

FINAL TECHNICAL REPORT
April 1, 2007, through June 30, 2008

Project Title: **APPLICATION OF PULSEWAVE DISINTEGRATION TO
COMMUNITION, DRYING AND CLEANING OF ILLINOIS COAL**

ICCI Project Number: 06-1/2.1A-2
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ABSTRACT

Pulsewave resonance disintegration technology is a breakthrough technology for the efficient and cost effective comminution of coals and other materials to fine particle sizes. The application of this technology to coal comminution for use with existing pulverized coal combustion (PCC) facilities has been investigated. Pulsewave comminution effectively and efficiently reduces coal to particle sizes suitable for PCC. Furthermore, this technology also reduces the inherent moisture content of Illinois coals by 60 to greater than 80%, with a concomitant ~10% increase in thermal value, without requiring additional heating of the coal or pulverizing unit. This technology is ready for deployment and should be demonstrated at full scale in a commercial PCC facility.

Application to deep coal cleaning has also been investigated. The inherent ability of the Pulsewave process to effectively liberate mineral matter from the coal matrix maximizes the mercury reductions that can be achieved by physical coal cleaning. Mercury was found to report preferentially to the liberated mineral matter (by a factor of 10-40 fold) and mercury reductions in the cleaned coal of 60-80% were achieved. However, improved large scale techniques for the separation of fine particles of distinct densities are necessary before this approach can be used for coal cleaning on a commercial scale.

EXECUTIVE SUMMARY

The overarching objective of this study was to investigate the application of Pulsewave resonance disintegration technology to Illinois coals. Two broad areas of interest were investigated:

1. Comminution of coal for application with pulverized coal combustion systems.
2. Deep coal cleaning.

Effort towards Objective 1 focused on investigation and quantification of the benefits of the application of this technology for use with pulverized coal combustion (PCC) systems. Results obtained demonstrate that Pulsewave resonance disintegration is highly effective for the comminution of Illinois coals to particle sizes suitable for use with existing PCC facilities. Furthermore, Pulsewave comminution effectively reduces coal moisture contents by 65-85%, resulting in an ~ 10% increase in thermal value, without requiring additional heating of the coal or pulverizing unit. This technology is ready for deployment and should be demonstrated at full scale in a commercial PCC facility.

Application to deep coal cleaning was also investigated. Removal of undesirable “contaminants”, such as mercury, prior to coal utilization is an alternative to post utilization contaminant capture technologies. The success of this approach depends on the ability to effectively liberate phases containing undesirable elements from the organic matrix of the coal, and then to separate these phases, rejecting those that contain the undesirable materials. Pulsewave resonance disintegration is currently the most effective technology for liberation of coal and mineral phases during comminution. Laboratory-scale separations of Pulsewave comminuted coal show that mercury reports preferentially to the mineral phases and can be effectively rejected by deep coal cleaning. Mercury was found to be enriched by factors of 10-40 fold in rejected materials and cleaned coal was found to show mercury reductions of 60-80% for coals comminuted using Pulsewave technology. This reduction was achieved even for coal that had already been processed through a conventional coal preparation plant. Further development of effective commercial-scale technology for separation of fine coal particles will need to be developed in order to utilize this approach on a commercial scale for coal cleaning applications.

OBJECTIVES

Pulsewave resonance disintegration technology is a breakthrough technology for the efficient and cost effective comminution of coals and other materials to fine particle sizes. This technology is expected to have significant applications in the coal utilization industry in two major areas, which represent short and medium-long term application targets respectively. These are:

- Comminution of coal for application with pulverized coal combustion systems.
- Deep coal cleaning.

The overall objective of this project is the demonstration and quantification of the benefits of Pulsewave resonance disintegration technology for Illinois coals in both of these target applications.

INTRODUCTION AND BACKGROUND

Comminution of coal, both for pulverized coal combustion and for deep coal cleaning by physical separation of associated minerals, is typically achieved by conventional grinding and milling technology, e.g. ball mills, ring mills and jet mills. At a fundamental level, all of the conventional technologies used for coal pulverization rely on physical impacting of the coal to achieve comminution and for coal cleaning applications, all suffer from the drawback that these processes do not efficiently liberate undesirable small mineral particles from the desirable carbonaceous materials. In fact, in many instances these technologies tend to “smear” mineral and carbonaceous phases together, making subsequent separation of individual organic and mineral phases impossible.

Pulsewave, LLC has developed a technology for reducing the particle size of coal and other materials by the application of destructive resonance, shock waves, and vortex-generated shearing forces¹. Preliminary testing with Illinois and other coals has demonstrated that this technology has significant advantages when compared with conventional coal comminution technology:

- The Pulsewave process results in a relatively clean liberation of component phases. This eliminates the “smearing” of the different organic and mineral fractions that can take place in conventional mills and should result in particle size reduction accompanied by a cleaner separation of the carbonaceous and mineral phases and less crossover of unwanted materials in each phase. This translates to a clean liberation of pyrite from coal.
- Significantly less energy is required for the work being done when compared with the energy required for traditional impact/grinding processes, and based on preliminary figures, the Pulsewave method of producing “clean coal” is very competitive with current technology.

¹ United States Patent 6726133

- Based on preliminary testing with Illinois and other coals, it appears that the Pulsewave process reduces the moisture content coal during the processing run. In preliminary testing with Illinois #5 coal, moisture reduction of > 50% was readily achieved in a single machine pass. This is an important benefit because, for thermodynamic reasons, lowering of the moisture content equates to a significant increase in BTU value and hence in overall efficiency.
- The particles produced by the Pulsewave process are generally statistically closer in terms of size and dimensions, than those produced by normal crushing methods, which should result in more uniform fuel consistency and improved combustion characteristics.

Application to Pulverized Coal Combustion (Objective 1)

Pulverized coal combustion (PCC) is a well established and widely deployed technology, both in Illinois and elsewhere (U.S. and international). Efficient PCC requires comminution to small mean particles sizes, typically less than 60 microns, to achieve desired combustion characteristics and minimize carryover of unburnt carbon in the residual ash. Existing PCC burners rely on conventional milling/pulverizing technology to accomplish this goal. A variety of conventional pulverizing technologies, including ring and ball mills are commonly used for this purpose. Pulsewave's resonance disintegration technology has significant advantages over conventional technology and hence has the potential to replace conventional pulverizing technology in both existing and advanced PCC systems. Pulsewave resonance disintegration is able to achieve comminution of Illinois (and other) coals to fine particle sizes at cost comparable to or less than conventional pulverizers. The additional benefit of simultaneous coal drying achievable with Pulsewave technology has the potential to provide overall energy efficiency gains in PCC systems that make this emerging technology attractive for PCC applications.

Application to deep physical coal cleaning (Objective 2)

The State of Illinois has proposed aggressive mercury (Hg) emission reduction goals of 90% reduction by 2009.² To achieve this goal, significant improvements in Hg management will need to be achieved. This is likely to involve a combination of pre-combustion coal cleaning and post-combustion capture and removal. Stakeholders, including industrial users and responsible regulatory agencies need to define strategies for compliance with the governor's policy. This aspect of this project was intended to provide these stakeholders with data defining the ultimate limits of S and Hg reductions achievable by physical coal cleaning. The unique properties of Pulsewave resonance disintegration in terms of efficient liberation of mineral matter and carbonaceous materials within the coal make Pulsewave's technology the methodology of choice for effective liberation of mineral and coal phases during comminution. Hence, this technique can be used to determine the ultimate limits to which Hg and other elements

² <http://www.illinois.gov/PressReleases/ShowPressRelease.cfm?SubjectID=3&RecNum=4565>

associated with mineral matter associated with Illinois coal can be removed by physical coal cleaning using the best available technology. These data can then be used to guide decision making regarding implementation strategies for compliance with the aggressive Hg emission reduction targets set by the State.

EXPERIMENTAL PROCEDURES

Sample collection

All samples used for this study were collected as random grab samples from the raw or washed coal piles of participating mines. Samples were placed in sealed 5-gallon buckets at the time of collection to minimize moisture loss during transportation and storage.

Pulsewave comminution

The Pulsewave resonance disintegration machine has been described elsewhere¹. The Pulsewave machine operates on the principle of resonance disintegration that reduces the particle size of various materials by the application of the physics of destructive resonance, shock waves and vortex-generated shearing forces. The basic operation of the RD machine entails feeding a material in the machine at a predetermined rate. For coal comminution, the input generally pre-sized to -2 inches (to assist in establishing an approximately constant feed rate. The coal is carried in a stream vertically through the machine, from top (input) side to the bottom (output) end. The more uniform the feed material, the cleaner the output in terms of particle size consistency. The coal is carried through the machine in a gaseous medium that can consist of ordinary air, nitrogen, carbon dioxide, helium or other gas.

DGC separations

A density gradient is produced by sequentially mixing distilled water and a cesium chloride (CsCl) solution to produce a gradient that ranges from 1.00 to 1.70 g/mL. A micronized coal sample is mixed with distilled water and a surfactant and carefully layered on top of the gradient. The gradient is then spun in a centrifuge for approximately 2 hours. The accelerated g forces (approximately 15,000 x g) force the coal/mineral particles to partition through the gradient until they reach a level at which the density of the medium is equal to the particle density. The centrifuge is carefully stopped and the gradient (containing the separated macerals) is fractionated and filtered. The filters are weighed to obtain the weight of each fraction. Each fraction and filter was assigned a density value during fractionation. These two values are plotted to produce a maceral distribution plot.

Analytical Measurements

Proximate, ultimate and trace element analyses were conducted by external vendors according to ASTM methodology for these analyses. All analyses were conducted "blind" (i.e.) the vendors were not informed of the nature of the samples or their interrelationships. A minimum of duplicate analyses were performed on all samples.

