method B: Float-and-Sink, Two Size Fractions

The coarse coal was first divided into two size fractions (12.5 × 5 mm and 2 × 0.5 mm) instead of one (12.5 × 0.5 mm) before being washed using the float-sink method (ASTM D4371-06, 2012). It is common in the coal industry to wash different coarse fractions separately in organic liquids.

New Method C: Roben Jig, Two Size Fractions

Pielot (2010) studied the results of various widths of grain-size classes being fed to jigs and found that the narrower the grain-size classes of the coal feed, the more precise was the jigging. To test Pielot’s theory, this method separated the coarse coal into size classes, each of which was jigged on its own.

Analysis

Clean-Coal Analysis

Each clean-coal composite was analyzed for various quality characteristics and was coked in the sole-heated oven (12 kg capacity) at CanmetENERGY in Ottawa, with coke characteristics subsequently being quantified. The clean coal resulting from the industrial-scale coal-washing plant was carbonized in the Carbolite pilot coke oven. The clean-coal composites were analyzed at GWIL Industries–Birtley Coal & Minerals Testing Laboratory for yield (percent), proximate analysis, free swelling index (FSI), specific gravity (sg), total sulphur, Hardgrove Grindability Index (HGI), calorific value (kcal/kg), mercury, ultimate analysis, mineral analyses of the ash, phosphorus in coal (calculated, percent), Gieseler maximum fluidity, Ruhr dilatation, ash fusion (oxidizing and reducing), chlorine, fluorine, alkali extraction–light transmittance test, Sapozhnikov X and Y indices, and caking index (G). Petrographic analysis of the coal and coke was carried out at both CanmetENERGY (Ottawa, ON) and David E. Pearson & Associates (Victoria, BC).

Carbonization

Coal samples (~20 kg each) from the float-and-sink washing with organic liquids and the Roben Jig washing with water were received at CanmetENERGY in Ottawa on August 7 and 10, 2018. In the case of 100% Plant Clean #183147, a significantly larger quantity of approximately 450 kg (three 45-gallon drums full) was received for carbonization tests in both the small-scale sole-heated oven (12 kg) and the pilot-scale coke oven (340 kg).

Upon reception, coals were air dried in open air in the laboratory for 12 hours (24 hours in the case of the larger sample of 100% Plant Clean) and homogenized prior to preparing charges for coking in CanmetENERGY’s 12 kg capacity sole-heated oven and its 340 kg capacity Carbolite pilot coke oven.

This section provides a description of the features and operating conditions for carbonization of coal in the sole-heated oven, including the preparation of coke samples from coals in this project for measurement of coke strength after reaction (CSR) and coke reactivity index (CRI) following a procedure developed at CanmetENERGY (MacPhee et al., 2013). It also provides a description of the Carbolite pilot oven used to carbonize a larger amount of the sample of 100% Plant Clean and assess its resulting coke quality.

Sole-Heated Oven (ASTM D2014-97(2010))

A 12 kg sample of coal (70–80% –3.35 mm or –6 mesh) was divided equally and each half charged into one chamber (approximately 280 mm in width, length and depth) of a double-chambered oven. A weighted piston applied a constant force corresponding to a pressure of 15.2 kPa (2.2 psi) to the top of the coal bed (thickness in the 76–90 mm range), which was heated from below according to a prescribed temperature program. The sole temperature was raised from 554°C to 950°C at a heating rate of 0.9–1°C/min during the test. The movement of the load was continuously monitored during the test, which was complete when the temperature at the top of the coal bed reached 500°C (normally reached after a period of 6–7 hours). The measured expansion or contraction of the sample was converted to a reference base of 833 kg/m^3 (52 lbs./cu. ft.) and 2% moisture.

After carbonization, the semi-coke was removed from the sole-heated oven and reheated in a stainless-steel holding box (229 mm wide, 292 mm long and 305 mm deep) that is hermetically sealed on top with a 3 mm thick section of stainless steel and lined with a 3 mm thick layer of ceramic-fibre insulation. The steel has an exit hole 1 cm in diameter in the centre for venting the hot coke gases. Also, the holding box is fitted on the bottom with a stainless-steel inlet tube (150 mm long and 6 mm inside diameter) connected to a cylinder of nitrogen gas, which allows for continuous flushing of the coke with the gas (5–10 L/min flow rate) to prevent its combustion. This treatment heated the semi-coke to 1100°C to complete the annealing of the coke.

A schematic of a sole-heated oven is presented in Figure 6 and a photo of the sole-heated oven used in this project is shown in Figure 7.

Cokes from the sole-heated oven were assessed for apparent specific gravity (ASG) and hot-strength properties, including CSR and CRI (following the ASTM D5341M-14 standard), and were analyzed for proximate (moisture, ash, volatile matter and fixed carbon), sulphur and carbon forms/textures using an optical microscope.

