Characterization of Coal Solvent Extraction Processes Using Petrographic, Chemical, and Physical Methods

FINAL REPORT

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ASTRACT

Nine bituminous coals were selected based on petrographic composition for solvent extraction in N-methyl pyrrolidone (NMP). The lowest rank coal had a vitrinite reflectance of 0.53 and the highest rank coal 1.53. All of the coals underwent laboratory extraction to determine which petrographic features were most amenable to solvent processing. In addition to detailed petrographic analyses of the feed coals, the NMP-insoluble residues and NMP-soluble extracts were also examined by a wide range of chemical and physical characterization methods. It was found that the liptinite and inertinite macerals and mineral matter were not significantly affected nor solubilized by the solvent. Vitrinite had the largest influence on yield and quality of the extract. Solvent etching of polished coal surfaces with NMP proved informative on describing the nature by which NMP attacks the organic coal portion. A combination of conventional test methods can be used to identify candidate coals. Coals most suitable to NMP extraction should be comprised predominantly of vitrinite with a mean maximum reflectance of about 1.1, should exhibit extensive Gieseler plasticity and Audibert-Arnu dilatometry, and should produce high free-swelling index buttons. Not only did the analysis show that rank parameters were important in establishing yields but also were influential in determining the optical microtexture of the cokes following simulated carbonization in the free-swelling index test: low rank bituminous coal extracts produced more isotropic cokes than extracts from high rank bituminous coals. Based on selection criteria developed in this project, a bituminous coal was chosen and successfully processed through the WVU coal extraction pilot plant.

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1 EXECUTIVE SUMMARY

The objective of the current proposal was to determine what petrographic, physical, and chemical characteristics of bituminous coals lead to the production of maximum extraction yield in N-methyl pyrrolidone (NMP). Detailed analyses of the feed coals and the examination of the coal maceral components that were solubilized by NMP were of great importance in establishing criteria for the selection of candidate coals. Normally petrographic data are determined on a volume percent basis by microscopic point techniques. Conversion of these data to a weight percent basis was made for comparison with the extract and residue weights. The characterization of extract quality and quantity as well as the coke textures and structures produced from carbonization of the extracts provided insight into the mechanism of NMP-solvent extraction.

Nine bituminous coals were chosen for extensive testing based on petrographic composition. The lowest rank coal had a vitrinite reflectance of 0.53 and the highest rank coal 1.53. All of the coals underwent laboratory extraction to determine which petrographic features were most amenable to solvent processing. It was found that coals with mean maximum vitrinite reflectance below about 1.0 were difficult to process by filtration. Some of these low rank bituminous coals appeared to generate solutions that were colloidal in nature, which resulted in blockage of the filter. Coals with vitrinite reflectance above about 1.20 processed well but produced moderate extraction efficiencies. Extraction of high rank coals produced low yields of soluble material. The coal most suitable for NMP extraction exhibited vitrinite reflectance of about 1.10 with yields approaching nearly 75wt% the mass of feed material.

In addition to detailed petrographic analyses of the feed coals, the NMP-insoluble residues and NMP-soluble extracts were also examined by a wide range of chemical and physical characterization methods. It was found that the liptinite and inertinite macerals and mineral matter were not significantly affected nor solubilized by NMP. Vitrinite had the largest influence on yield and quality of the NMP extract. Solvent etching polished coal surfaces with NMP showed that the lower rank coals were most affected by NMP in terms of physical alteration and staining of vitrinite.

Conventional test methods can be used to identify candidate coals. Coals most suitable to NMP extraction should be comprised predominantly of vitrinite with a mean maximum reflectance of about 1.1, should exhibit extensive Gieseler plasticity and Audibert-Arnu dilatation, and should produce high free-swelling indexes. Coals with these characteristics would generally fall within the hvA bituminous rank.

Analyses showed that rank parameters were important not only in establishing yields but also were influential in determining the optical microtexture of the cokes following simulated carbonization in the free-swelling index test: low rank bituminous coal extracts produced more isotropic cokes than extracts from high rank bituminous coals.

Based on selection criteria developed in this project, a bituminous coal was chosen to test whether laboratory-scale extraction processes could be verified on a larger level. This was successfully accomplished with the Kingwood bituminous coal through the WVU coal extraction pilot plant.

2 INTRODUCTION

The Consortium for Premium Carbon Products from Coal has as its mission the development of coal-based materials of high value. As prior CPCPC research efforts have shown, a wide range of carbon precursors is attainable by solvent extraction of coal. By manipulating solvent extraction conditions, materials suitable for binder pitches, anode cokes, carbon fibers, and isotropic graphites can be obtained. Potential applications of the solvent-extract technology that are attracting considerable attention are the use of coal extracts in the development of nuclear-grade graphite and low-cost cellular structures otherwise know as carbon foams.

Solvent processing under mild conditions in which the coal is not thermally degraded results in an essentially mineral-matter-free extract. These extracts often exhibit rheological and carbonization characteristics similar to that of the original coal. Little, if any, CPCPC-sponsored work has focused on the characterization of coals prior to, during, and after exhaustive solvent extraction or of the nature of the coal on extract quality and yield. Thus, this research effort sought to establish criteria necessary for the selection of optimal coals for solvent extraction and to develop an accurate model for predicting extract yields.

The coal extracts were produced in this project under mild conditions using solvent extraction technology developed at West Virginia University (WVU). Porosity development, pore size distribution, and chemical characterization of the coal and its insoluble residue were followed as a function of extraction efficiency. Coal Tech Petrographic Associates (CPA) examined the feed coals, extracts, and residues petrographically. In addition, the research groups co-operated on measuring pertinent rheological and other chemical properties. Moreover, petrographic techniques were used in determining the development of optical microstructure of the coal extracts from cokes made in the Free Swelling Index (FSI) determination. The resultant optical microtexture relating to coal rank, type, and extract yield were determined. Finally, a bituminous coal was chosen, based on the results of the research, for processing through the WVU coal extraction pilot plant.

3 BACKGROUND

3.1 Coal Formation Primer

Coal is a complex organic mixture consisting primarily of the elements carbon, hydrogen, oxygen, nitrogen, and sulfur. How these elements are bonded, their relative proportion, spatial distribution and conformation, give rise to a very nearly infinite variability in terms of reactivity and utility. The presence of inorganic matter is an additional facet affecting coal in all of its uses. Whether as discrete minerals or chemically associated with the organic portion of coal, mineral matter can be troublesome, by forming ash during combustion, or beneficial, by catalyzing reactions during direct coal liquefaction or gasification. Furthermore, since coal is solid, inherent porosity can trap or release gas, water, or solvent-soluble material, and influence accessibility of reagents. The interplay among the organic, inorganic, and physical components ultimately determines many of the applications for which coal can be used.

It certainly is not possible to cover all aspects of coal formation, structure, or analyses within the confines of this project. Nonetheless, despite the lack of depth covered here, it is pertinent to address various aspects on the nature of coal, especially as they pertain to solvent extraction. For those so inclined, a wealth of detailed information on a wide range of coal topics can be found in earlier texts of Lowry [1,2], Elliot [3], and Stach [4], more recently van Krevelen [5], and in the prevalent refereed journals. An updated review on solvent extraction of coal is also available by Dilo Paul [6].

Since the crust is not fixed, the Earth is a dynamic celestial body and its surface features continually change over eons of time. Imagine that the continental United States was at one time much closer to the equator but because of plate tectonics, now resides in northern latitudes. While the continents were gradually shifting and environments and climates slowly changing, the prevailing vegetation evolved, from primitive mosses and horsetails, to more modern spore producers, and eventually to seed producing plants. The basic organic compounds comprising all of these plants were probably similar chemically, but their proportions could be quite different: the building blocks of plants—cellulose, lignin, waxes, and resins—are variable in quantity from species to species and from era to era. This is important because the composition of plant material going into forming coal could have an effect on its properties [7].

Although the process of plant evolution proceeded uninterrupted, the conditions necessary for the deposition of coal were specialized and sporadic on a geological time scale. When a plant dies and lies on the forest floor, the usual outcome is decay, resulting in the complete conversion of tissues and specialized structures into carbon dioxide and water. To form coal, however, the dead plant material should be deposited in swampy or boggy conditions. Under water, attack of the dead plants by oxygen and microorganisms can still occur, but is greatly retarded. In this environment, the plant materials become peat, which is enriched in carbon compared to the original organic input, because of the loss of methane, carbon dioxide, and water. Additional plants could continue to grow, die, and deposit in the swamp intermittently or continually for many years until thin seams or thick deposits are made. Eventually, the peat becomes covered with sediment and forced by geological processes to subside.

Coalification is a two-part process that plant materials undergo on their course toward coal. The first, the biochemical stage, ends by the time peat is formed. The second, the geochemical stage, depending on pressure, temperature, and time, transforms the peat material into either lignite, subbituminous coal, bituminous coal, or ultimately anthracite. Lignite can be considered an “immature” coal since it has undergone the least metamorphosis and, at the other extreme, anthracite the most “mature” coal.

Differences in plant material and the conditions during decomposition and burial result in petrographic entities known as macerals. Macerals are fossilized plant remains, distinguishable by observation under the optical microscope, and are categorized into three main groups: vitrinite (huminite in low rank coals), liptinite, and inertinite. Within each main group there are a number of other maceral components that can be identified by their size, morphology, and reflective properties: it is abundantly apparent that coal is not homogeneous. Neavel [8] said the assemblage of macerals in coal is analogous to a fruitcake, as is evident in Figure 1.

The predominant macerals fall within the vitrinite group and exert the greatest influence on the behavior of coal during its utilization. Vitrinite is derived primarily from woody antecedents. Liptinite is higher in hydrogen content than vitrinite and is comprised of more-specialized fossilized plant remains such as spores, pollen, cuticles, and resins. Inertinite is higher in carbon content than vitrinite and often displays a charcoal-like structure. Although occurring in lesser amounts than vitrinite, the affects of liptinite and inertinite must not be ignored or underestimated.