Optical/Petrographic analysis

Optical/petrographic analyses were conducted by modified ASTM point counting methods with a Leitz Orthoplan polarizing microscope in reflected light. An oil immersion objective lens with 10x eyepieces gave a magnification of about 500x. Raw samples were crushed to -20 mesh, mounted in epoxy pellets and polished for analysis. All macerals were counted in white light as well as a second analysis of the liptinites in mercury-arc excited blue fluorescent light. In white light two sets of 500 points were counted on each sample and in fluorescent light two sets of 250 points were counted. In blue light all non-liptinite macerals were counted as other. No voids, mounting media or mineral matter were included in any of the counts. The white light and blue light results were combined to yield a total analysis.

The same samples were processed through the Pulsewave device and similar petrographic pellets were prepared. These were analyzed with the same Leitz Orthoplan polarizing microscope in reflected light at the same magnification. In these samples four particle types were counted. These were: single maceral, single mineral, mixed maceral particles, and particles that contain both macerals and minerals.

RESULTS AND DISCUSSION

As outlined in the original proposal, Pulsewave resonance disintegration technology is a breakthrough technology for the efficient and cost effective comminution of coals and other materials to fine particle sizes. This technology is expected to have significant applications in the coal utilization industry in two major areas, which represent short and medium-long term application targets respectively. These are:

- Comminution of coal for application with pulverized coal combustion systems.
- Deep coal cleaning.

For organizational purposes, effort in this project was broken into the following tasks, which reflect effort aimed at investigation of both of these objectives. In accordance with ICCI guidelines, this report is also organized according to these project tasks.

Comminution and drying

Task 1.	Sample collection
Task 2.	Comminution
Task 3	Laboratory analysis (I)

Deep coal cleaning

Task 4.	Optical analysis
Task 5.	Density Separation
Task 6.	Laboratory Analysis (II)

This report completes effort under task 7, reporting, of this project.

Task 1: Sample collection

For the purposes of this investigation, six fresh coal samples were collected from active

Illinois coal mines. In all cases, mine operators were supportive of the project and provided access for sample collection. In some instances, mine operators requested that their mine not be identified in public documents. Therefore, the coals used are referred to only by coded identifiers throughout this document. A table identifying each sample is has been provided to ICCI for evaluation only by ICCI staff and other authorized personnel.

A total of six coals were collected for use in this project. In all cases, five 5-gallon buckets of coal were collected. Five of the samples were unsorted, random grab-samples of mine fresh coal taken from the fresh coal pile at each mine. One sample of washed coal was also collected for comparison with unwashed coal from the same mine. Samples were crushed by hand to -1" and well mixed to ensure representativeness, before sub-sampling. Approximately 15 gallons (three buckets) were then used for Pulsewave comminution with the remainder being used for control and comparison experiments or held in reserve.

Task 2: Comminution

Coals were subjected to resonance disintegration at Pulsewave's Charleston, Illinois, facility. The unit used for this purpose is illustrated in Figure 1.



Figure 1. Pulsewave's Resonance Disintegration demonstration unit, Charleston, Illinois.

The particle size distributions of the resulting pulverized products were all determined using a Malvern Mastersizer 2000 particle size analyzer. All analyses were run in triplicate. The results of these analyses (average of three runs) are summarized in Table 1 below. An example particle size distribution is illustrated in Figure 2.

Coal	$d_{(0.1)}$	$d_{(0.5)}$	$d_{(0.9)}$
A	3.81	28.24	176.25
B	4.64	35.52	192.67
C	5.41	38.52	220.61
D	3.94	26.26	178.58
E	2.24	20.78	165.04
F	4.07	25.77	159.89

Table 1. Particle size results for Pulsewave comminuted Illinois coals. Results are given in μm . $d_{(0.\#)}$ indicates fraction smaller than indicated particle size; e.g. $d_{(0.9)}$ indicates 90% smaller than indicated particle size.

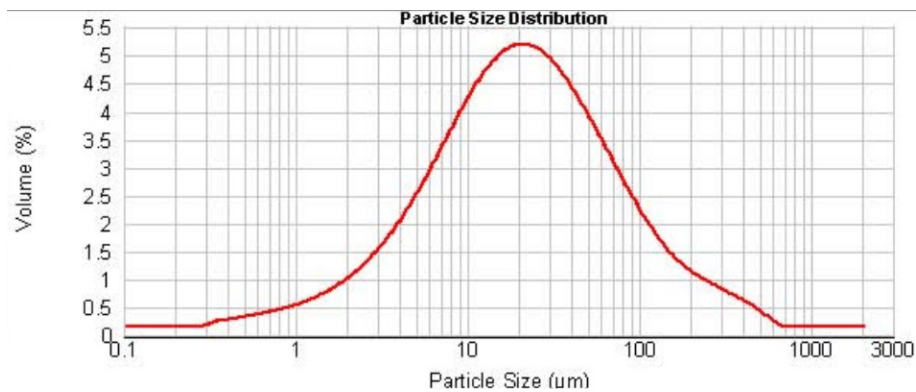


Figure 2. Representative particle size distribution for Pulsewave comminuted Illinois coal.

In all cases, resonance disintegration was able to successfully comminute the coal to fine particle sizes in a single machine pass, demonstrating that this technology can effectively and efficiently produce coal suitable for introduction to a pulverized coal burner. In addition, during disintegration, significant moisture reduction is achieved. Quantification of this benefit is described in the following section of this report.

Task 3: Lab Analysis I (Drying and Calorific Value)

The objective of this task was to quantify the effectiveness of Pulsewave technology for the simultaneous drying and pulverizing of Illinois coal. Due to the thermodynamics of the evaporation of water, combustion of wet coal is less efficient than combustion of dried coal. Therefore, utilities and others utilizing pulverized coal combustion generally take steps to minimize the inherent moisture content of the fuel prior to combustion. Typically, pulverized coal is prepared by conventional ring or ball-milling. In some cases, these mills are operated at elevated temperature ($>80^{\circ}\text{C}$) to reduce the moisture content of the coal prior to combustion. Previous studies of coal comminution using resonance disintegration had suggested that Pulsewave's technology also effectively reduced the inherent moisture content of the coal during comminution without the need for additional heating.

To quantify this effect, the moisture, calorific value and other coal properties were determined prior to and following resonance disintegration. All analyses were conducted by external analytical services and duplicate analyses were performed on all samples. All analyses were conducted “blind” (i.e. the analytical services were not told the relationships between any of the samples tested) to ensure data integrity. The results of these analyses are summarized in Tables 2 and 3, and Figures 3 and 4 below.

Coal	Moisture	Ash	Vol. matter	Fixed carbon	BTU/lb
A	7.85	19.9	30.9	41.3	10243
B	10.74	12.1	30.3	46.8	11060
C	9.67	6.4	33.3	50.7	12004
D	4.35	18.9	33.6	43.1	11475
E	7.50	39.0	23.7	29.9	7554
F	8.23	21.1	27.3	43.4	10102

Table 2. Proximate and calorific data for RAW coals used in present study. Data are averages of duplicate analyses. Vol. matter, Fixed carbon, and elemental compositions are reported on as received basis and are not corrected for moisture or ash content.

Coal	Moisture	% Moisture Removal vs Raw coal	Ash	Vol. matter	Fixed carbon	BTU/lb	% Increase in BTU/lb vs Raw coal
A	2.55	67%	18.3	32.7	46.5	11169	+9.0%
B	2.20	80%	12.3	30.8	54.8	12310	+11.3%
C	3.31	66%	7.4	33.0	56.3	12876	+7.3%
D	0.93	79%	17.0	33.2	48.9	11847	+3.2%
E	1.15	85%	38.9	24.1	35.8	8347	+10.5
F	1.85	77%	21.8	27.5	48.9	10950	+8.4

Table 3. Proximate and calorific data for Pulsewave comminuted coals used in present study. (Data are averages of duplicate analyses) Vol. matter, Fixed carbon, and elemental compositions are reported on as received basis and are not corrected for moisture or ash content.

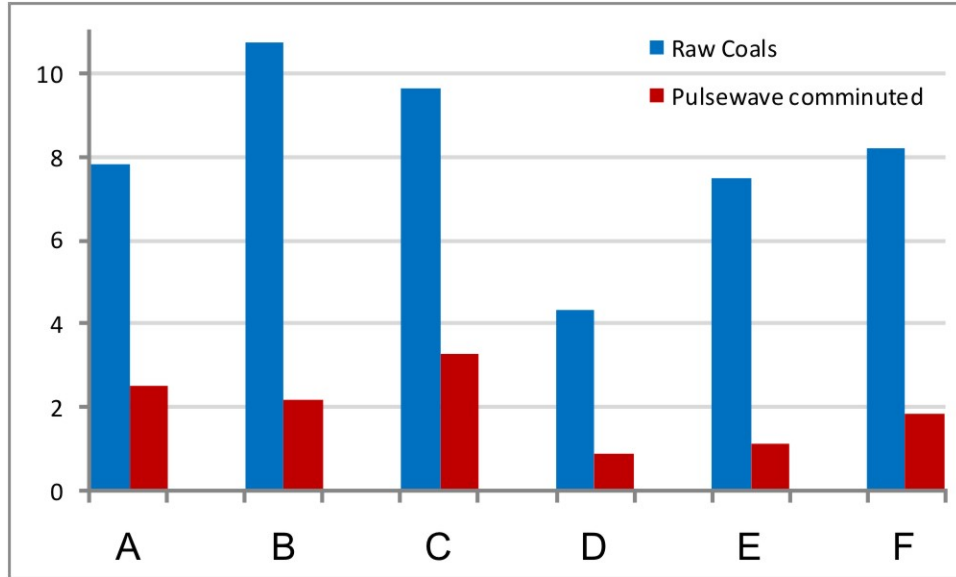


Figure 3. Moisture content of raw (blue bars) and Pulsewave comminuted (red bars) Illinois Coals. Data are in wt% moisture (measured according to ASTM standards by independent laboratories).