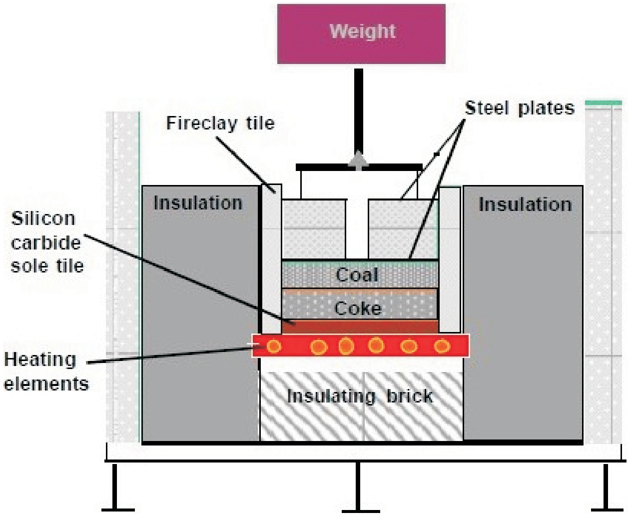

Figure 6. Schematic diagram of the CanmetENERGY sole-heated oven.

The ASG of coke is defined as the ratio of the mass of a volume of dry coke to the mass of an equal volume of water. Coke ASG varies with the rank and ash content of the coal carbonized, the bulk density of the coal charge in the oven, the carbonization temperature and the coking time (Price and Gransden, 1987). In this project, the ASG of cokes was determined following a method developed at CanmetENERGY and related to the ASTM D167-93 (2004) and ISO 1014:1985 standards.

According to ASTM D5341-14, the CRI is the percentage weight loss of the coke sample after reaction in CO_2 at 1100°C for 2 hours. The cooled, reacted coke is then tumbled in an I-drum for 600 revolutions at 20 rpm. The cumulative percentage of +9.5 mm coke after tumbling is denoted as the CSR.

Microscopic analysis of the textures was also performed on the sole-heated cokes to measure the carbon forms. This technique is extremely useful for understanding the behaviour of coal during coking and for interpreting pressure generation and coke-quality results.

Carbon-form analysis in cokes in this project was carried out using a combination of the US Steel method (Gray and DeVanney, 1986) and the CanmetENERGY method, which is based on work carried out by Marsh in 1978–1981 and published in the book *Introduction to Carbon Science* (Edwards et al., 1989). A single point count is made for each measured field of view. For each field, the stage is rotated in order to determine the possible highest rank carbon form. Normally, 500-point counts are performed on a sample. Each carbon form is derived from an assumed parent-coal vitrinite type. From the coke-texture analysis, one can determine the effective coal reflectance (%Ro).

Carbolite Pilot Oven

Specifications of the Carbolite pilot-scale movable-wall coke oven (Carbolite Gero Ltd., Sheffield, United Kingdom) are listed in Table 1 and the oven is shown in Figure 8.

Figure 7. CanmetENERGY sole-heated oven (12 kg capacity) used in this study.

Table 1. Specifications of the CanmetENERGY Carbolite pilot movable-wall coke oven.

Coke-oven specification	Carbolite pilot-scale movable-wall coke oven
Chamber width (mm)	460
Chamber volume (m^3)	0.401
Charge weight (kg)	340–350
Percentage of coal passing 3.35 mm	80–85
Charge density in oven (dry; kg/m^3)	809–825
ASTM bulk density (wet; kg/m^3)	773–783
Charge moisture (%)	2.5–3.2
Heating control (flue temperature; °C)	875°C start; increase 15°C/h to 1130°C
Pushing time (h)	3 h after centre temperature reaches 950°C (usually around 18 h)
Quench	Water (wet) normally; N_2 gas (dry) is also possible
Coke treatment (conditioning)	Client specified (usually 1 or 3 drops from 3 m height)

To simulate industrial coking, the temperature of the oven is kept low (875°C) at the beginning of the carbonization cycle, to limit the heat input to the coal, and then gradually raised (15°C/h) until the flue temperature reaches 1130°C. The oven is normally charged with coal 85 ±5% of which is less than 3 mm, and the coal moisture is adjusted so as to achieve a dry-coal bulk density in the oven in the range 810–825 kg/m^3. The oven is discharged 3 hours after the centre temperature of the coke reaches 950°C. The coke is water quenched and dropped 3 m onto a concrete floor in order to condition or stabilize it. This process is carried out in preparation for the drum testing, followed by measurement of the resultant coke properties.

The coke discharged from the Carbolite oven is assessed for size distribution, proximate analysis, sulphur, coke stability and hardness using the ASTM tumbler method, the cold strength drum index (DI) test of the Japanese Industrial Standard (JIS), CSR/CRI, ASG and texture.

Fundamentals of Coal Science

A small budget was set aside to further investigate the science behind the chemical reaction between perchloroethylene and coal macerals. The intent was to carry out the investigation using the micro-FTIR machine, but the work could not be completed during 2018. Therefore, the applicability of a family of liquids called Novec 7000 series in washing coal was tested, in collaboration with a research scientist at 3M.