Upon moving from the low-rank lignite to the high-rank anthracite, many chemical and physical characteristics change in a more or less systematic manner [9,10]. Correlations between coal behavior and extent of coalification are very useful in most applications, especially in the establishment of a coal classification system. Classifying coals helps in determining whether carbonization or gasification, for example, would be appropriate. Attempts to place coals into certain groups or similar categories have evolved since the early 19th century in parallel with the growth of the industrial revolution, which became increasingly reliant on worldwide-coal trade for fuel. Some of the classification tests are based on 1) chemical analysis, 2) thermoplastic behavior, or 3) petrographic analysis. The Economic Commission for Europe Coal Committee developed a comprehensive system that involves a 14-digit code based on the results of all three tests [11].

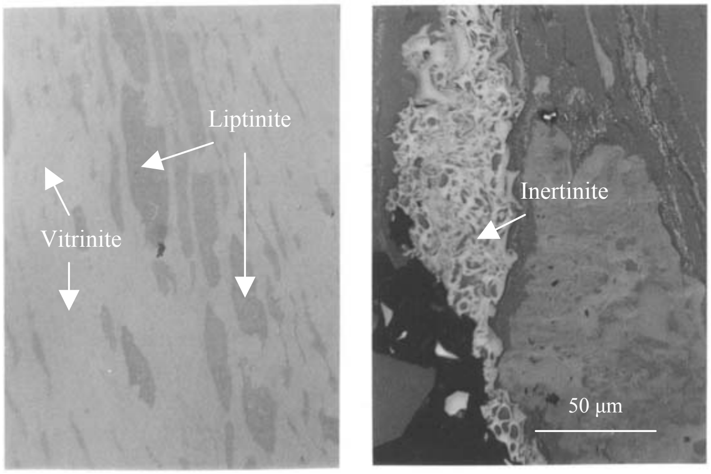
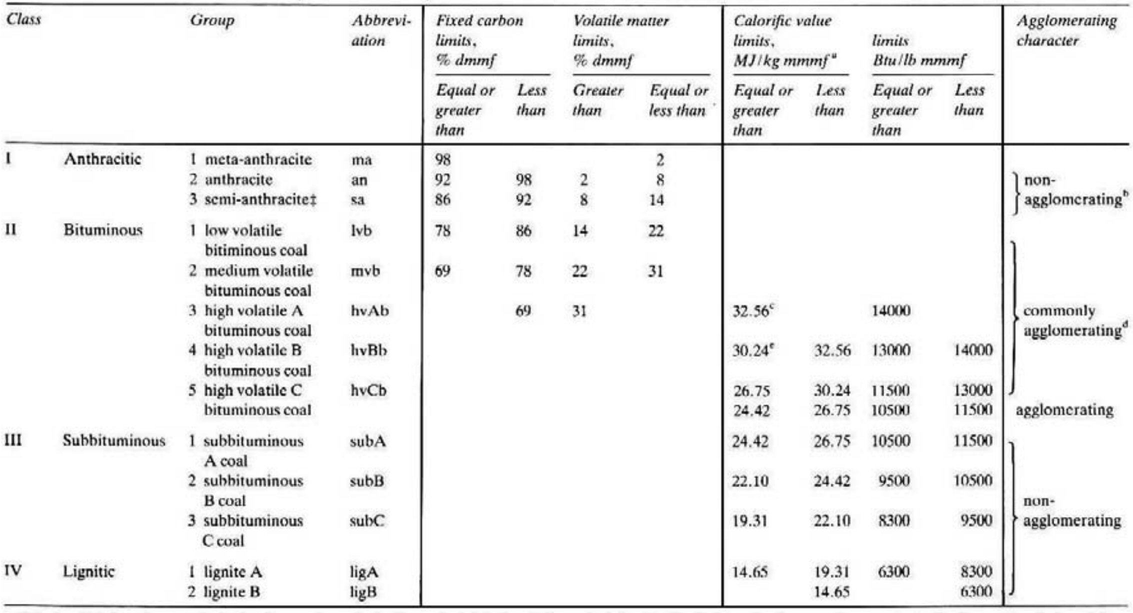


Figure 1. Photomicrographs of main maceral groups in a bituminous coal.

In the United States, coals are classified according to rank as defined by the American Society for Testing Materials (ASTM D388). The U.S. classification is based on volatile matter content or calorific value, as indicated in Table 1.

Table 1. Coal classification according to ASTM.



Class		Group	Abbreviation	Fixed carbon limits, % dmmf		Volatile matter limits, % dmmf		Calorific value limits, MJ/kg mmmf[a]		limits Btu/lb mmmf		Agglomerating character
				Equal or greater than	Less than	Greater than	Equal or less than	Equal or greater than	Less than	Equal or greater than	Less than	
I	Anthracitic	1 meta-anthracite	ma	98			2					non-agglomerating[b]
		2 anthracite	an	92	98	2	8					
		3 semi-anthracite‡	sa	86	92	8	14					
II	Bituminous	1 low volatile bitiminous coal	lvb	78	86	14	22					commonly agglomerating[d]
		2 medium volatile bituminous coal	mvb	69	78	22	31					
		3 high volatile A bituminous coal	hvAb		69	31		32.56[c]		14000		
		4 high volatile B bituminous coal	hvBb					30.24[e]	32.56	13000	14000	
		5 high volatile C bituminous coal	hvCb					26.75	30.24	11500	13000	
								24.42	26.75	10500	11500	agglomerating
III	Subbituminous	1 subbituminous A coal	subA					24.42	26.75	10500	11500	non-agglomerating
		2 subbituminous B coal	subB					22.10	24.42	9500	10500	
		3 subbituminous C coal	subC					19.31	22.10	8300	9500	
IV	Lignitic	1 lignite A	ligA					14.65	19.31	6300	8300	
		2 lignite B	ligB						14.65		6300	

Several physicochemical tendencies are evident on going from low rank coal to high rank coal. Some of these trends include increases in carbon content; decreases in oxygen, moisture, and volatile matter content; and increases in maceral reflectance, molecular weight, and aromaticity. For coals with about 87-89wt% carbon, a minimum in true density, surface area, and porosity is measured, and a maximum in extractable material and fluidity is obtained.

3.2 Action of Solvents on Coal

Solvent extraction under mild conditions has been and continues to be actively pursued, primarily as a tool to probe the molecular properties and structure of coal and, in some instances, for commercial applications. Research shows that the effectiveness of solvents for coal extraction is influenced by coal type, rank, and petrographic composition, and by the chemical nature of the solvent.

Whether the vitrinite in coal is a micellar complex [12] or three-dimensional network [13] remains to be proven, and whether solvent effectiveness can be explained entirely by solubility parameter [14], donor-accepter number [15], hydrogen-bonding [16], solvent-synergism [17], or other associative forces [18] continues to be investigated. These fundamental aspects on coal-solvent interactions are beyond the scope of this project.

Though still relevant today, Oele et al. more than fifty years ago classified solvents used to extract coal into four groups [19]. In general, the efficiency of solvents in this system is closely related to temperature. Solvents in the first category, *non-specific extraction*, extract only a few percent of the coal at temperatures below about 100°C. These solvents (for example benzene, hexane, carbon tetrachloride, and ethanol) extract only a few percent coal thought to be primarily resins and waxes. In *specific extraction* the coal material solubilized can typically amount to about 20-40wt% at temperatures up to about 200°C. Many of these solvents are nucleophilic, have electron donor-acceptor properties and high internal pressure. Pyridine, quinoline, and N-methyl pyrrolidone are examples of solvents used for specific extraction. It is noteworthy that remarkable ambient-temperature extraction efficiencies have been reported by Iino with mixed carbon disulfide—N-methyl pyrrolidone solutions [20]. During *extractive disintegration* temperatures approaching the thermal decomposition of coal, up to 350°C, can be encountered. Under these conditions it can be anticipated that solvent components which can redistribute hydrogen from and to reactive coal components by a shuttling mechanism appear to be most effective [21-22]. For example, extraction of a bituminous coal with phenanthrene (boiling point 340°C) produced 95wt% extract yield, but extraction of the same coal in anthracene (boiling point 354°C) produced only 24wt% extract [23]. Since the two solvents are isomers of similar boiling point, it was suggested that phenanthrene was more active than anthracene in hydrogen exchange reactions [24]. The *extractive chemical disintegration* process can be called direct coal liquefaction. Here, solvents rich in hydroaromatic components are especially suited in extracting nearly all of the reactive coal macerals. These types of solvents actively participant chemically in bond breakage and stabilization, are consumed or structurally changed, and are normally used at temperatures considerably in excess of 300°C. One should be mindful, however, that there could be no clear operational or mechanistic distinction between extractive disintegration and extractive chemical disintegration processes.

3.3 Role of Extracts on Thermoplastic Properties

When certain bituminous coals are heated, they soften and become fluid commensurate with the evolution of gas and tar [25-26]. The plastic behavior is transient and the mass eventually thickens, swells, and fuses to form a porous solid or coke. This phenomenon is of the utmost importance to the production of metallurgical coke, and in other processes sensitive to coal caking and agglomeration. Because of the impact of plastic behavior on industrial processes, a great deal of early research effort was directed toward understanding coal composition and the relation of soluble extracts to the "coking" principle [27-29]. The plastic behavior in coals has been explained by the hypothesis that the development of plasticity is a transient, in situ, hydrogen donor process [30]. The solvent-soluble material contains more hydrogen than the parent coal, and is thought to be responsible for solvation and hydrogen stabilization of the molecular units in coal as they become mobile with increasing temperature. It is believed that the stability of the metaplast produced by coking coals during their softening and resolidification, which is emulated in their plastic range, is responsible for the quality and quantity of extract produced. These extractable components are also influential in other processes such as direct coal liquefaction [31].