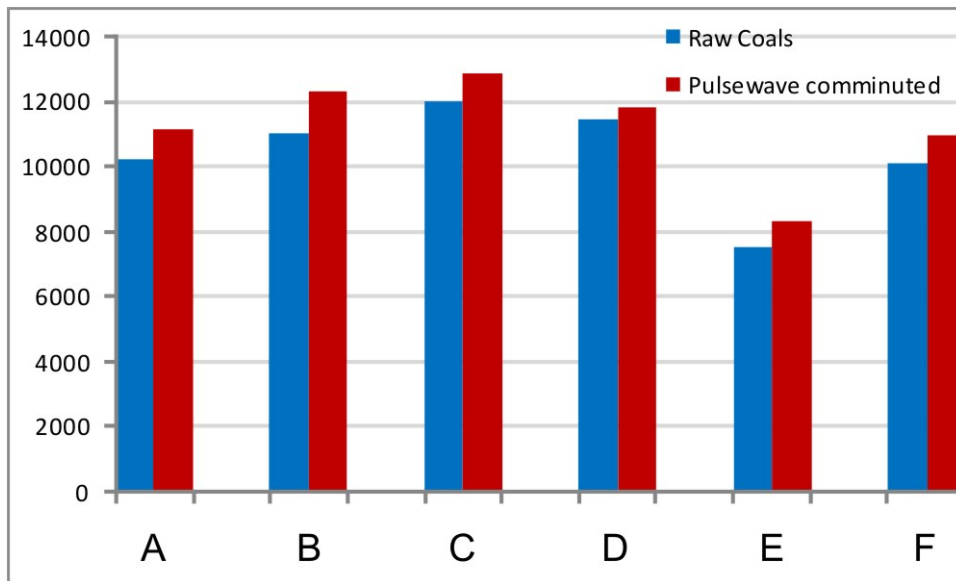


Figure 4. Thermal value of raw (blue bars) and Pulsewave comminuted (red bars) Illinois Coals. Data are in BTU/lb moisture (measured by according to ASTM standards by independent laboratories).

In all cases investigated Pulsewave comminution resulted in significant decreases in

inherent moisture content without requiring additional separate drying or heating of the coal to remove moisture. In general for typical Illinois coal, Pulsewave comminution results in removal of 60% to >80% of the inherent moisture present in the coal with an approximately 10% concomitant increase in thermal value.

Deep coal cleaning:

Effort under tasks 4-6 was aimed at investigating the limits of physical coal cleaning. Coal cleaning is necessary and desirable to remove entrained minerals, especially including pyrite, prior to coal utilization. This reduces levels of sulfur and other undesirable elements in the coal which reduces the environmental footprint of coal utilization and also increases the value of the coal. Conventional coal cleaning, as currently practiced, removes entrained macroscopic rock and mineral inclusions that occur with the coal when mined. Deep coal cleaning (as defined for the purposes of this report) aims to remove a much larger fraction of the entrained and extraneous inorganic materials that co-occur with the coal, including microscopic mineral inclusions that are not removed by conventional coal cleaning.

Ultimately, the efficacy of physical coal cleaning is limited by the extent to which coal constituents containing undesirable elements (e.g., S, Hg, As and others) can be efficiently liberated and separated from other coal constituents. For example, removal of elements randomly associated with the organic phase of coal cannot be achieved without unacceptable rejection of carbon. As noted above, preliminary studies have shown that Pulsewave's technology results in coal comminution with a high degree of liberation of discrete phases. That is, mineral matter is effectively liberated from the organic components present in the coal.

Based on this observation it was suggested that this technology represents the best available opportunity for evaluating the limits of physical coal cleaning, i.e. determining the level to which sulfur, mercury and other trace elements can be liberated from the organic matrix of the coal and separated by physical methods. It was not an objective of this effort to develop or test commercially feasible coal cleaning methods. Rather this effort focused on determining the ultimate limit of physical cleanability, based on the best available technology for physically liberating coal and co-occurring minerals.

To evaluate this, Pulsewave comminuted coal was subjected to petrographic analysis optical to determine the degree of phase liberation and to density gradient centrifugation (DGC) to achieve physical separations of liberated particles. (Optical analysis was subcontracted and the report of these analyses is attached as an appendix to this report.) DGC disperses particles in an aqueous medium of variable density from ~1.0 to ~1.6 g/cm³ that is then centrifuged to disperse all particles to their correct respective density. Minerals such as clays, calcite, silicates and pyrite have densities significantly greater than those of the organic constituents of coal. Hence, these phases can be physically separated by DGC and the separate fractions can then be recovered and analyzed to determine the partitioning of individual elements between the fractions.

If all phases are completely liberated (individual particles consist only of pure individual phases) then DGC separation will result in essentially perfect coal cleaning. Imperfect

liberation of mineral and organic phases will result in “carry over” of mineral matter into the organic phases and rejection of coal into the mineral phases. Pulsewave comminution is currently the best available technology for comminution of coal with effective liberation of individual phases. Hence, DGC separation of Pulsewave comminuted coal can be used to assess the currently achievable limit of physical coal cleaning. However, mineral phases smaller than the final particle size of the comminuted coal may still be present in individual particles. Hence, even with Pulsewave comminution, absolute mineral liberation may not be achievable for micron and sub-micron mineral phases. DGC density profiles of the six coals used for the present study are illustrated in Figures 5-10. In each case, the DGC profile of the Pulsewave comminuted coal is compared with the density profile for micronized coal prepared by fluid energy milling (FEM). This later technique produces a product with a mean particle size of ~ 1 micron and is the standard technique used for DGC studies.

It is a remarkable observation that in all cases the apparent density of the Pulsewave comminuted material is lower than the apparent density of the FEM milled material. This observation has not yet been satisfactorily explained and will be the subject of separate investigation. This phenomenon does not affect the data or conclusions described in the remainder of this report.

The data illustrated in Figures 5-10 reflect the density distributions of all particles with net particle densities less than 1.5 g/cm^3 . This includes all of the organic phases present in the coal, and mixed phase particles, i.e. those including phases incorporating unliberated mineral matter. Mineral particles and mixed phase particles with densities greater than 1.5 g/cm^3 collect as the traditional “sink” fraction on the centrifuge wall and are collected separately. Typically for fundamental studies, individual density fractions are collected and separately analyzed (this is the basis for isolation of pure individual coal macerals by DGC). For the purposes of this project, all fractions with density less than 1.5 g/cm^3 were combined as “float”. In this case “float” is analogous to cleaned coal and “sink” represents rejected material. Given that Pulsewave comminution achieves the highest degree of liberation of individual organic and mineral phases achievable with currently available technology, the distribution of sulfur, mercury and other elements between these fractions reflects the best achievable physical coal cleaning.

Recovery data for these separations are given in Table 4 below. These data show that in general about half the ash content present in the coal reports to the sink fraction. This is true even for coal C which was “cleaned” in conventional preparation plant prior to collection for these studies. (Coal C is the only cleaned coal used in this study.) Coal E, which contained the highest initial ash content gave a much higher separation, probably due to the presence of massive pyrite nodules (which are easily separated by both conventional and deep coal cleaning) in this sample.

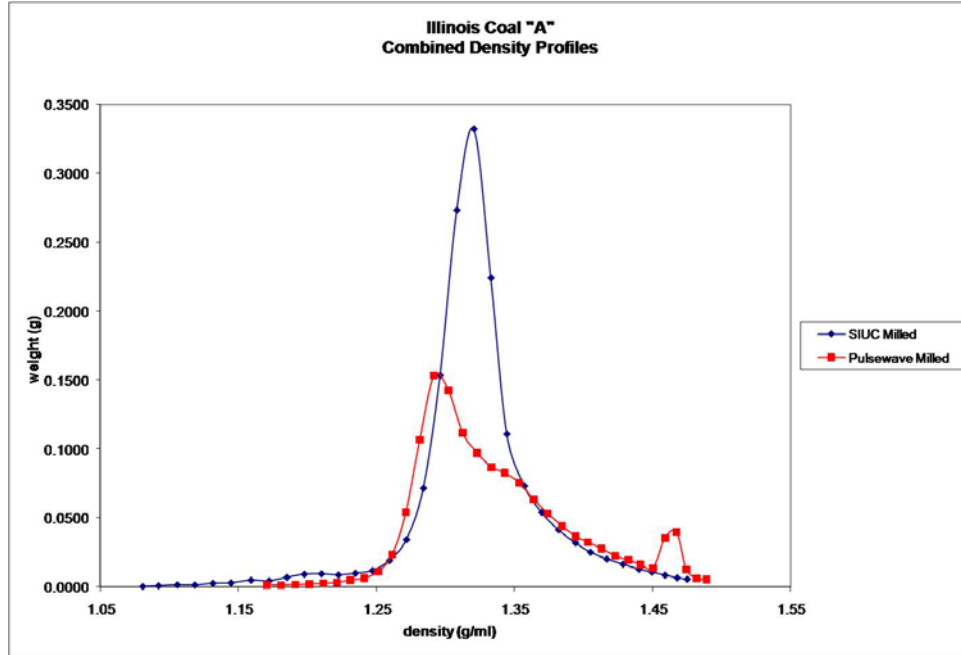


Figure 5. DGC Profiles for Coal A.