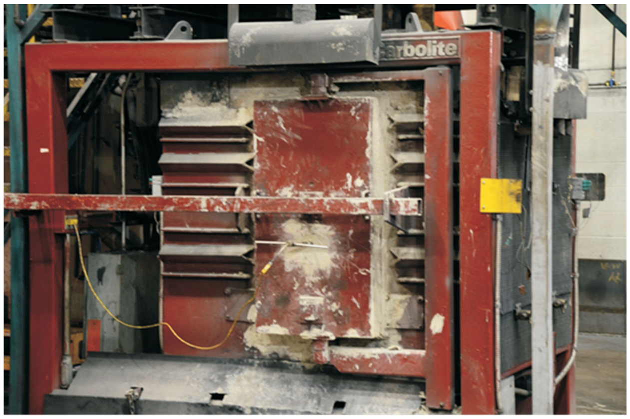

Figure 8. CanmetENERGY Carbolite pilot-scale coke oven (340 kg capacity) used in this study.

Novec 7000, also known as 1-methoxyheptafluoropropane, is an engineered liquid developed by 3M. It has a low toxicity, is not known or suspected to cause cancer, and is nonflammable and noncorrosive. It has a specific gravity of 1.40. Novec 7700, also known as Furan (2,3,3,4,4-pentafluorotetrahydro-5-methoxy-2,5-bis[1,2,2,2-tetrafluoro-1-(trifluoromethyl)ethyl), was also engineered by 3M. This liquid has the same benign characteristics as Novec 7000; in fact, it is suggested that it can be ingested without any medical concerns. The specific gravity of Novec 7700 is 1.797. The two liquids are miscible and can be used to create a range of liquids from 1.40 to 1.80 sg.

To complete detailed washability studies with coal, customers often require float densities over that range. This Novec fluid is a potential candidate for the replacement of harmful organic liquids in the float/sink procedure. Since 3M was prepared to donate the liquids for use in this research, it was thought that this was the opportune time to carry out the research. Novec 7000 and 7700 liquids were mixed in varying proportions to create heavy liquid baths with the following specific gravities: 1.40, 1.50, 1.60, 1.70 and 1.80. Raw coal underwent float-and-sink analysis according to ASTM D4371 in both traditional organic liquids (perchloroethylene, white spirits and methylene bromide) and the Novec solutions. The float-and-sink data were then compared for yield and quality characteristics. Clean coals resulting from both studies were also compared for a range of quality parameters. Results are not finalized as yet.

Results

This research project had the opportunity to obtain raw and clean coal from a single-seam plant run. Because of this, clean-coal samples derived from a number of methodologies were compared. As mentioned, the ability to compare laboratory-washed coal to that cleaned in an industrial-sized wash plant is considered the gold standard in validating a laboratory method for washing coal. Table 2 shows how these clean-coal samples compared.

On an ash basis, the clean coal derived from the float-and-sink process was a cleaner product. with ash less than 7%. The float/sink process in organic liquids provides ‘perfect separation’ between coal and rock. Care was taken not to create a clean-coal product that would be impossible to create in an industrial plant. Therefore, the specific gravity cut point remained less than 1.70, which is the cut-off achievable for a processing plant. The two factors, limiting the specific gravity cut point and the organic liquids achieving perfect separation (with no misplaced particles), resulted in the ash of the clean coal being lower than those from the Roben Jig or the industrial plant.

Other coal-quality parameters remained similar between the samples washed with organic liquids and those washed in the jig, even when changing the coarse size fractions. Re-jigging the clean coal that resulted from the traditional jigging method resulted in a lowering of the ash content from 7.80 to 6.68%. The petrography for these samples is still outstanding.

The free-swelling index of the six clean-coal composites ranged from 8.0 to 8.5, with only two of the composites obtaining the 8.5 value. The chlorine content was higher in the clean-coal composites washed in the perchloroethylene

Table 2. Clean-coal quality parameters (air-dried basis). Abbreviations: CCC, clean-coal composite; db, dry basis; ddpm, dial divisions per minute; FS, float-and-sink; REJIG, re-jigging; RJ, Roben Jig; SD 2.5, dilatation based on a 60 mm pencil per gram of air-dried coal in the test sample, multiplied by a constant of 2.5.

Clean coal quality (air-dried basis)	2018					
	FS CCC sizes	JIG CCC sizes	FS CCC 12.5 x 0.5 mm	JIG CCC 12.5 x 0.5 mm	REJIG CCC 12.5 x 0.5 mm	Plant product
Moisture (%)	1.29	1.21	1.20	1.38	1.22	1.00
Ash (%)	6.81	7.51	6.74	7.80	6.68	8.01
Volatile matter (%)	29.32	30.15	29.59	29.15	29.36	28.42
Fixed carbon (%)	62.58	61.13	62.47	61.67	62.74	62.57
Sulphur (%)	0.54	0.54	0.54	0.54	0.55	0.53
Free swelling index	8.5	8.0	8.0	8.0	8.0	8.5
Chlorine (ppm)	2280	365	2570	432	321	-
Flourine (ppm)	171	172	158	162	163	143
Hardgrove grindability index	79	78	78	79	80	-
Specific gravity	1.37	1.37	1.31	1.35	1.36	-
% phosphorus in coal (db)	0.040	0.042	0.043	0.045	0.044	0.045
Gieseler maximum fluidity (ddpm)	290	274	281	298	322	212
Ruhr dilatation						
% contraction	25	25	28	25	26	28
% dilatation	107	121	112	118	125	149
% total dilatation	132	146	140	143	151	177
% SD 2.5	108	120	118	127	133	150
Mineral analysis of ash						
$Si0_2$ (%)	58.50	59.76	57.56	59.57	58.27	58.86
Al_2O_3 (%)	31.44	29.60	32.79	29.61	31.16	29.78
TiO_2 (%)	1.57	1.47	1.56	1.41	1.52	1.53
CaO (%)	1.43	1.46	1.97	1.26	1.83	1.48
BaO (%)	0.28	0.31	0.31	0.32	0.29	0.28
SrO (%)	0.15	0.14	0.14	0.14	0.17	0.12
Fe_2O_3 (%)	1.46	1.59	1.50	1.62	1.60	3.17
MgO (%)	0.48	0.58	0.53	0.56	0.51	0.48
Na_2O (%)	0.08	0.08	0.05	0.07	0.07	0.09
K_2O (%)	1.16	1.51	1.22	1.66	1.41	1.22
P_2O_5 (%)	1.32	1.26	1.46	1.30	1.49	1.28
SO_3 (%)	0.27	0.27	0.30	0.23	0.23	0.21
Undetermined (%)	1.86	1.97	0.61	2.25	1.45	1.50