It is well established that there is a correspondence between the amount of material extractable from coal using specific solvents and rank, as shown in Figure 2. The quantity of extracts in pyridine increases as the carbon content increases, peaks around 86wt% carbon, and then drops precipitously.

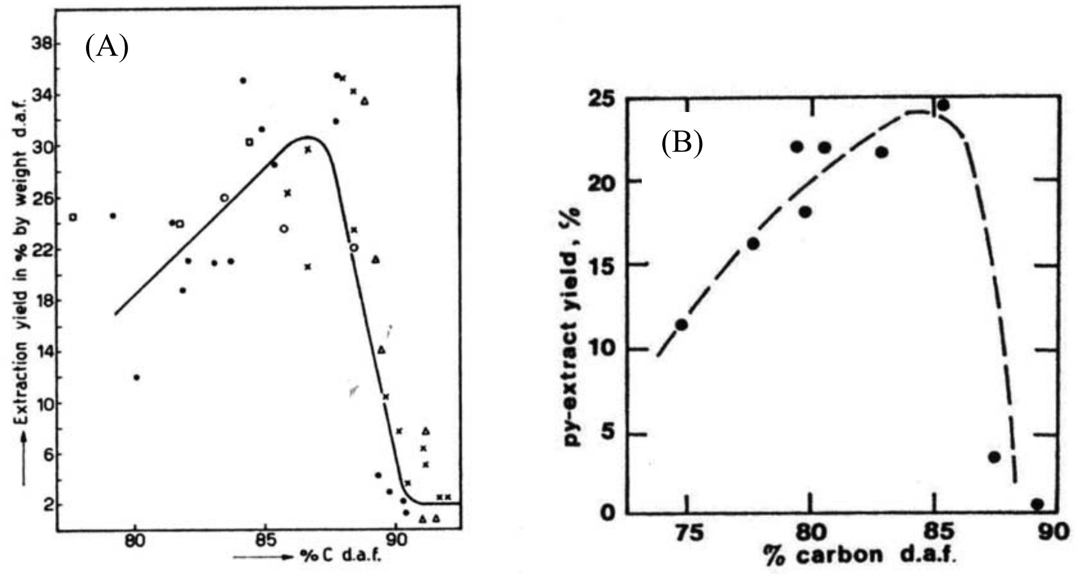


Figure 2. Yield of pyridine extracts vs. coal rank: A from ref. 35; B from ref. 36.

Furthermore, there also is a correspondence or relationship between the amount of material extractable from bituminous coal and thermoplascitity, see Figure 3. The significance of the extracts on plasticity is made clear when extracts from good coking coals are blended with non-coking coals to induce fluidity [32-33], and extracting prime coking coals leaves a residue devoid of plastic behavior [34].

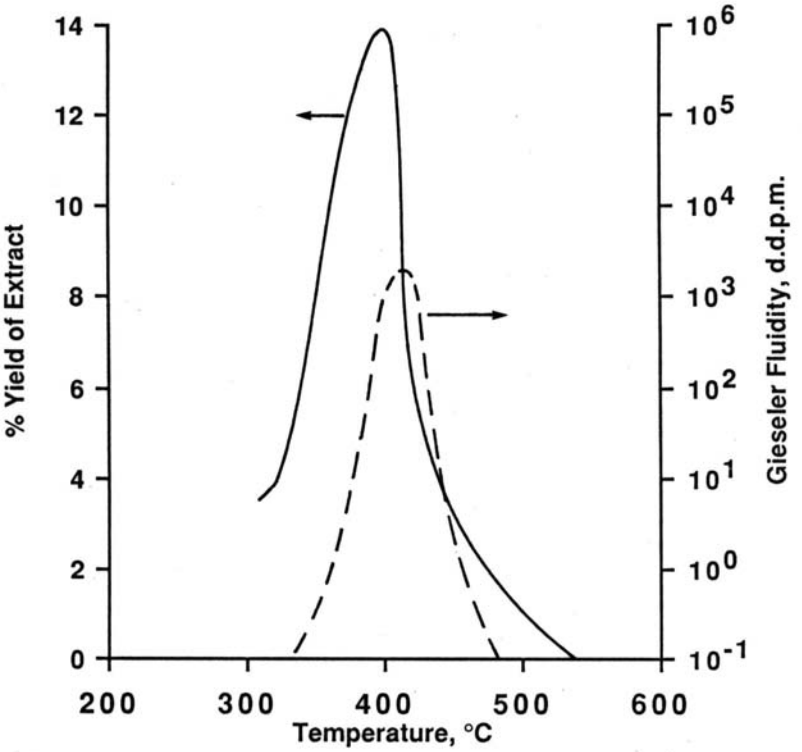


Figure 3. Variation of extract yield and Gieseler fluidity [37].

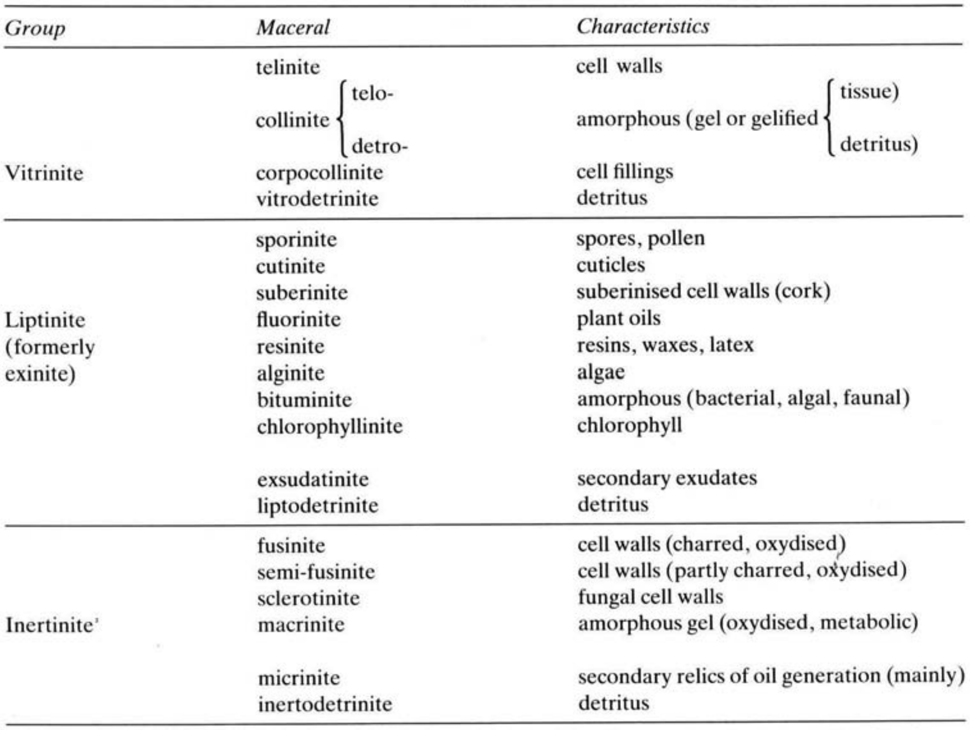
3.4 Optical Microscopy as Applied to Coal, Coal Products, and Coke/Carbon Forms

Coals are bioclastic sedimentary solid fossils of plant origin that consist of macerals, minerals, water, and gas. In addition to variations in rank, coals also vary in type, from sapropelic to banded, and in grade, from carbonaceous rock to relatively pure organic material. The one technique that uniquely fingerprints coal in terms of rank, type, and grade is optical microscopy. Petrographic analysis of coal has become immensely important on a worldwide scale in selection of coals for development, utilization, and marketing. As such, the American Society for Testing Materials has developed standards for preparing coal samples for microscopical analysis by reflected light [38], for megascopic description of coal seams and microscopical determination of volume percent of coal components [39], and for the microscopical determination of the reflectance of organic components of coal [40].

Petrology is that branch of geological science that deals with the study of rocks in the earth's crust. Petrography of coal is the descriptive part of petrology, and deals with the characterization and designation of coal as a rock. The simplest method of characterization can be accomplished by casual observation. To the unaided eye coal can appear either banded or non-banded. Examples of the latter include cannel and boghead coals: sporinite is predominant in cannel coal and algonite in boghead coal. However, we usually are more familiar with the banded coal varieties, in which a systematic effort to describe the visible bands, or lithotypes (literally "rocktype"), began to emerge in the 1920's. Differences in the appearance, luster, and hardness identify lithotypes as 1) vitrain: a black, lustrous, relatively homogeneous bands in bituminous coals that break conchoidally; 2) clarain: a finely banded, lustrous black material, with a silky texture, that breaks horizontally; 3) durain: a dull, matt-like, granular appearing, black solid material; 4) fusain: a dull, generally lenticular, friable solid that resembles charcoal: 5) duroclarain and clarodurain: intermediate between clarain and durain. Presently coal petrography centers mostly around microscopic techniques to identify and quantify the individual fossilized plant components which makeup the lithotypes and, since 1957, to use spectral reflectance in identifying coal according to rank.

When light strikes a flat surface of coal, a portion, not all, is reflected. The nature and intensity of the reflection can provide information on the chemical and physical state of coal [42]. Of the macerals in the vitrinite group, shown in Table 2, collinite is selected to determine coal rank because of its preponderance and homogeneity. The reflectance of all macerals differs and changes with rank, but the reflectance of collinite changes in a very regular fashion with changes in rank, see Figure 4. A collinite reflectance of R = 1.5, for example, means that 1.5 percent of the incident light is reflected: the magnitude of only 1.5 means coals absorb a large portion of visible light. Reflectance values are often reported as V-types. For instance, V-6 includes all of the vitrinite with a reflectance of 0.60 through 0.69 percent.