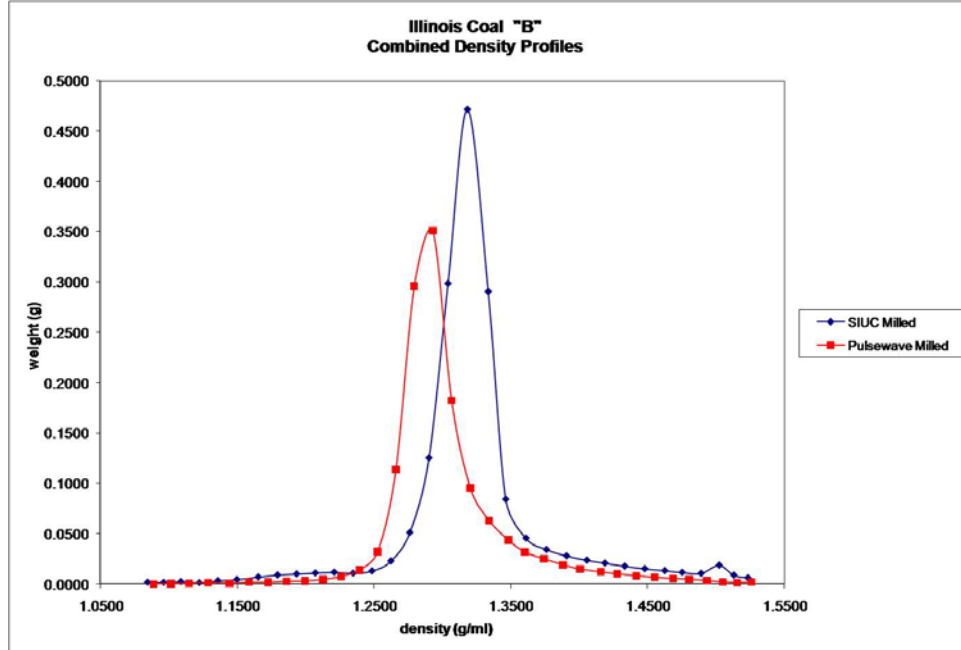


Figure 6. DGC Profiles for Illinois coal B.

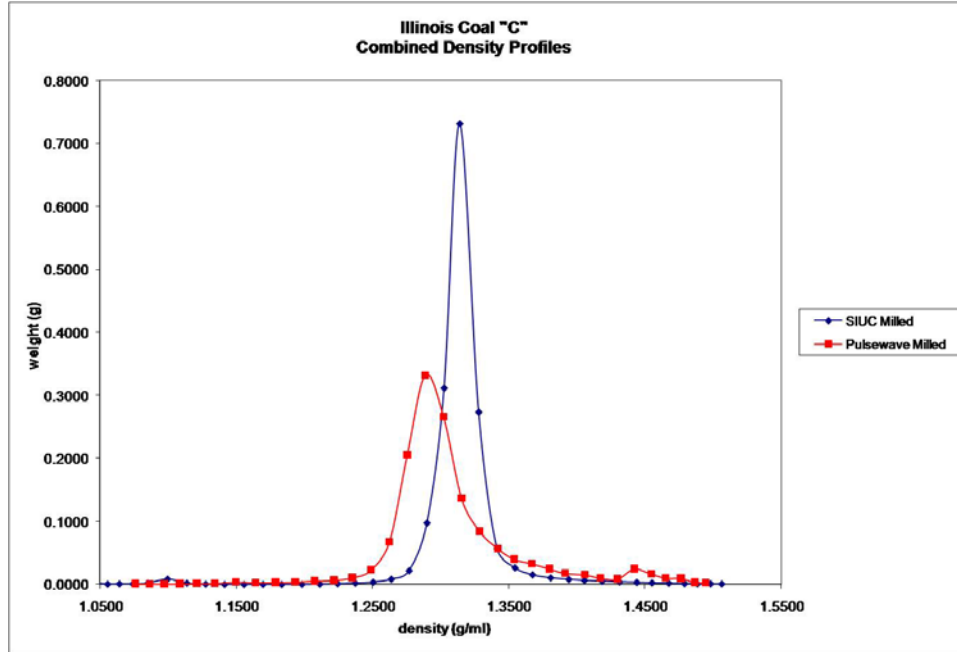


Figure 7. DGC Profiles for Illinois coal C.

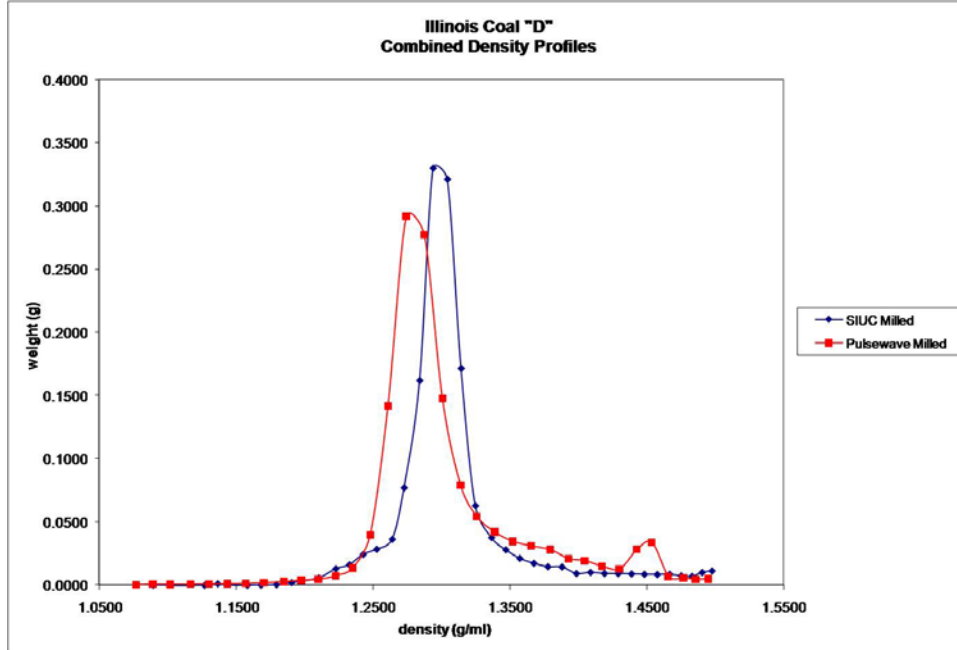


Figure 8. DGC Profiles for Illinois coal D.

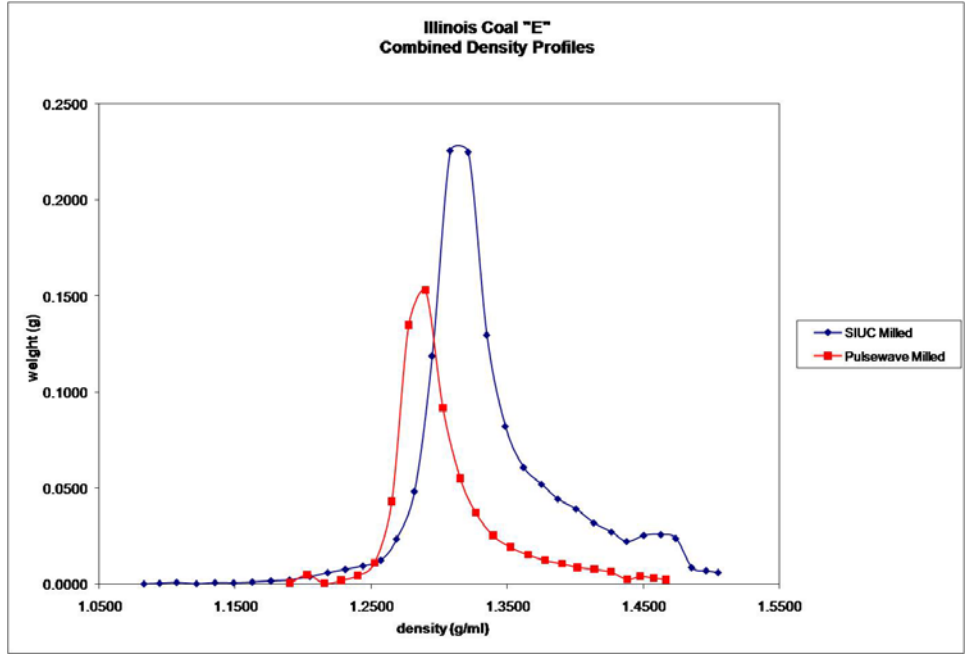


Figure 9. DGC Profiles for Illinois coal E.

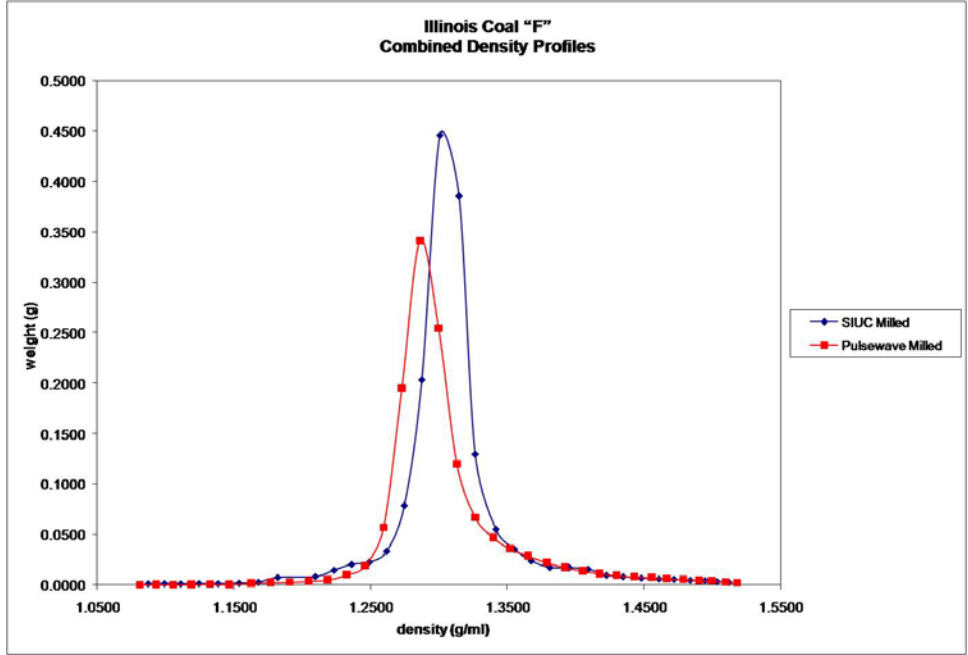


Figure 10. DGC Profiles for Illinois coal F.