float-and-sink process. This was expected due to the chlorine content of the liquids. The Hardgrove Grindability Index, fluorine, % phosphorus in coal, and mineral analysis of ash were similar for all six composites.

The Gieseler maximum fluidity was measured on all composites within a two-day period, the values ranging from 212 to 322 dial divisions per minute (ddpm). Although small differences were observed, the fluidity of this coal did not appear to be significantly decreased due to the treatment with perchloroethylene.

The timeline for the dilatation testing was not kept as tight for all six samples. Therefore, the difference between the plant dilatation value of 149% and those of the other samples (107 to 125%) could have be partially due to aging.

Phase 1 Method: Float-and-Sink and Roben Jig, One Coarse Fraction

As was found with the work completed in 2017, the Roben Jig was able to create a clean-coal composite similar to that created using organic liquids except that, in the work reported here, the ash in the clean coal from the jig was higher. This was because the specific gravity cut-point was limited to what the processing plant could achieve at the mine. It is known that organic liquids provide a perfect separation and there is usually a ‘plant factor’ added to yield information coming from studies such as these to account for the imperfection of a processing plant. If the limitation was not imposed on the cut point, it would have been possible to make the ashes the same.

Also of note is that the Gieseler maximum fluidity values were comparable between the water-based method and the organic liquids. This could be due to there being less fusinite and semifusinite macerals in the coal. The petrography results will confirm this.

Misplaced Material

One of the objectives of this project was to identify and quantify the misplaced material that occurs in the column during the jigging process. A few clean slices were taken from the jigged clean coal and washed in organic liquids. Figure 9 illustrates the specific gravities of the particles that made up slice #13, one of the cleaner slices, which had an average relative density of 1.18. Washing the slice by float-and-sink showed that most of the particles fell at or below the 1.30 sg, but there were also particles from the 1.40, 1.60 and +1.90 specific gravities.

Figure 10 shows slice #1 from the Roben Jig. This was the slice with the highest ash content and had an average apparent relative density of 2.28. The float-and-sink process separated the coal particles into the correct specific gravity baths. Most of the particles fell into the +1.90 sg class, which is what one would expect to see as the highest ash ’sink’. This would be where one would find the rock. Particles contained in slice #1 also fell into the –1.30, 1.30, 1.40, 1.50, 1.60, 1.70 1.80 classes.

A further step was to take the clean-coal composite, as compiled from the Roben Jig method, and wash it in organic liquids to determine the density of the particles. The cut-point of the clean-coal composite had an ARD of 1.54. Table 3 shows the mass percentage falling into each specific-gravity class. Approximately 94% of the particles fell below the 1.54 sg. Five percent of the particles forming the clean-coal composite was misplaced from higher specific gravities, including 1.3% from the rock particle (+1.80 sg) sink. Although these misplaced particles exist, they do not seem to negatively impact the clean-coal quality parameters.

New Method A: Re-Jigging

The clean coal resulting from the Roben Jig traditional method was re-jigged to determine if a second cleaning action could remove misplaced particles. After re-jigging, the clean coal created during the second jigging process underwent float-and-sink analysis in organic liquids. Table 4 shows the percentage of particles falling into each specific gravity range. After re-jigging, only 2.9% of the particles in the clean-coal composite were misplaced from other higher specific gravity ranges. Re-jigging was confirmed to be a useful method of reducing the amount of misplaced material.

New Methods B and C: Float-and-Sink and Roben Jig, Two Size Fractions

Changing the size range of the coarse particles that undergo float-and-sink washing is a standard practice in the laboratory washing of coal. It was because of this, and some information from Pielot (2010), that it was proposed to investigate washing different coarse fractions separately in the Roben Jig. Pielot thought that narrowing the size fraction resulted in more precise jigging. In this study, no difficulties were encountered while jigging the 12.5 × 2 mm frac-

Table 3. Float-and-sink mass percentages falling within specific-gravity ranges of a rewashed clean coal produced by the Roben Jig. Abbreviations: FLT, float; SNK, sink.