Table 2. Maceral groups and macerals in hard coals (from ref. 5, pg. 118).



Group	*Maceral*	*Characteristics*
Vitrinite	telinite	cell walls
	collinite { telo- detro-	amorphous (gel or gelified { tissue) detritus)
	corpocollinite	cell fillings
	vitrodetrinite	detritus
Liptinite (formerly exinite)	sporinite	spores, pollen
	cutinite	cuticles
	suberinite	suberinised cell walls (cork)
	fluorinite	plant oils
	resinite	resins, waxes, latex
	alginite	algae
	bituminite	amorphous (bacterial, algal, faunal)
	chlorophyllinite	chlorophyll
	exsudatinite	secondary exudates
	liptodetrinite	detritus
Inertinite'	fusinite	cell walls (charred, oxydised)
	semi-fusinite	cell walls (partly charred, oxydised)
	sclerotinite	fungal cell walls
	macrinite	amorphous gel (oxydised, metabolic)
	micrinite	secondary relics of oil generation (mainly)
	inertodetrinite	detritus

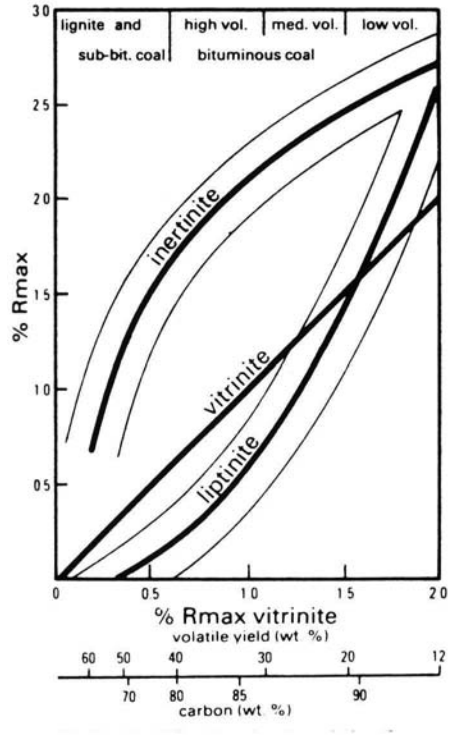


Figure 4. Reflectance of macerals vs. rank (from ref. 5, pg. 373).

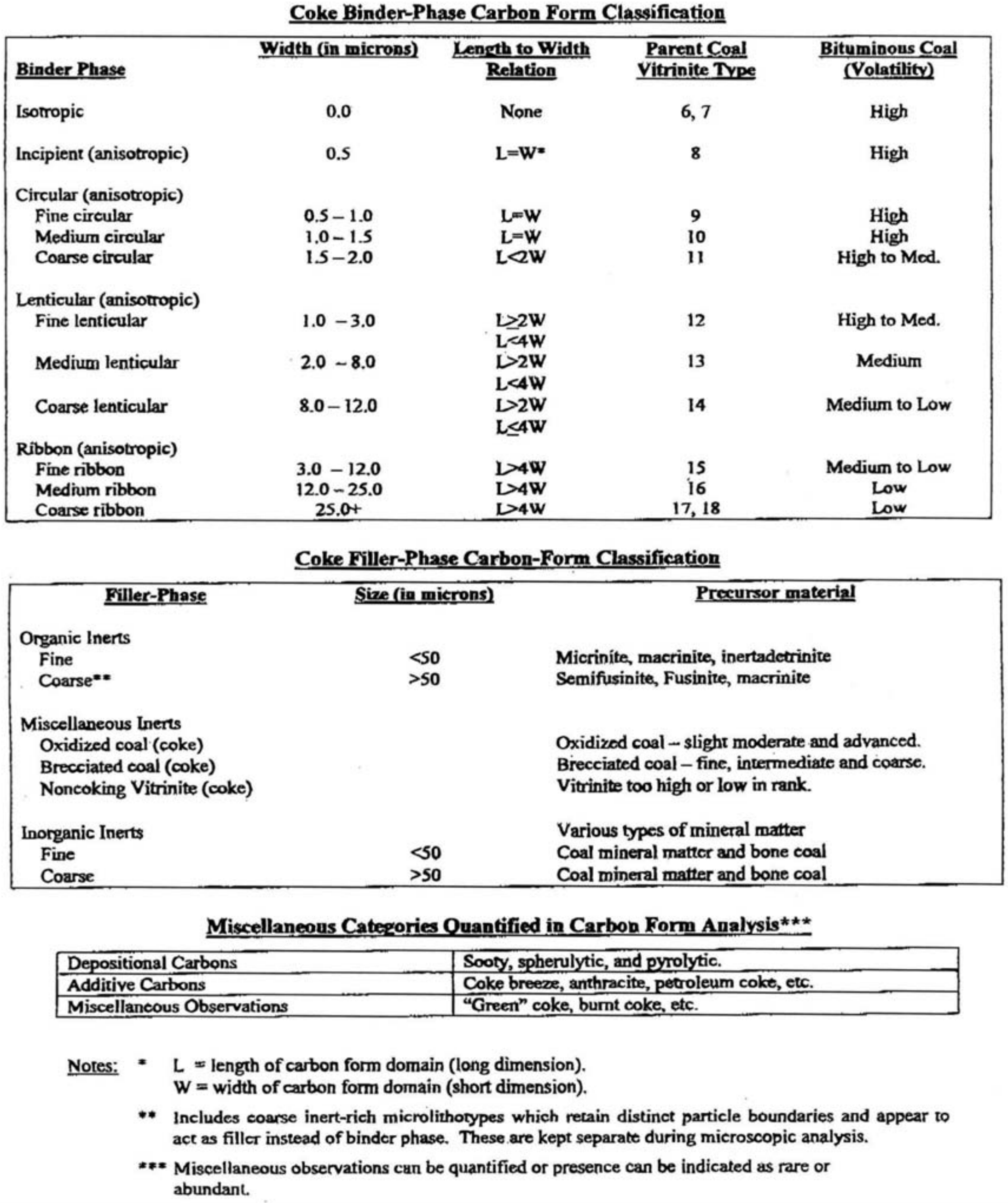
In the maceral analysis of coal, the sample is prepared by mounting a representative sample of minus 20 mesh coal in a plastic binder and forming the mixture into a cylindrical pellet under pressure to exclude air. After the plastic has cured, the surface of the pellet containing the coal is ground and polished to a mirror-smooth finish for microscopic examination. Magnifications of about 300 to 700 with oil immersion objectives are commonly used. The specimen is observed through an ocular equipped with a measuring device, such as a whipple disc, and the stage moved in fixed incremental distances in the x and y directions to permit counting.

In conducting a quantitative analysis the sample is moved in fixed increments or fields of at least one-half the size of the largest particle in the sample. The fields are spaced along a traverse or line through the sample. Parallel traverses are run through the sample until sufficient materials are identified. A uniform sampling grid is used to sample randomly dispersed particles. Each field along a traverse is sampled identifying materials that occur at the intersections of the corner lines in a rectangular grid in the ocular. In general, two sets of 500 macerals are counted. If the separate sets are near duplicates then the 1000 points are averaged and reported as volume or area percentages. If the duplicate 500 points counted do not reproduce then additional points are counted up to 2000 points.

Petrographic techniques are also applicable in the characterization of carbon and coke forms obtained from coal [43]. The cokes from coals have a variety of microstructures that are optically distinct in polarized light. The graphitizability, electrical resistivity, reactivity to CO_2 at elevated temperatures, and strength of carbons are related to the optical properties of the coke carbon forms, which vary in amount, size, and degree of anisotropy. The system of coke microscopy used in this project is shown in Table 3 and some of the forms that cokes exhibit are shown in Figure 5.

Cokes from coals consist of binder and filler phases. Binder phase carbons are produced from the reactive macerals vitrinite, exinite, resinite, and reactive semifusinite. All reactive macerals soften during carbonization. Filler phase carbon forms are derived from inert semifusinite, fusinite, possibly micrinite, macrinite, and inertodetrinite macerals, which do not soften appreciably during carbonization. The binder and filler phase coke analyses are combined to complete the petrographic examination. The relation of coal rank based on vitrinite reflectance to coke carbon forms (microstructures) in the binder phase is also shown in Table 3.

Table 3. A system of coke microscopy.



Coke Binder-Phase Carbon Form Classification

Binder Phase	Width (in microns)	Length to Width Relation	Parent Coal Vitrinite Type	Bituminous Coal (Volatility)
Isotropic	0.0	None	6, 7	High
Incipient (anisotropic)	0.5	L=W*	8	High
Circular (anisotropic)				
Fine circular	0.5 – 1.0	L=W	9	High
Medium circular	1.0 – 1.5	L=W	10	High
Coarse circular	1.5 – 2.0	L<2W	11	High to Med.
Lenticular (anisotropic)				
Fine lenticular	1.0 – 3.0	L≥2W L<4W	12	High to Med.
Medium lenticular	2.0 – 8.0	L>2W L<4W	13	Medium
Coarse lenticular	8.0 – 12.0	L>2W L≤4W	14	Medium to Low
Ribbon (anisotropic)				
Fine ribbon	3.0 – 12.0	L>4W	15	Medium to Low
Medium ribbon	12.0 – 25.0	L>4W	16	Low
Coarse ribbon	25.0+	L>4W	17, 18	Low

Coke Filler-Phase Carbon-Form Classification

Filler-Phase	Size (in microns)	Precursor material
Organic Inerts		
Fine	<50	Micrinite, macrinite, inertadetrinite
Coarse**	>50	Semifusinite, Fusinite, macrinite
Miscellaneous Inerts		
Oxidized coal (coke)		Oxidized coal – slight moderate and advanced.
Brecciated coal (coke)		Brecciated coal – fine, intermediate and coarse.
Noncoking Vitrinite (coke)		Vitrinite too high or low in rank.
Inorganic Inerts		Various types of mineral matter
Fine	<50	Coal mineral matter and bone coal
Coarse	>50	Coal mineral matter and bone coal

Miscellaneous Categories Quantified in Carbon Form Analysis***

Depositional Carbons	Sooty, spherulytic, and pyrolytic.
Additive Carbons	Coke breeze, anthracite, petroleum coke, etc.
Miscellaneous Observations	"Green" coke, burnt coke, etc.