Coal	Float, %	Sink. %
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A	90.1	9.9
B	92.3	7.7
C	97.6	2.4
D	91.1	8.9
E	71.0	29.0
F	89.8	10.2

Table 4. Recovery data for float/sink separations of Illinois coals used in the present study.

Results of analysis of these fractions for each of the six coals investigated are summarized in Tables 5 (raw coal), 6 (float) and 7 (sink), below.

Coal	Ash Wt%	C Wt%	H Wt%	N Wt%	O Wt%	S Wt%	Hg $\mu\text{g/g}$
A	18.84	59.38	4.13	1.57	11.87	4.21	0.09
B	13.39	68.85	4.56	1.61	9.53	2.07	0.10
C	7.59	73.99	4.95	1.86	10.01	1.60	0.08
D	17.00	67.96	4.39	1.46	6.17	3.02	0.29
E	34.08	52.11	3.64	1.11	6.05	3.01	0.20
F	19.85	59.88	3.98	1.45	13.05	1.78	0.09

Table 5. Ultimate analysis for raw coals used in the present studies. Data are reported on a dry basis.

Coal	Ash Wt%	% Ash reduction	C Wt%	H Wt%	N Wt%	O Wt%	S Wt%	Hg $\mu\text{g/g}$	% Hg removal
A	7.56	60	75.43	5.33	1.80	7.93	1.95	0.03	67
B	4.39	67	77.10	5.14	1.79	10.75	0.83	<0.02	80
C	5.26	31	75.61	5.08	1.88	11.26	0.91	<0.02	75
D	6.83	60	75.91	5.17	1.80	8.53	1.76	0.10	66
E	8.83	74	74.23	5.01	1.65	8.40	1.88	0.05	75
F	5.36	73	77.28	5.09	1.91	9.50	0.86	0.02	78

Table 6. Ultimate analysis for float (cleaned coals) used in the present studies. Data are reported on a dry basis.

Coal	Ash Wt%	C Wt%	H Wt%	N Wt%	O Wt%	S Wt%	Hg $\mu\text{g/g}$
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A	65.82	23.17	1.47	0.38	3.32	5.84	0.31
B	79.43	10.45	1.06	0.23	0.05	8.78	0.59
C	68.09	17.04	1.31	0.30	3.65	9.61	0.84
D	73.10	13.89	1.03	0.25	0.08	11.65	1.17
E	81.11	9.51	0.90	0.24	0.14	8.10	0.45
F	77.14	14.39	1.25	0.27	2.96	3.99	0.37

Table 7. Ultimate analysis for sink (rejected coal + mineral matter) recovered from coals used in the present studies. Data are reported on a dry basis.

The data given in Tables 5 and 6 indicate that while physical coal cleaning can substantially reduce sulfur levels, in most cases sulfur levels remain significant. This result is expected given that numerous previous studies have shown significant levels of organic sulfur in these coals and no amount of rejection of minerals by physical cleaning will affect this.

Data for mercury removal are also given in Tables 4-6 and are illustrated in Figures 11 and 12 below. The data illustrated in Figure 11 and described in Table 6 indicate that significant mercury reductions can be achieved by deep physical coal cleaning. For the six coals investigated in this study, mercury reductions of 66-80% were readily achievable. This is true even for coal that had been cleaned in a conventional coal preparation plant prior to Pulsewave comminution and laboratory separation (coal C). In all cases investigated, the sink fractions are mercury-enriched relative to the float fractions by factors of 10-40 fold, indicating that mercury is highly associated with the mineral phases present in the coal. This suggests that in theory, mercury reductions of 90% may be achievable by physical cleaning provided that these phases are effectively liberated from the organic phases and that physical separation of these phases can be effectively achieved. (For comparison, current conventional coal cleaning technology removes only approximately 55% of the mercury present in the raw coal.^{3,4}) However, at this point in time, using the best available commercial scale comminution technology and laboratory scale separation technology, this target was not achievable.

³ Crelling, (2005) Prediction of Mercury Removal Efficiencies with Current Coal Washing Practices. ICCI Report 04-1/2.1C-2.

⁴ Patel, (2007) Life Cycle Assessment of Mercury in Illinois Coals. ICCI Report 06-1/10.1A-7

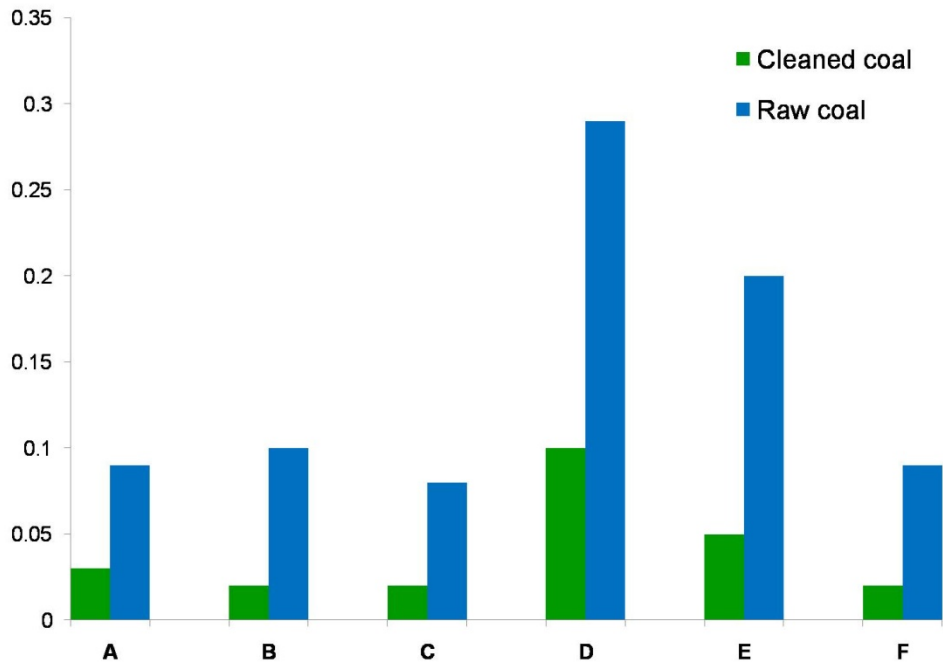


Figure 11. Mercury content of raw and cleaned (Pulsewave comminuted and DGC separated) coals. Data are reported in $\mu\text{g Hg}$ per g of coal.

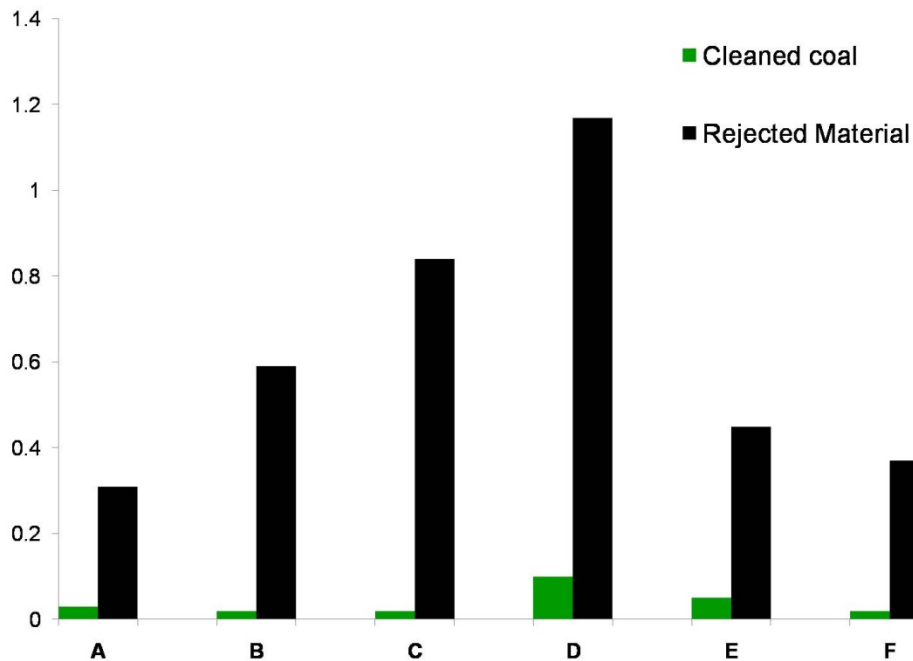


Figure 12. Mercury contents of the cleaned (Pulsewave comminuted and DGC separated) coals and the rejected “sink” fractions recovered, showing that mercury preferentially reports to the sink fractions indicating that it can be effectively removed by comminution and physical cleaning. Data are reported in $\mu\text{g Hg}$ per g of coal.

CONCLUSIONS AND RECOMMENDATIONS

Pulsewave resonance disintegration technology is highly effective for the comminution of Illinois coals. In addition to efficiently pulverizing the coal to particle sizes suitable for use with existing pulverized coal combustion (PCC) facilities, Pulsewave comminution effectively reduces coal moisture contents by 65-85%, resulting in an ~ 10% increase in thermal value. This technology is ready for deployment and should be demonstrated at full scale in a commercial PCC facility to:

- Quantify efficiency gains that may be achievable with technology.
- Gain commercial scale continuous operational experience.

Pulsewave comminution technology also gave promising results in coal cleaning applications. This aspect of this project was designed to measure the achievable limits of physical coal cleaning, using the best available comminution technology (Pulsewave resonance disintegration) to liberate coal from extraneous undesirable entrained mineral matter, coupled with laboratory scale density separation. The separation protocol used, density gradient separation, results in near-perfect partitioning of the coal/mineral particles based on density and hence, the degree of separation of organic and inorganic phases is limited only by the degree of liberation of these phases into discrete particles during comminution.