Specific gravity	Mass percentage
1.30 FLT	67.4
1.30 - 1.40	22.6
1.40 - 1.50	4.1
1.50 - 1.60	1.4
1.60 - 1.70	0.9
1.70 - 1.80	0.7
1.80 SNK	3.0

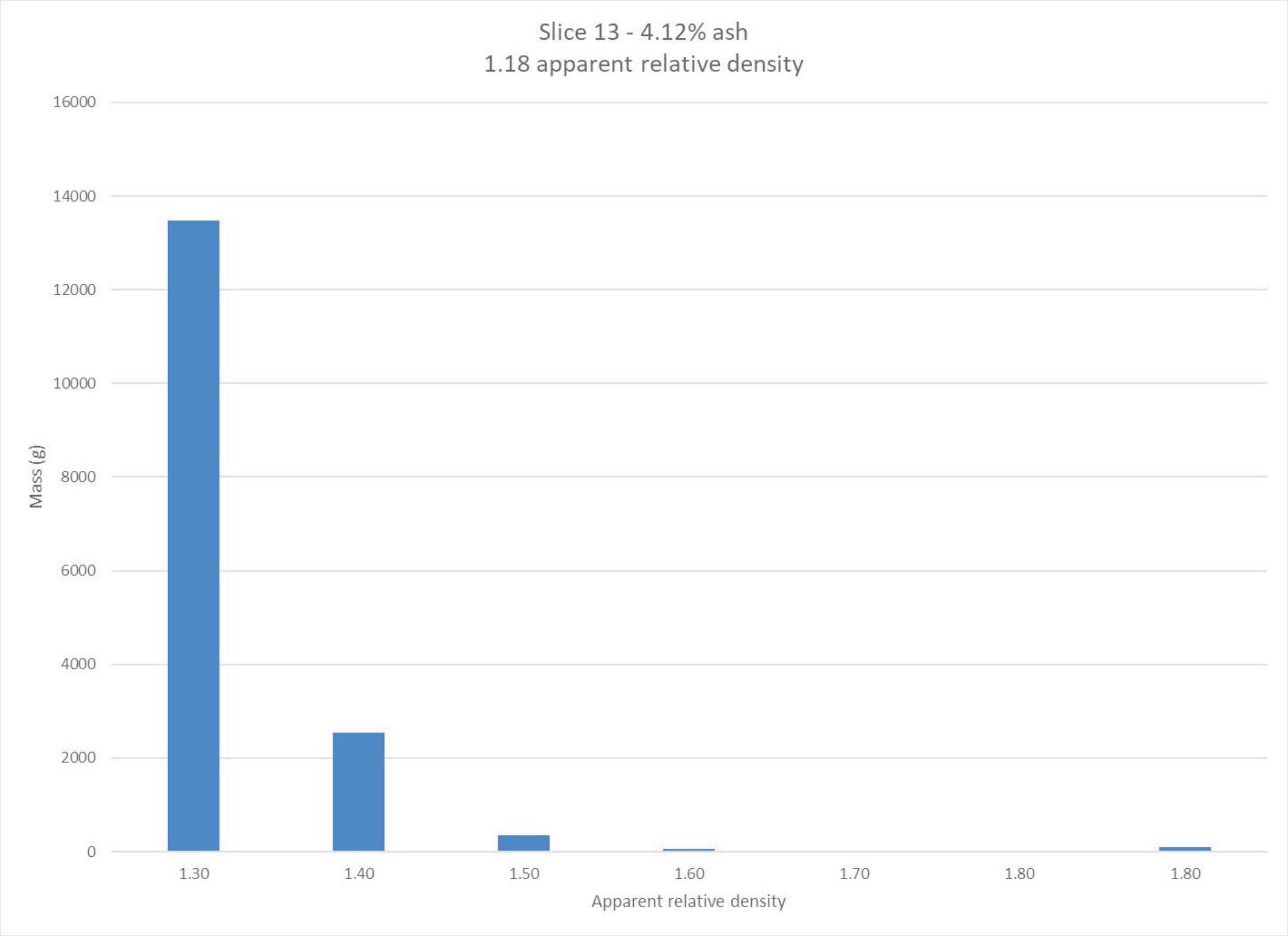

Figure 9. Particle-density distribution, expressed as proportions by mass, of low-density jig slice.

tion. However, jigging the finer 2 × 0.5 mm fraction proved difficult: it was found that there were inconsistencies between slices. Usually, the relative density of slices behaves predictably: the highest relative density occurs at the bottom of the column, with lower relative densities occurring in a stepwise fashion moving to the top of the column. The inconsistencies between slices of the finer fraction suggested that there was misplaced material. When the clean coal from this fraction was washed by float-and-sink, it was found that there were 9% misplaced particles. Table 5 shows the specific-gravity distribution of the coal particles at the 1.40 sg cut point.

Table 4. Float-and-sink mass percentages falling within specific-gravity ranges of a rewashed clean coal produced by re-jigging. Abbreviations: FLT, float; SNK, sink.