Notes: * L = length of carbon form domain (long dimension).
W = width of carbon form domain (short dimension).

** Includes coarse inert-rich microlithotypes which retain distinct particle boundaries and appear to act as filler instead of binder phase. These are kept separate during microscopic analysis.

*** Miscellaneous observations can be quantified or presence can be indicated as rare or abundant.

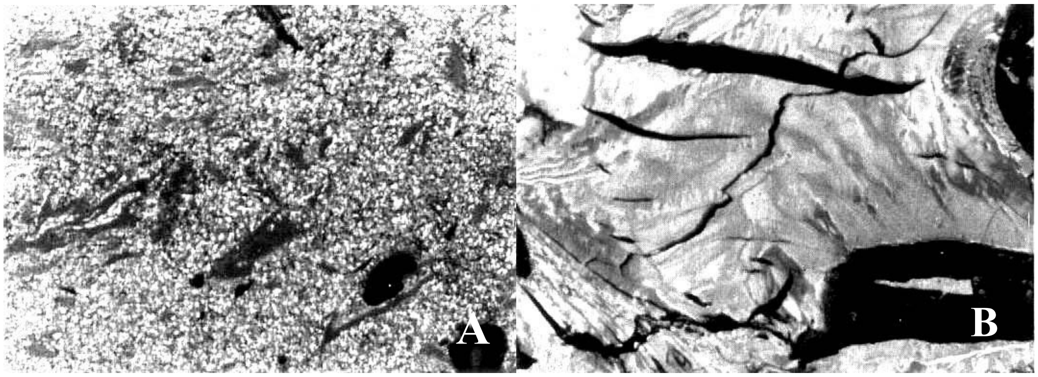


Figure 5. Two examples of coal coke microstructure: A, circular anisotropic (coarse); B, ribbon anisotropic (coarse).

4 EXPERIMENTAL

4.1 Feed Coal Characterization and Solvent Processing

4.1.1 Coal Selection, Preparation, and Characterization

Twelve different coals or fractions (9 for coal rank, 3 for coal type) were acquired for this project, ranging from about 0.5 to about 1.6% vitrinite reflectance. Figure 6 is an outline of the steps followed in the preparation of the coal for characterization and solvent extraction. Approximately 80 pounds or more of as-received coal were air dried and then crushed to ¼ x 0 inch. The coal was riffled and 15 pounds of this material crushed to 8 x 0 mesh. The remainder was sealed and retained for other testing, such as washability. Five pounds of the riffled material was ground to minus 20 mesh. A portion of the minus 20-mesh coal underwent reflectance measurements, and maceral type and content determination. The remaining minus 20-mesh coal was ground and sized as shown in order to undergo other tests. Table 4 identifies the coals and provides some of their basic characteristics. Additional data are located in Appendix 1.

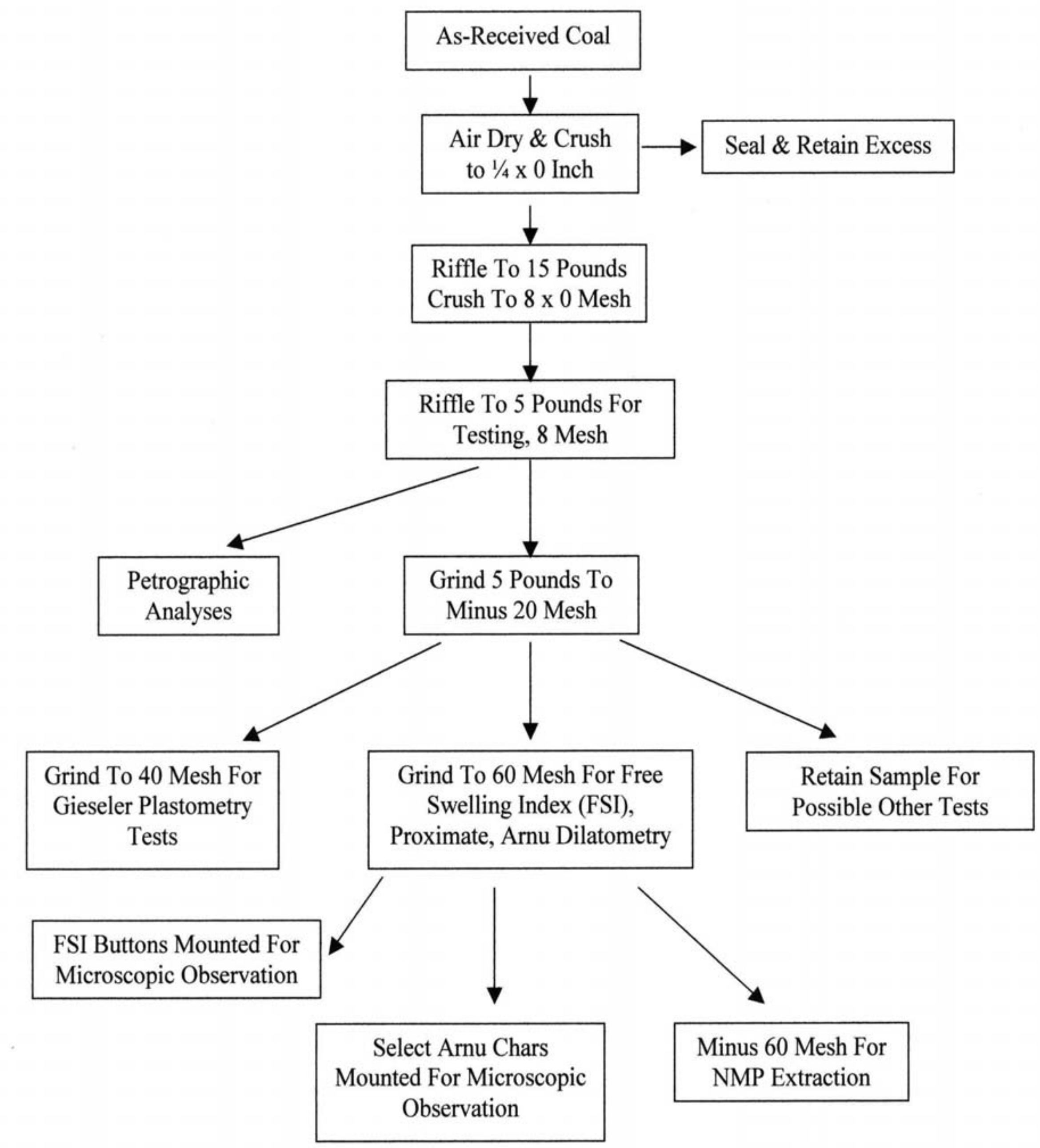


Figure 6. Coal preparation and testing outline steps.

Table 4. Characteristics of bituminous coals selected for testing in this project.

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell /WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Mean Max Vitrinite Reflectance R_o, %	0.53	0.77	0.83	0.98	1.02	1.08	1.23	1.36	1.53
Sulfur, %dry	3.26	3.12	1.91	0.76	1.06	1.02	0.74	0.92	1.11
Proximate Analysis, %dry									
VM	36.65	38.06	36.22	35.00	33.45	33.17	27.42	22.77	19.54
FC	53.52	51.92	56.11	59.27	60.05	57.91	68.09	70.63	76.63
Ash	9.83	10.02	7.67	5.73	6.50	8.92	4.49	6.60	3.83

4.1.2 Coal Extraction in NMP (N-methyl pyrrolidone)

The coal samples (<60mesh, dry) were tested to determine extraction efficiency. Ten grams of coal were added to 100mL of NMP in a 250mL ground-glass-joint Erlenmeyer flask. A water-cooled condenser was attached to the flask and provision was made for purging the system with nitrogen gas. Mixing was accomplished with a magnetic stirrer and heating supplied by a temperature-variable hotplate. The solution was brought to reflux at about 202°C for one hour after which the solution was vacuum filtered hot through a 1.6μm glass-fiber filter using a conventional Buchner-type filtration apparatus. The filter cake was washed with about 100mL of hot NMP and then was dried overnight in a vacuum oven at about 150°C while maintaining a slow purge of nitrogen gas. The filtrate was isolated from the NMP by rotary evaporation before vacuum drying along with the insoluble residue. After cooling to room temperature, the coal residue was weighed to determine extraction yield according to the following equation:

$$\text{Yield} = [(\text{wt dry coal} - \text{wt residue})/(\text{wt dry coal})] \times 100.$$

Each coal extraction was repeated four times to establish statistical reliability. In addition, each coal underwent solvent extraction sufficient to produce about 40-50g each of soluble extract and insoluble residue for additional characterization.

4.1.3 Determination of Plastic Properties and Proximate Analysis

Standard tests were used to characterize feed coal, extracts, and residues for thermoplastic behavior according to dilatation, fluidity, and free swelling. The changes in coal length during heating under controlled conditions can be accomplished using an Audibert-Arnu dilatometer. A sample of powdered coal material is pressed into a pencil and inserted into a steel tube. A piston is inserted into the tube on top of the pencil. The assembly is then heated at a proscribed rate. As the sample changes dimensions to the response of heat, the piston is displaced and the distance traveled is recorded. The movement of the piston is expressed as a percentage relative to the original length of sample. The determinations are reported as percent maximum contraction, percent maximum expansion, initial softening temperature, initial dilatation temperature, and maximum dilatation temperature.