The data obtained show that for coals comminuted using Pulsewave technology, ash reductions of >60 were readily obtainable for raw coals. This is roughly double the ash reduction obtained with conventional coal cleaning. It was also found that mercury reports substantially to the sink fraction in physical cleaning. Mercury reductions of 66-80% were observed in all cases, even for coal that had been previously cleaned by conventional coal cleaning. The observation of 10-40 fold enrichment of mercury in the sink fractions (high density material “rejected” by density separation) confirm that deep physical coal cleaning, if adopted and reduced to commercial practice, will require comminution techniques that maximize liberation of mineral particles from the organic matrix. However, improved large scale techniques for the separation of fine particles of distinct densities are necessary before this approach can be used for coal cleaning on a commercial scale. Further studies of this approach utilizing different separation techniques should be considered in future.

DISCLAIMER STATEMENT

This report was prepared by Richard Sumner, Pulsewave LLC, with support, in part, by grants made possible by the Illinois Department of Commerce and Economic Opportunity through the Office of Coal Development and the Illinois Clean Coal Institute. Neither Richard Sumner, Pulsewave LLC, nor any of its subcontractors, nor the Illinois Department of Commerce and Economic Opportunity, Office of Coal Development, the Illinois Clean Coal Institute, nor any person acting on behalf of either:

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Appendix 1

Report on the Petrographic Analysis of Illinois Coal Samples

ICCI Project: 06-1/2.1A-2

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Background

As a part of ICCI grant 06-1/2.1A-2 (Application of Pulsewave Disintegration to Comminution, Drying, and Cleaning of Illinois Coal) the maceral composition and particle type distribution of the project samples were determined under contract by Professor Crelling. This was done because the maceral composition of a coal strongly influences the various properties of coal that are of industrial importance.

Procedures

The six samples of Illinois coal were designated A through F were prepared into pellets for petrographic analysis by point count analysis. The analyses were conducted in both white and blue light illumination by modified ASTM methods. In white light eight macerals, vitrinite, pseudovitrinite, fusinite, senifusinite, macrinite, semimacrinite, micrinite, and liptinite were determined by standard point count methods. All of the various liptinite macerals were counted as just liptinite. The individual liptinite macerals sporinite, cutinite, resinite, fluorinate, and liptodetrinite were determined in blue light illumination. This was done because experience and indeed the results all of the present

analyses show an increase in the amount of total liptinite observed.

While the vitrinite group macerals, normal vitrinite and pseudovitrinite are derived from coalified woody tissue they have different properties in petrography, density, combustion reactivity, coking reactivity, chemistry, etc. Under the microscope normal vitrinite appears to have been macerated usually and occurs as a matrix material in which other macerals are imbedded. Pseudovitrinite appears to have survived the coalification process intact and usually occurs as single particles that often show cell texture (see figure 1). The inertinite macerals are also derived from coalified woody tissue but they have been thermally altered by forest fires into charcoal. Under the microscope fusinite and semifusinite have open cell texture and an higher reflectance than the vitrinite macerals in the same coal. Fusinite always has a higher reflectance than semifusinite (see figure 2).

The liptinite macerals are derived from the waxy and resinous parts of plants. Sporinite, usually the most abundant liptinite maceral, is derived from the waxy spore exine. Cutinite is derived from the cuticle of plant leaves, roots, and stems and resinite is derived from coalified plant resins. Fluorinite is only reliably identified in fluorescent light and is thought to be derived from essential plant oils (see figures 3 and 4). Liptodetrinite is just fine detrital particles of liptinite too small to be differentiated.

In white light two sets of 500 points were counted on each sample and in fluorescent light two sets of 250 points were counted. In blue light all non liptinite macerals were counted as other. No voids, mounting media or mineral matter were included in the counts. The white light and blue light results were combined to yield a total analysis.

All of the samples were processed through the pulsewave device and petrographic pellets were also prepared. These were analyzed on the basis of counting randomly selected particles with each sample being analyzed twice with reproducibility of five mean %. Four particle types were counted. These were single maceral, single mineral, mixed maceral particles, and particles that contain both macerals and minerals. These particle types are illustrated in figure 5.

Results

Coal Sample Analysis

The results of the combined analyses are presented in Table 1. The results for each maceral are presented as volume % and totals for each maceral group are also presented. The pseudovitrinite - vitrinite ratio is also presented. The results for the Illinois coals D, E, and F typical of Illinois Basin coals with the vitrinite group macerals making up 75% more of the total. Coal A has a lower than normal total vitrinite and a corresponding increase in liptinite and inertinite macerals. Following a trend that has been noted in Appalachian Basin coals in this coal the presence of pseudovitrinite diminishes when the liptinite and inertinite macerals increase. Thus for coal A the pseudovitrinite - vitrinite ratio is very low at 0.22. Coal C is similar to the Illinois D, E, and F samples. However, sample B is exceptional in having an extremely high amount of pseudovitrinite present. Thus, the pseudovitrinite - vitrinite ratio is very high at 0.86. In fact this may be the

highest ratio ever recorded for a high volatile bituminous coal.

Pulsewave Particle Analysis

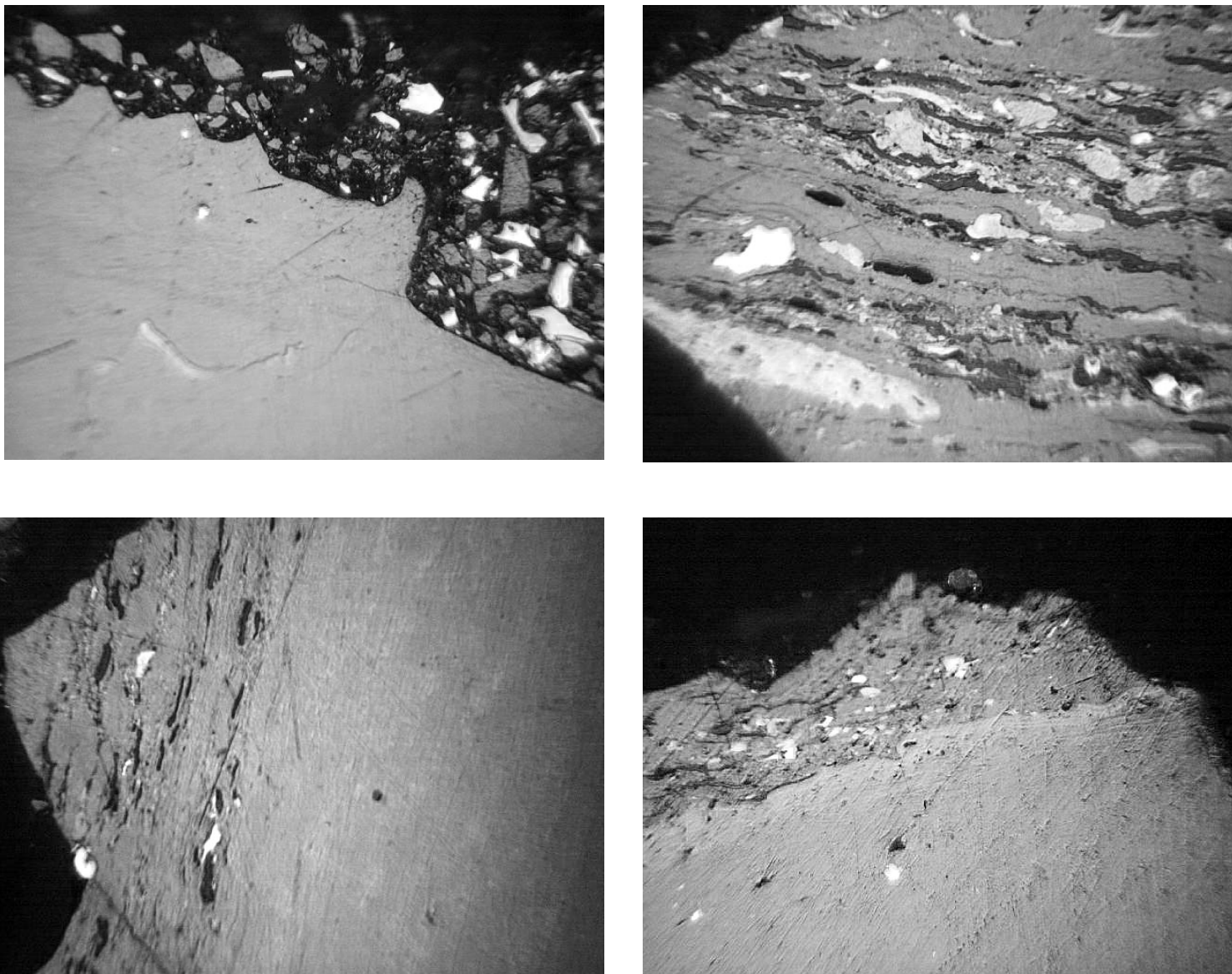
The results of the particle analysis are given in Table 2. Again the Illinois C, D, E, and F are similar with about 50% of the particles being single macerals. Although it was not recorded, almost all of the single maceral particles counted were vitrinite group macerals which is not surprising because are the most abundant macerals present. Likewise in these four coals mixed macerals made up about 25% of the total particles. In coal A which has the highest amount of both liptinite and inertinite macerals and the lowest amount of pseudovitrinite the mixed maceral percentage was much higher. Coal B had the highest amount of single maceral particles. This is consistent with its higher content of pseudovitrinite which usually contains no other included macerals (see figure 1).