Specific gravity	Mass percentage
1.30 FLT	69.9
1.30 - 1.40	23.8
1.40 - 1.50	3.5
1.50 - 1.60	1.0
1.60 - 1.70	0.6
1.70 - 1.80	0.4
1.80 SNK	0.9

From an operational standpoint, jigging the finer size fraction was also more time consuming, as the operator had to continually check the work that was done. This method of misplaced-material mitigation did not prove to be the best method.

Clean-Coal Carbonization

Table 6 presents analytical data (available thus far) for cokes made in the sole-heated oven from coals studied in this project.

Contraction levels range from –15% for 100% plant clean product crushed to 12.5 mm to –9% for 100% JIG CCC 12.5 × 0.5 and 100% REJIG 12.5 × 0.5. The type of washing medium, namely organic liquids for float-and-sink and water for Roben Jig, had a minor effect on the level of sole-heated oven contraction observed, –11% and –10%, respectively. The low amount of volatile matter remaining in the cokes (<1%) provides clear evidence that the coals were

Table 5. Float-and-sink mass percentages falling within specific-gravity ranges of a rewashed clean coal produced by jigging the 2 x 0.5 mm size fraction. Abbreviations: FLT, float; SNK, sink.

Specific gravity	Mass percentage
1.30 FLT	72.2
1.30 - 1.40	18.8
1.40 - 1.50	3.7
1.50 - 1.60	1.8
1.60 - 1.70	0.8
1.70 - 1.80	0.5
1.80 SNK	2.2

essentially fully carbonized by a combination of coking in the sole-heated oven and heat treatment of the resulting semi-coke to 1100°C under nitrogen to complete the annealing of the coke.

The apparent specific gravity (ASG) of coke ranged between 0.96 (100% REJIG 12.5 × 0.5) and 1.01 (100% plant clean product crushed to 12.5 mm). As stated earlier, the rank and ash content of the carbonized coal dictates the coke ASG. The low ash content of 6.8% (db) in 100% REJIG 12.5 × 0.5 results in the lowest ASG coke, whereas the high ash content of 8.2% (db) in 100% plant clean product crushed to 12.5 mm results in the highest ASG coke.

The coke strength after reaction (CSR) of the clean coal resulting from all washing methods was compared. Comparison of New Methods B (FS CCC sizes) and C (JIG CCC sizes) revealed that Method C (Roben Jig) washing improved the CSR by 5 points relative to Method B (float-and-sink). Method C (JIG CCC sizes) also resulted in a 6-point increase in CSR when compared to the Phase 1 Method (JIG CCC 12.5 × 0.5). Washing the coal using New Method B (FS CCC sizes) resulted in a 3 point decrease in the CSR compared to the Phase 1 Method (FS CCC 12.5 × 0.5).

A comparison of the CSRs resulting from coal washed using the Phase 1 methods of jigging and float-and-sink (one coarse fraction of 12.5 x 0.5 mm) shows that Roben Jig washing led to a CSR 4 points lower than float-and-sink