Plasticity was determined in a Gieseler plastometer. A powdered coal sample is compressed in a cylindrical container fitted with a rabble-arm stirrer. The cylinder is placed in a solder bath and heated at a controlled rate. A constant torque is applied to the stirrer, which moves in response to changes in coal fluidity. The units of plasticity are reported as dial divisions per minute (ddpm). The information collected includes maximum fluidity, temperature at maximum fluidity, softening temperature, solidification temperature, and plastic-range temperature.

The free-swelling index (FSI) is a measure of the increase in volume of coal material when heated under specified conditions. Since the determination is conducted such that the swelling and dimensional changes occur unimpeded, it is possible for the coal sample to flow and form a button. The free-swelling index is then determined by comparing the button with a series of standard profiles, which are numbered 1 to 9 in 0.5 units. A large FSI value indicates considerable expansion of the coal upon heating.

Proximate analysis is determined by heating coal material under controlled conditions and recording associated weight changes. The method provides percentage moisture, volatile matter, and ash. Fixed carbon is determine by subtracting the percentages of moisture, volatile matter, and ash from 100

4.2 Analyses of Feed Coals, Residues, and Extracts

4.2.1 Petrographic Method Applied to Feed Coals, Residues, Extracts, and Cokes

Conventional sample preparation and polishing techniques, and established quantitative microscopical methods were followed in the petrographic examination of coals, extracts, and residues. Chars and cokes produced from the extracts in the free-swelling index determination were also mounted in plastic and prepared for optical microscopy. In addition, coal samples were mounted to determine whether a solvent etching procedure with NMP could be developed and correlated with extraction yield and some microscopic feature. The solvent etching consisted of placing a drop of NMP onto the polished surface of coal in an identifiable area. The pellet with NMP was then placed in a microwave oven for various periods of time. It was determined that 30 seconds in the microwave was sufficient to etch the various ranks of coal being tested. The etched surface was then rinsed in water and/or alcohol and allowed to air dry before optical microscopy.

4.2.3 Physical and Chemical Characterization of Feed Coals, Residues, and Extracts

Several analyses were conducted to determine surface and bulk properties, and chemical nature of the feed coals, extracts, and residues. Surface and bulk characteristics were determined by helium pycnometry (ASTM D-4892), surface area by the BET method using liquid nitrogen, and porosity by mercury intrusion (ASTM-4284). The chemical characterization tests consisted of elemental analysis, thermogravimetric analysis, and infrared spectroscopy. The determination of carbon, hydrogen, nitrogen, and sulfur was conducted on a ThermoQuest elemental analyzer. Following calibration of the instrument with compounds of known composition, coal samples were analyzed and the average of at least three determinations reported. Infrared spectra were collected on a Nicolet 510P Fourier-transform infrared (FTIR) spectrometer by co-adding 256 interferograms at a resolution of 2 cm^{-1}. Samples were prepared using a conventional KBr pellet process with a specimen concentration of about 3wt%. Sample weight loss with temperature was determined on a Leco TGA analyzer under nitrogen at a heating rate of 3°C/min up to 900°C.

5 RESULTS AND DISCUSSION

All of the experimental data are tabulated in Appendix 1 through 16. Several hundred photomicrographs were produced and, thus, it was not possible to include them all in this report. The more relevant findings are presented in the following sections.

5.1 Results of NMP Extraction

Early in the project there was discussion as to whether channel samples from newly mined areas, free from weathering, should be selected. However, it was acknowledged that this might not be always possible or cost effective. If channel samples or raw-run-of-mine samples were to be used, they would require laboratory washing using solvents of select specific densities. It was not known if specific gravity chemicals would affect the solvent extraction process or extract quality. To address the concern, coal sample A was washed in Centigrav (1.45 specific gravity) and the float/sink fractions combined. In addition, the 1.35 specific gravity float fraction of coal B was extracted in NMP. The results presented in Table 5 indicate that laboratory washing of coal B had little, if any, affect on extraction efficiency. However, the float fraction of coal A appeared to extract slightly more than the feed coal perhaps because of the reduction in the mass of mineral matter or the more dense maceral components, or a concentration of fractions more amenable to extraction. In any case, the results for coal A sample are nearly within experimental error.

Table 5. NMP-extraction of laboratory washed coals.

	Coal A		Coal B	
	Feed Coal	1.35sg Float	Washed Centigrav F/S Combined	Not Washed
NMP Extraction Yield, wt%	66.7 $\sigma = 0.35$	71.7 $\sigma = 2.12$	37.5 $\sigma = 1.74$	36.8 $\sigma = 0.69$

σ = Standard deviation of four extractions.

A suite of about 10 different coals or fractions (7 for coal rank, 3 for coal type) was initially planned for sampling, detailed microscopic analysis, rheological properties, and limited chemical analyses to be undertaken prior to NMP extraction. Testing some of the initial candidate coals did not accomplish the objective in determining the lower and upper rank boundaries amenable to solvent extraction. Thus, two additional coals were selected and ordered to clarify the upper, lower, and optimum rank and fluidity ranges where extraction yield diminishes or reaches optimum. Therefore, coal “type” studies were very limited under the current constraints of this year’s budget allocation. Consequently, coal sampling entailed all production samples. For each sample, location, seam, mine name, depth, mine section, and other important information were obtained whenever possible.

Thus, it was decided that solvent extraction and detailed characterization would be focused on the nine coals based solely on rank rather than type. The results of NMP extraction of these coals are provided in Table 6 and shown graphically in Figure 7.

Table 6. Yield of NMP-soluble extracts from bituminous coals.

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Mean Max Vitrinite Reflectance R_o, %	0.53	0.77	0.83	0.98	1.02	1.08	1.23	1.36	1.53
Yield, wt%	30.3	34.1	30.9	32.4	45.3	66.7	40.6	30	5
Standard Deviation	0.73	2.1	0.69	0.55	1.97	0.35	0.84	1.96	0.41
Yield, daf%	33.6	37.9	33.5	34.4	48.4	73.2	42.5	32.1	5.2

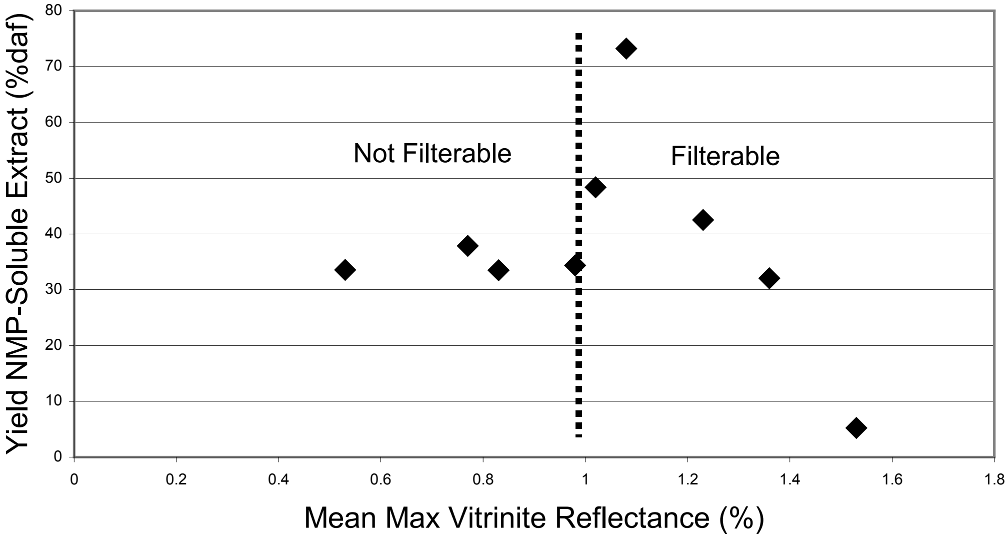


Figure 7. Effect of coal rank on NMP extraction efficiency.

There is a trend in increasing extraction yield up to a vitrinite reflectance of about 1.0 with decreasing yields evident at the higher ranks. Important in terms of processing was the observation that coal solutions to the left of the vertical dotted line in Figure 7 were extremely difficult to filter. For these coals the solutions immediately blind the filter, which had to be replaced frequently to allow reasonable filtration rates. A drop of filtrate from the Shoemaker coal (reflectance 0.77) was placed on a glass slide and examined under a microscope where reticulated coal fragments suspended in the solution were observed. Presumably the extracts from the low rank bituminous coals are not true solutions but are colloidal-like mixtures, which would be difficult to remove by simple filtration. Coals with reflectances above 1.0 filtered without any difficulty. The highest extraction was obtained with the Kingwood coal at 73.2% on a daf basis. This coal filtered quite easily.

5.2 Correlation of Coal Characteristics with Solvent Extraction Performance

There was no clear relationship between extraction behavior and rank with true density, surface area, or porosity of the feed coals and insoluble residues, see the data compiled in Appendix 2. This lack of correspondence is probably because of the small population of coals and the fact that these physical attributes do not vary much within the range of rank investigated [44].

On the other hand, firm correlations were noted with extraction and thermoplastic behavior with a range of coal rank parameters. Some examples are provided in Figures 8 through 12. Although arguments with Audibert-Arnu dilatometry and FSI would lead to similar conclusions drawn with the Gieseler plastometry results, correspondence with the former two methods were more scattered.