Sample	Coal A	Coal B	Coal C	Coal D	Coal E	Coal F
Vitrinite	57.8	45.3	56.5	48.7	51.2	53.8
Pseudovitrinite	12.8	39.9	26.1	27.3	31.2	22.8
Fusinite	1.5	2.3	1.3	4.4	1.2	1.8
Semifusinite	11.9	2.7	2.5	4.8	6.6	8.8
Macrinite	0.0	0.0	0.0	0.0	0.1	0.0
Semimacrinite	0.0	0.0	0.0	0.1	0.0	0.5
Micrinite	2.4	0.7	0.2	0.2	0.0	0.1
Sporinite	11.8	8.6	10.2	14.0	8.6	11.2
Cutinite	0.2	0.8	0.6	0.8	0.0	0.2
Resinite	0.2	0.8	1.4	0.0	0.0	0.0
Fluorinite	1.4	0.0	0.4	0.0	0.1	0.8
Liptodetrinite	0.0	0.0	0.8	0.4	0	0.0
Total Liptinite	13.6	10.2	13.4	15.4	8.7	12.2
Total Vitrinite	70.6	84.2	82.6	75.1	82.4	76.7
Total Inertinite	15.8	5.7	4.0	9.5	8.9	11.1
Pseudovit/Vitrinite	0.22	0.86	0.46	0.56	0.61	0.42

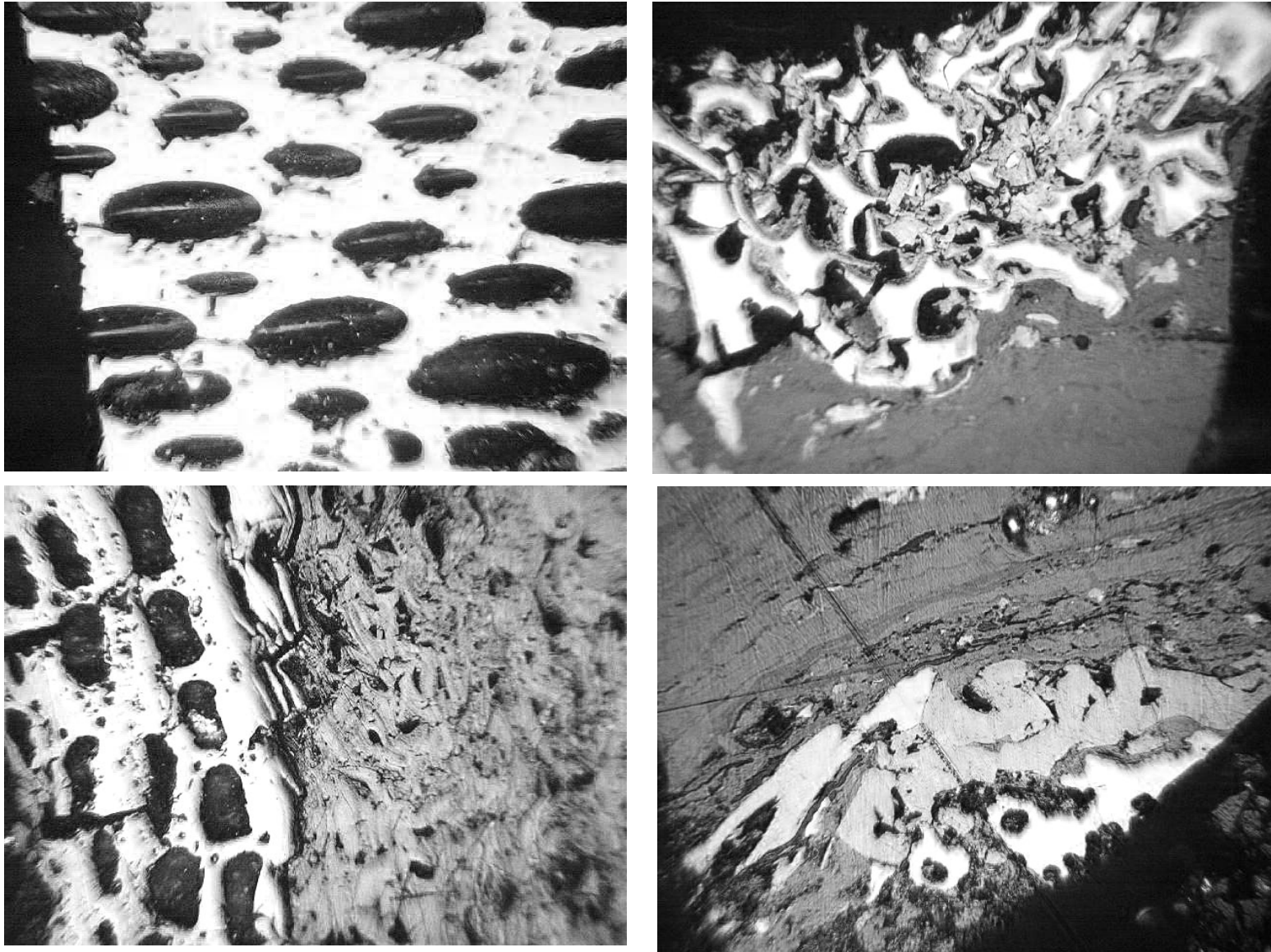
Appendix 1, Table 1, Results of petrographic analysis if coals investigated in the present study. Petrographic composition of initial whole coals.

Sample	Coal A	Coal B	Coal C	Coal D	Coal E	Coal F
Single Maceral	29	62	51	56	46	48
Mixed Maceral	48	22	29	18	27	29
Single Mineral	3	6	4	10	16	6
Mixed maceral/ Mineral	20	10	16	16	11	17

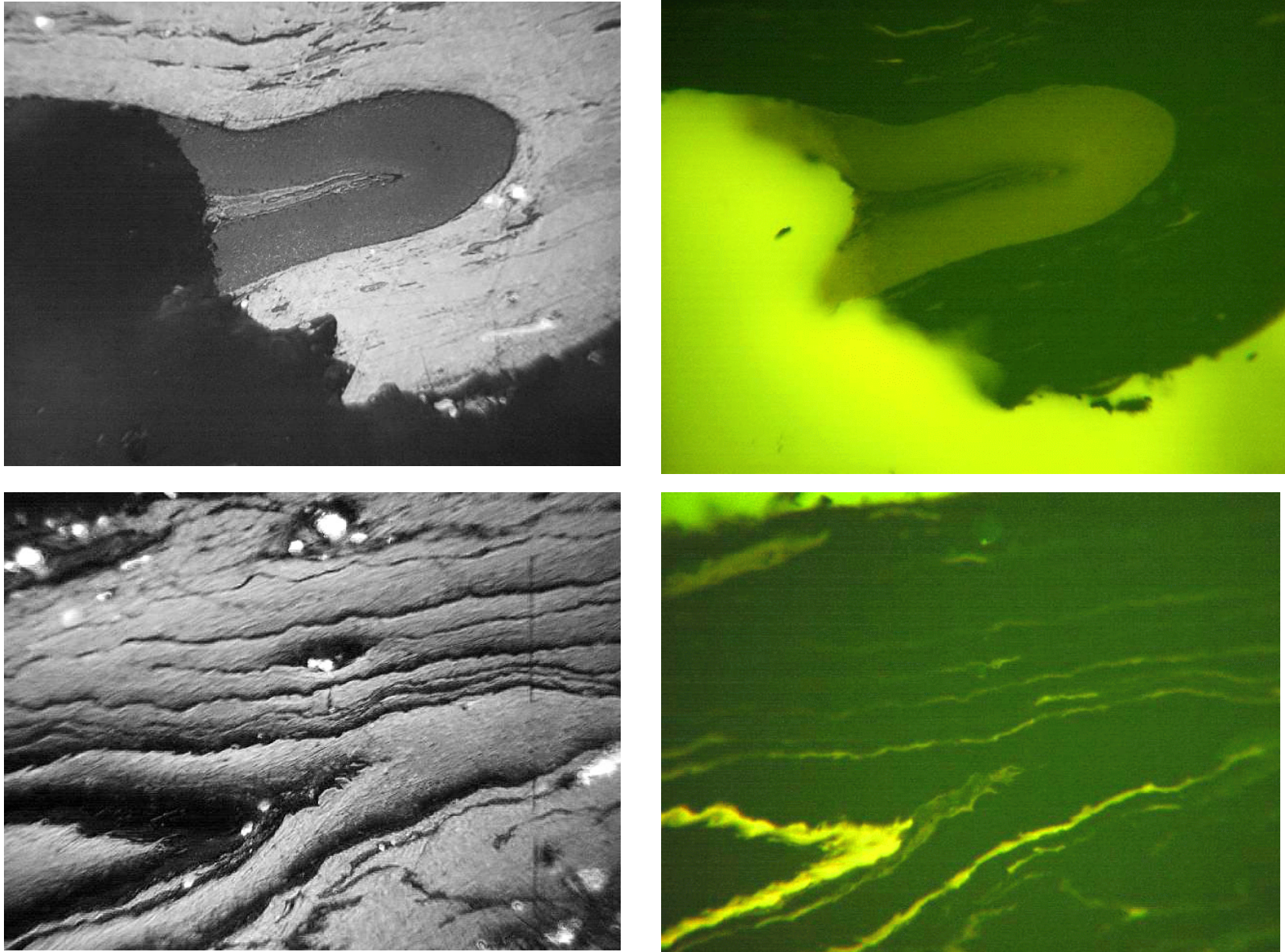
Appendix 1, Table 2, Maceral/maceral and maceral/mineral liberation for the six coals investigated as part of the present study.