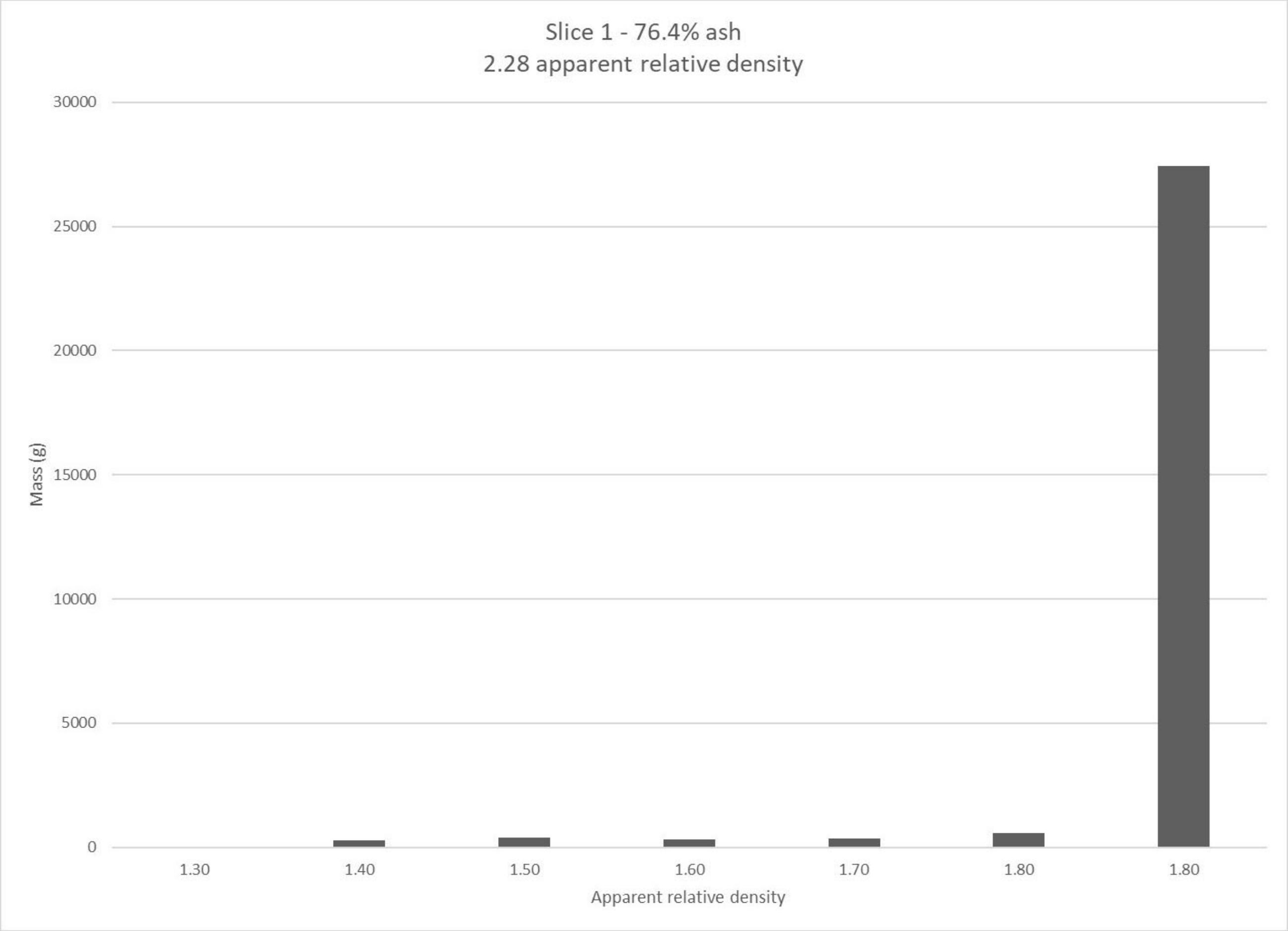

Figure 10. Particle-density distribution, expressed as proportions by mass, of high-density jig slice.

Table 6. Quality of coke samples obtained from sole-heated oven tests. Abbreviations: CCC, clean-coal composite; FS, float-and-sink; JIG, Roben Jig; REJIG, re-jigging.

	2018						
Coke property	**FS CCC sizes**	**JIG CCC sizes**	**FS CCC 12.5 x 0.5**	**JIG CCC 12.5 x 0.5**	**REJIG CCC 12.5 x 0.5**	**Plant product**	**Crushed plant product**
Expansion/contraction	–11.8	–11	–11	–9.2	–9.4	–11.6	–14.5
Coke strength after reaction (CSR)	65.5	70.5	68.3	64.3	65.0	61.5	64.2
Coke reactivity indiex (CRI)	21.3	17.2	18.5	19.2	20.6	25.2	23.0
Apparent specific gravity (ASG)	0.970	0.995	0.988	0.989	0.963	1.004	1.012

washing. Re-jigging of this coal led to a negligible change/improvement in CSR.

The CSR of the plant-washed coal (plant product) was 61.5. After crushing the plant-washed coal to pass 12.5 mm, the CSR increased to 64.2. Crushing of the product to 12.5 mm led to a 3 point improvement in CSR relative to the complete size range. It should be noted that the repeatability of this test is 3.3. This would indicate that the CSRs are actually quite similar.

Table 7 presents analytical data for coke made in the Carbolite oven from 100% plant clean product (sample #183147).

Assessment of the quality of cokes made in the sole-heated oven and the Carbolite pilot oven from 100% plant clean product found ASTM stability and hardness of 56 and 66, respectively; JIS DI30/15 and DI150/15 of 94 and 85, respectively; and CSR and CRI of 58 and 27, respectively. The CSR and CRI from the larger pilot-scale coke oven (340 kg) are found to be inferior to those from the smaller sole-heated oven (12 kg), which are 62 and 25, respectively (Figure 11).

The superior CSR and CRI of coke produced in the smaller sole-heated oven is expected, since the carbonization process took place under a significantly higher load/pressure (15.2 kPa) than that in the larger pilot oven (4–8 kPa). This led to the formation of a coke with higher apparent density (ASG of 1.00), and thus lower porosity, from the sole-heated oven compared to that from the larger pilot oven (ASG of 0.92).

Conclusions

The Canadian coal industry needs a reliable method of washing small metallurgical-coal samples whereby the exposure of both the coal sample and the laboratory technicians to perchloroethylene and other toxic organic liquids can be eliminated. This study evaluated the use of the Roben Jig and varying methodologies, as well as two engineered liquids (Novec 7000 and 7700), in satisfying these requirements.

When comparing the quality characteristics of clean coal samples, it is apparent that the Roben Jig was able to produce a clean-coal sample similar to that from the industrial coal-washing plant that uses the float-and-sink method