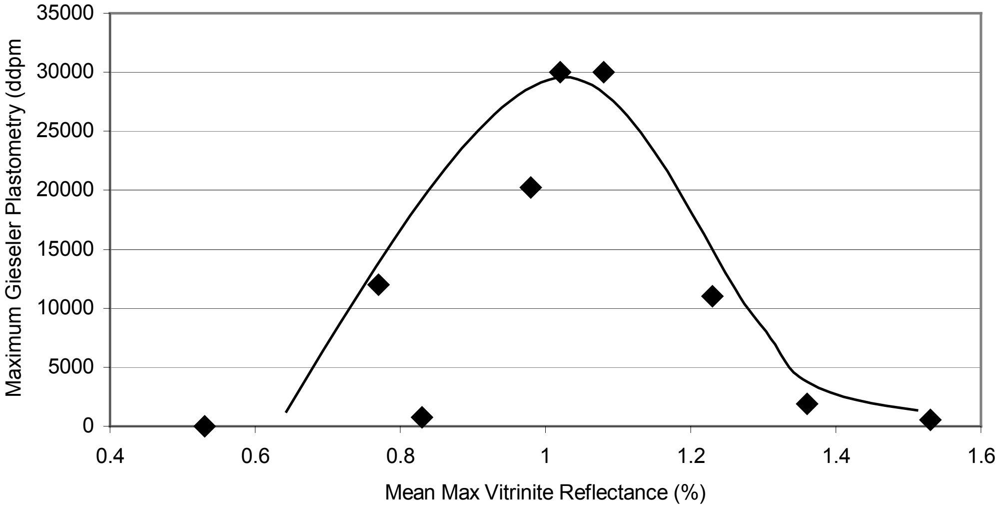


Figure 8. Reflectance (rank) vs. maximum Gieseler plasticity.

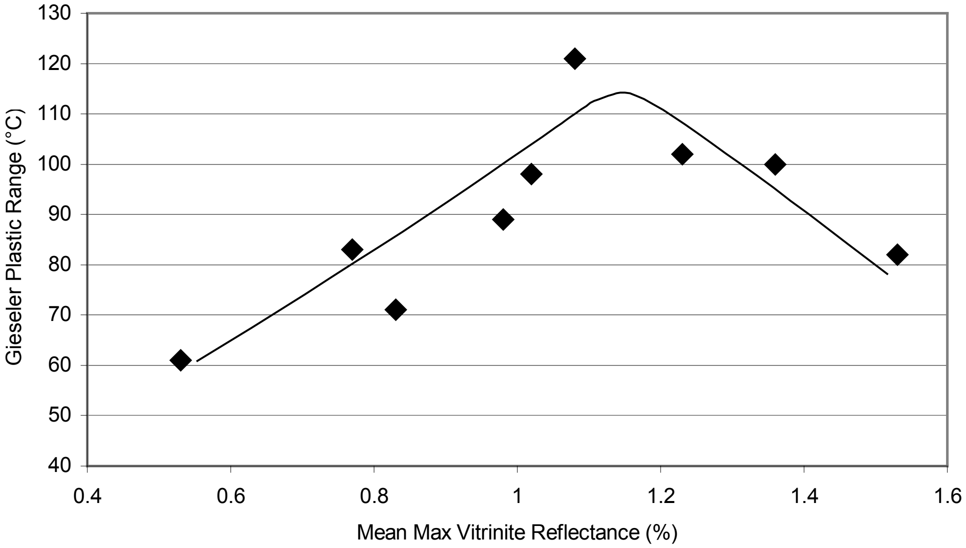


Figure 9. Reflectance (rank) vs. Gieseler plastic range.

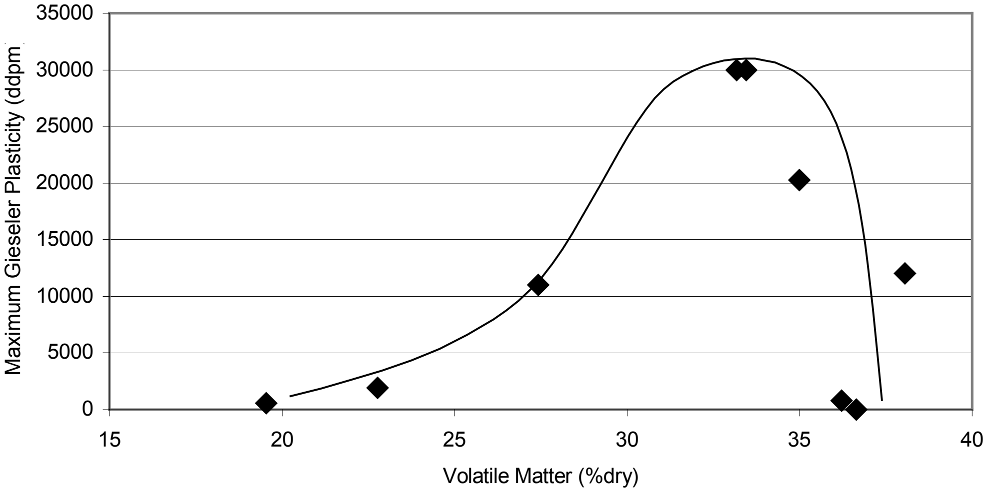


Figure 10. Volatile matter vs. maximum Gieseler plasticity.

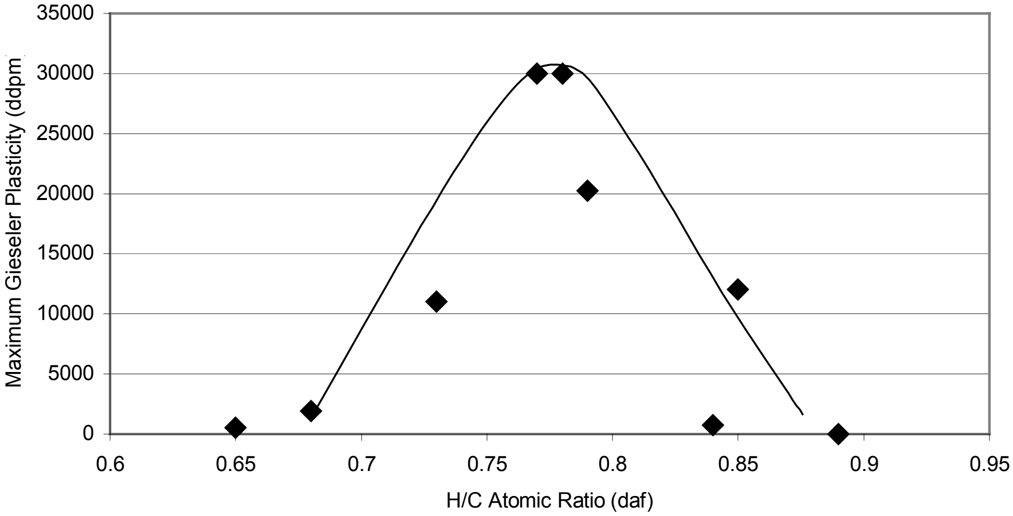


Figure 11. H/C atomic ratio of feed coals vs. maximum Gieseler plasticity.

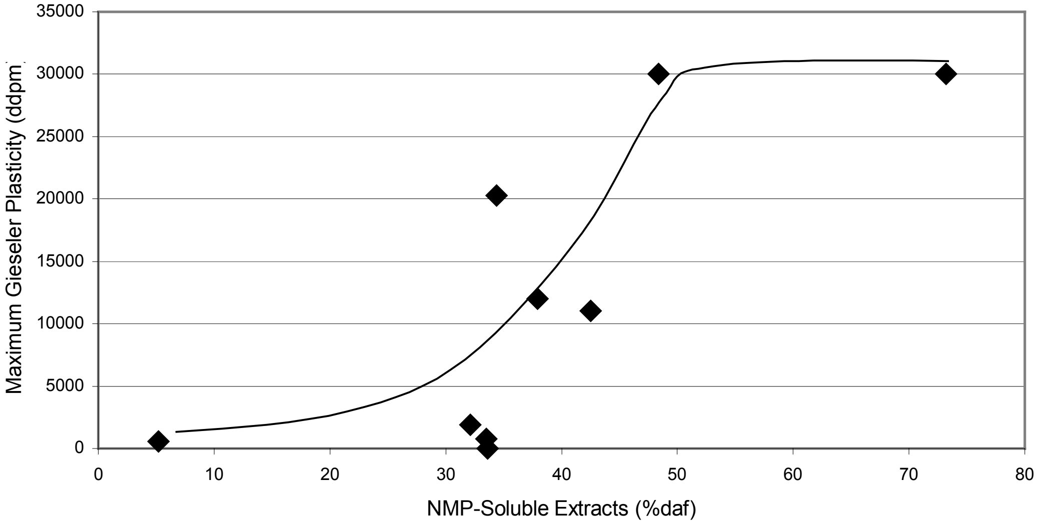


Figure 12. NMP extract yield vs. maximum Gieseler plasticity.

All feed coals and their residues and extracts were examined by FTIR for functional group comparisons. In particular, the peaks associated with the aromatic C-H (H_{ar} at about 3050 cm^{-1}) and aliphatic C-H (H_{al} at about 2900cm^{-1}) vibrational stretching modes were integrated after baseline correction. The ratio H_{ar}/H_{al} can be used as an indicator of aromaticity [45] and, thus, can be considered a rank parameter. Example spectra are provided in Figure 13, which compares the infrared spectra for the Kingwood feed coal, extract, and residue. Figure 14 compares the H_{ar}/H_{al} ratio with C/H atomic ratio calculated from knowing the elemental composition of the feed coals. Not unexpected, the two ratios appear to be correlated in that the feed coals become more aromatic as rank increases. Interestingly, the H_{ar}/H_{al} ratio of the feed coal and extracts increase as their respective reflectances increase, as seen in Figure 15. Using FTIR to determine H_{ar}/H_{al} ratio was not possible for all of the residues, however, because for some of these products the peak intensities were too weak to integrate. FTIR and elemental analysis are summarized in Appendix 5 and 16, respectively.