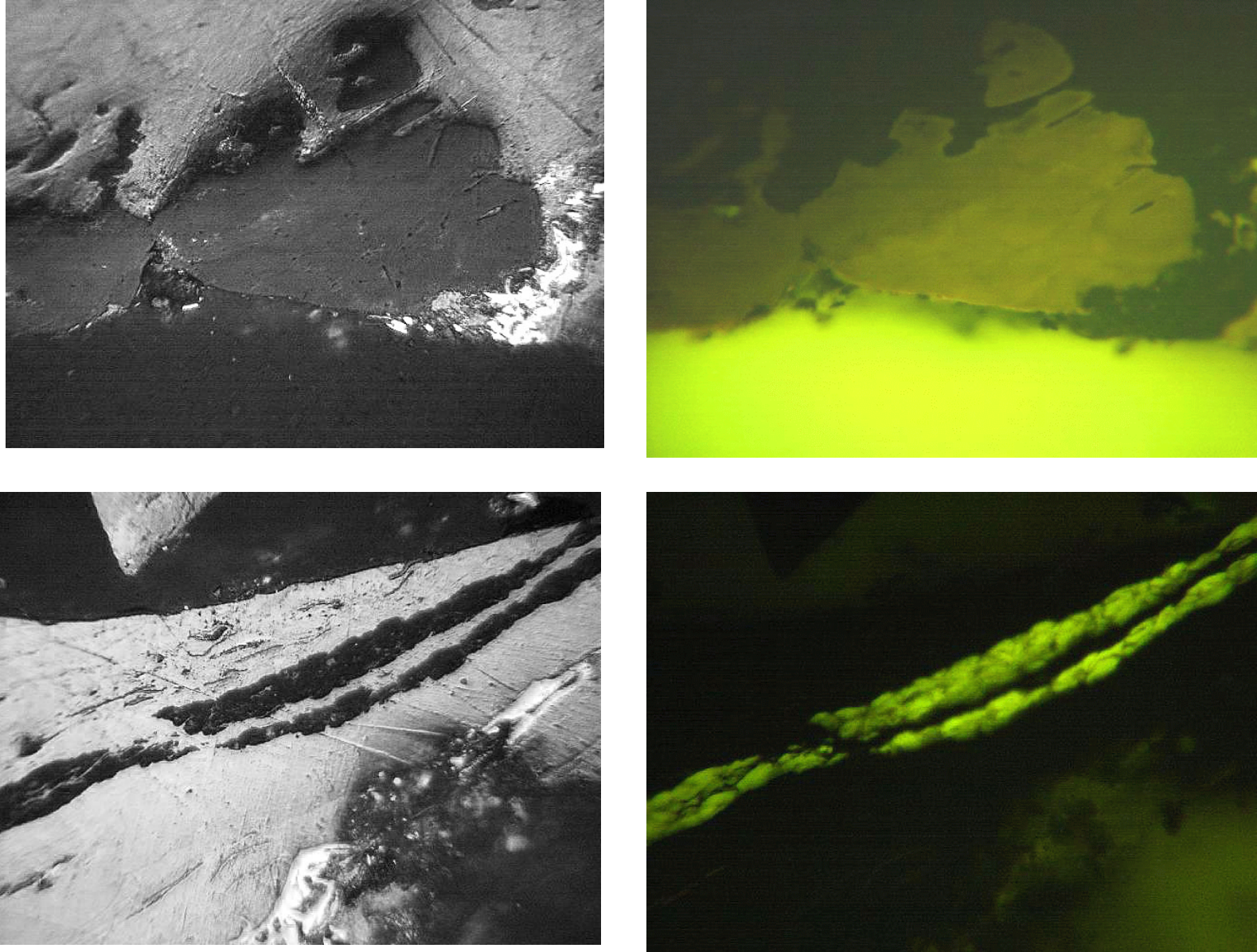
Appendix 1, Figure 1. Photomicrographs of vitrinite macerals: top left, pseudovitrinite - note the lack of inclusion; top right, normal vitrinite - note the inclusions of dark liptinite and bright inertinite; bottom left, normal vitrinite at left and pseudovitrinite at right; bottom right, normal vitrinite at top and pseudovitrinite at bottom. The width of the field (left to right) is about 300 micrometers.



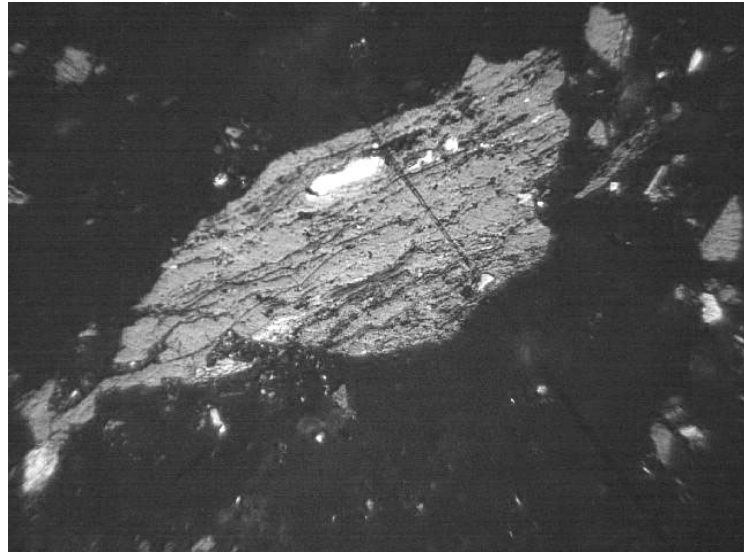
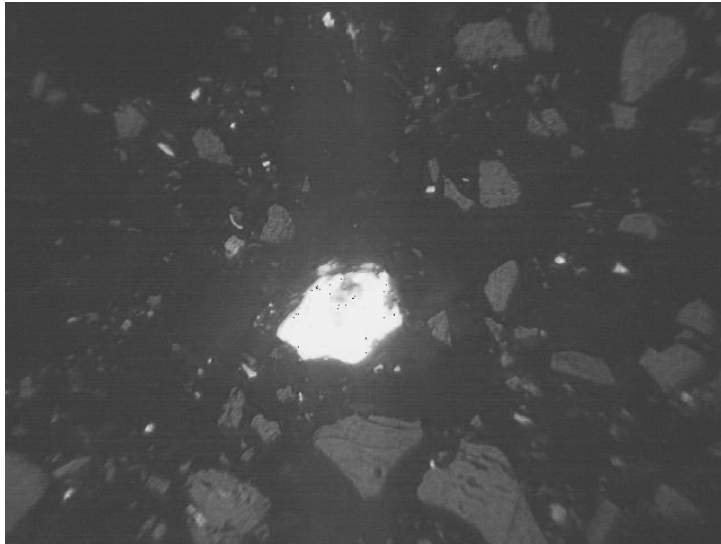
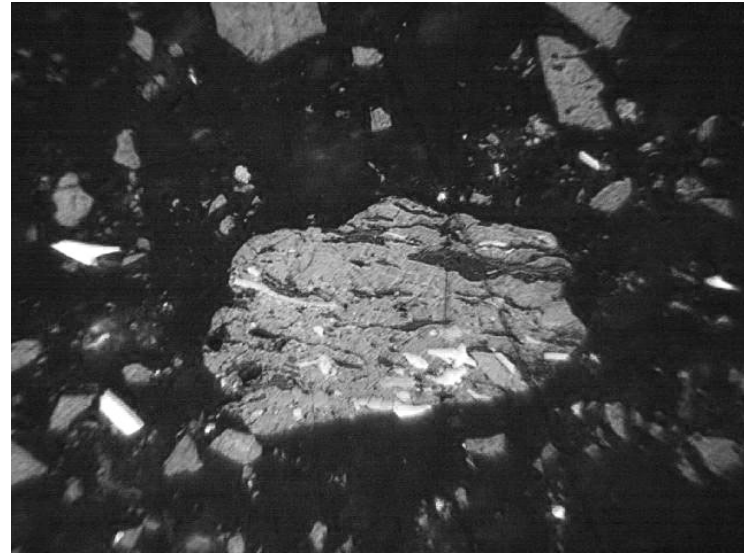
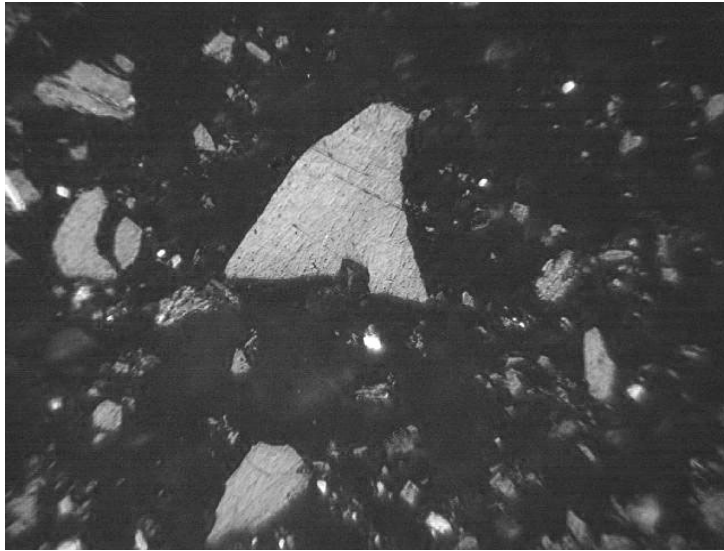
Appendix 1, Figure 2. Photomicrographs of inertinite macerals: top left, fusinite - note the high reflectance and open cell structure; top right, fusinite and vitrinite; bottom left, fusinite at left and semifusinite at right; bottom right, vitrinite at top, semifusinite at middle and fusinite at bottom. The width of the field (left to right) is about 300 micrometers.



Appendix 1, Figure 3. Photomicrographs of liptinite macerals: top left the dark structure is sporinite; top right, same field of sporinite fluorescing in blue light excitation; bottom left, dark linear structures are cutinite; bottom right, same field of cutinite fluorescing in blue light excitation. The width of the field (left to right) is about 300 micrometers.



Appendix 1, Figure 4. Photomicrographs of liptinite macerals: top left the dark structure is resinite; top right, same field of resinite fluorescing in blue light excitation; bottom left, dark linear structures are fluorinite; bottom right, same field of fluorinite fluorescing in blue light excitation. The width of the field (left to right) is about 300 micrometers.



Appendix 1, Figure 5. Photomicrographs of particle types counted: top left, particle of vitrinite counted as a single maceral particle; top right, a single mixed maceral particle; bottom left, particle of pyrite counted as a single mineral particle; bottom right, a mixed maceral particle with pyrite (bright spot) counter as mixed maceral/mineral particle. The width of the field (left to right) is about 300 micrometers.