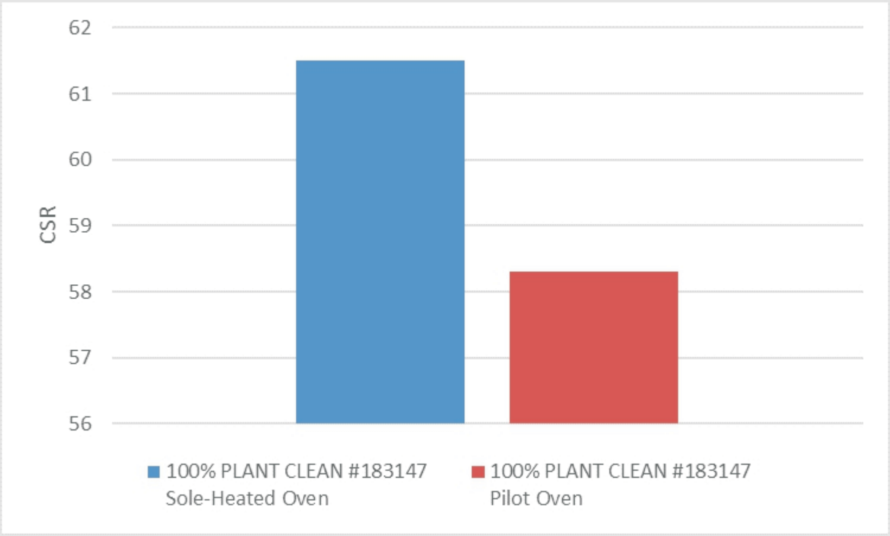

Figure 11. CSR comparison of cokes made in the sole-heated oven and Carbolite pilot-scale oven from 100% plant clean product (sample #183147).

Table 7. Quality of coke samples obtained from Carbolite pilot oven tests on 100% plant clean #183147.

	Date received		AUG/7/18
	Weight received		3-DRUMS
	Project		CCRA 90 - Roben Jig Phase 2
	Coal index		26799
	Description		100% PLANT CLEAN #183147 C-2733
Sole-heated oven test	Expansion/contraction	%	-11.6
Coke moisture	Moisture	%	x
Coke proximate (db)	Ash	%	x
	Volatile matter	%	x
	Fixed carbon	%	x
	Sulphur	%	x
Carbonization results	Oven test number		C-2733
	Test date		SEP/7/18
	Moisture in charge	%	3.0
	Net dry charge weight	kg	336.8
	ASTM BD	kg/m^3	773.7
	Oven dry BD	kg/m^3	815.6
	Coking time	h:min	18:14
	Final centre temperature	°C	1074
	Time to 900°C	h:min	14:48
	Time to 950°C	h:min	15:14
	Time to 1000°C	h:min	15:51
	Time to max. wall pressure	h:min	2:45
	Max. wall pressure	kPa	4.1
	Max. gas pressure	kPa	6.7
	Coke yield	%	72.4
Sieve analysis of coke (cumulative)	100 mm sieve	%	0.4
	75 mm sieve	%	7.6
	50 mm sieve	%	51.3
	37.5 mm sieve	%	83.6
	25.0 mm sieve	%	94.3
	19.0 mm sieve	%	95.3
	12.5 mm sieve	%	96.1
	Passing 12.5 mm sieve	%	3.9
	Mean coke size	mm	53.0
ASTM coke tumbler test	Stability		56.3
	Hardness		66.3
JIS coke tumbler test	50 mm sieve, 30 rev.		21.2
	25 mm sieve, 30 rev.		90.6
	15 mm sieve, 30 rev.		93.6
	50 mm sieve, 150 rev.		8.0
	25 mm sieve, 150 rev.		79.4
	15 mm sieve, 150 rev.		85.1
	CSR		58.3
	CRI		27.2
	ASG		0.920
Coke texture	Isotropic	%	2.7
	Very fine mosaic	%	7.4
	Fine mosaic	%	34.9
	Medium mosaic	%	32.3
	Coarse mosaic	%	3.8
	Total mosaic	%	78.4
	Elongated fine flow	%	4.2
	Elongated medium flow	%	0.9
	Elongated coarse flow	%	0.0
	Total flow	%	5.1
	Domain flat flow	%	0.0
	Domain undulating	%	0.0
	Domain ribbon	%	0.0
	Total domain	%	0.0
	Fusinite	%	3.5
	Semifusinite	%	9.5
	Unidentifed inerts	%	0.8
	Altered vitrinite	%	0.0
	Total inert	%	13.8
	Coal Ro calculated		1.04
	Coke mosaic index		2.02

with organic liquids. The ash value of the clean coal from the jig was higher than that from the float-and-sink method, but only because of limitations on cut-points imposed by the methodology. Misplaced material was found in clean-coal composites created in the jig, but in small enough proportions that it did not have a negative effect the clean-coal quality. A re-jigging action was found to be the most useful in reducing the percentage of misplaced particles. Narrowing the size range of particles during jigging proved to be operationally difficult for the finer size fraction (2 × 0.5 mm).

With respect to carbonization, evaluation of the cokes produced from the plant clean-coal product after washing of the complete size range of coal particles in the two types of media showed that the Roben Jig (New Method C) had a CSR of 70 compared to values of 65.5 and 68.3 for the float-sink methods (Phase 1 Method and New Method B). The New Method C of washing two coarse size fractions separately in the Roben Jig led to a 6-point improvement in CSR relative to washing only the 12.5 × 0.5 mm size fraction. Re-jigging the coal (New Method A) led to a negligible change/improvement in CSR.

Future Work

Study in this research project is ongoing. Future work will involve summarizing the results from the following tasks:

- Coal petrography
- Coke-texture petrography
- Novec 7000 series washability

Acknowledgments

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