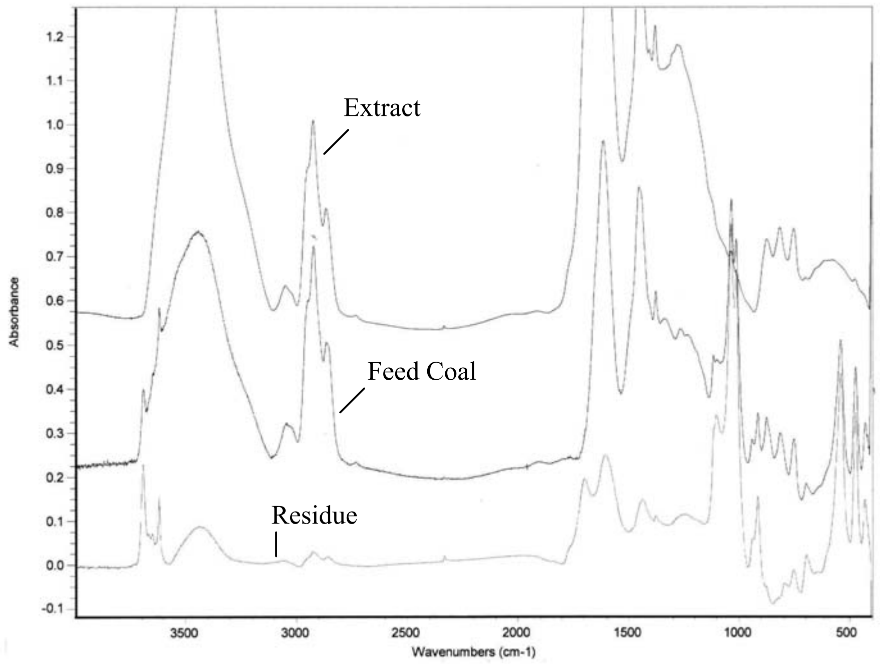


Figure 13. FTIR spectra of Kingwood coal, extract, and residue.

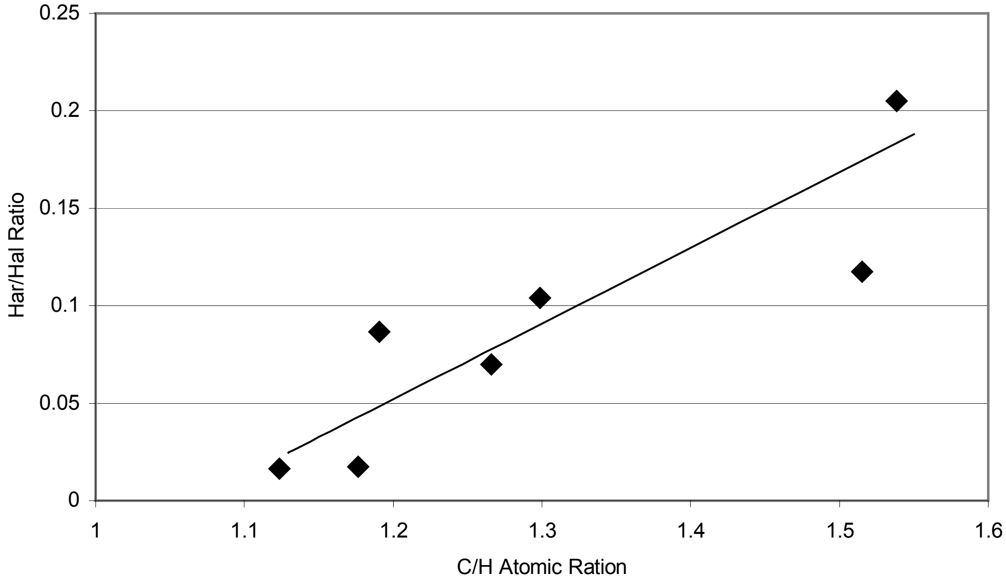


Figure 14. Atomic C/H ratio vs. H_{ar}/H_{al} ratio of feed coals.

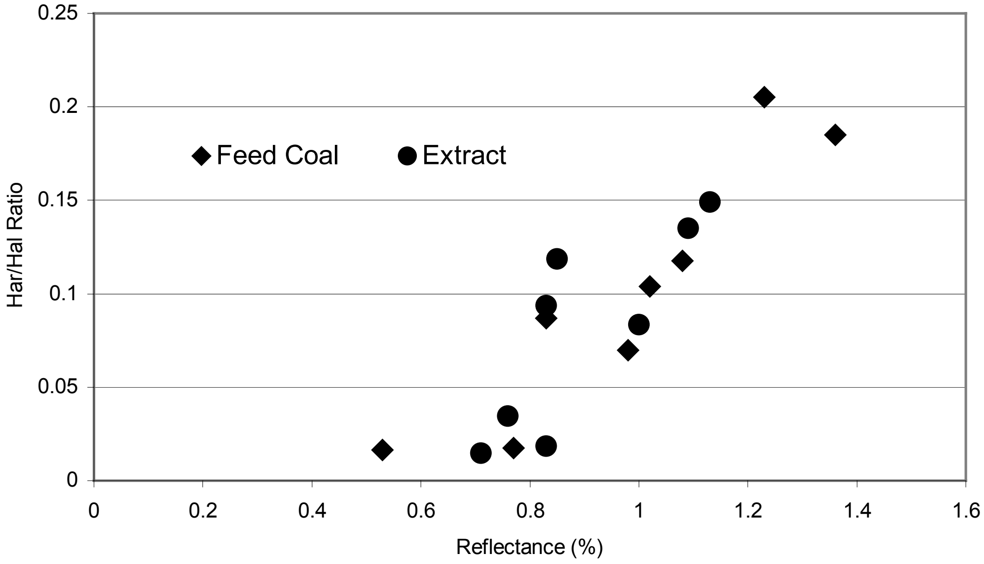


Figure 15. Reflectance vs. H_{ar}/H_{al} ratio of feed coals and extracts.

Figure 16 compares the thermogravimetric analysis of the feed coals, extracts, and residues. Weight change with temperature for the feed coals follows what would be expected with rank in that volatile material decreases as rank increased. The extracts lose more weight than either the feed coals or residues and did not follow weight changes associated with rank as closely as the feed coals. It is interesting that the insoluble residues still contain significant amounts of volatile material even after solvent extraction.

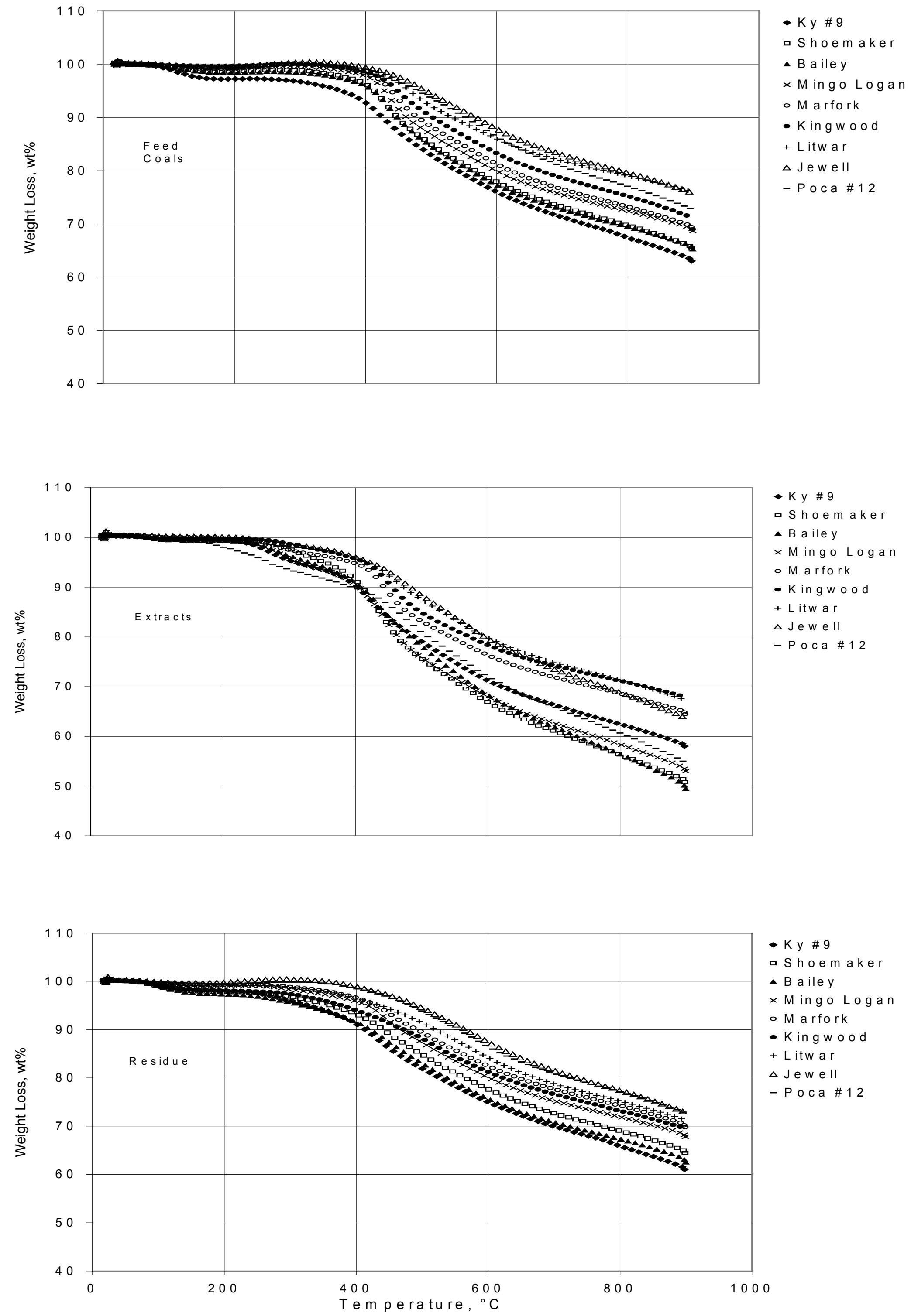


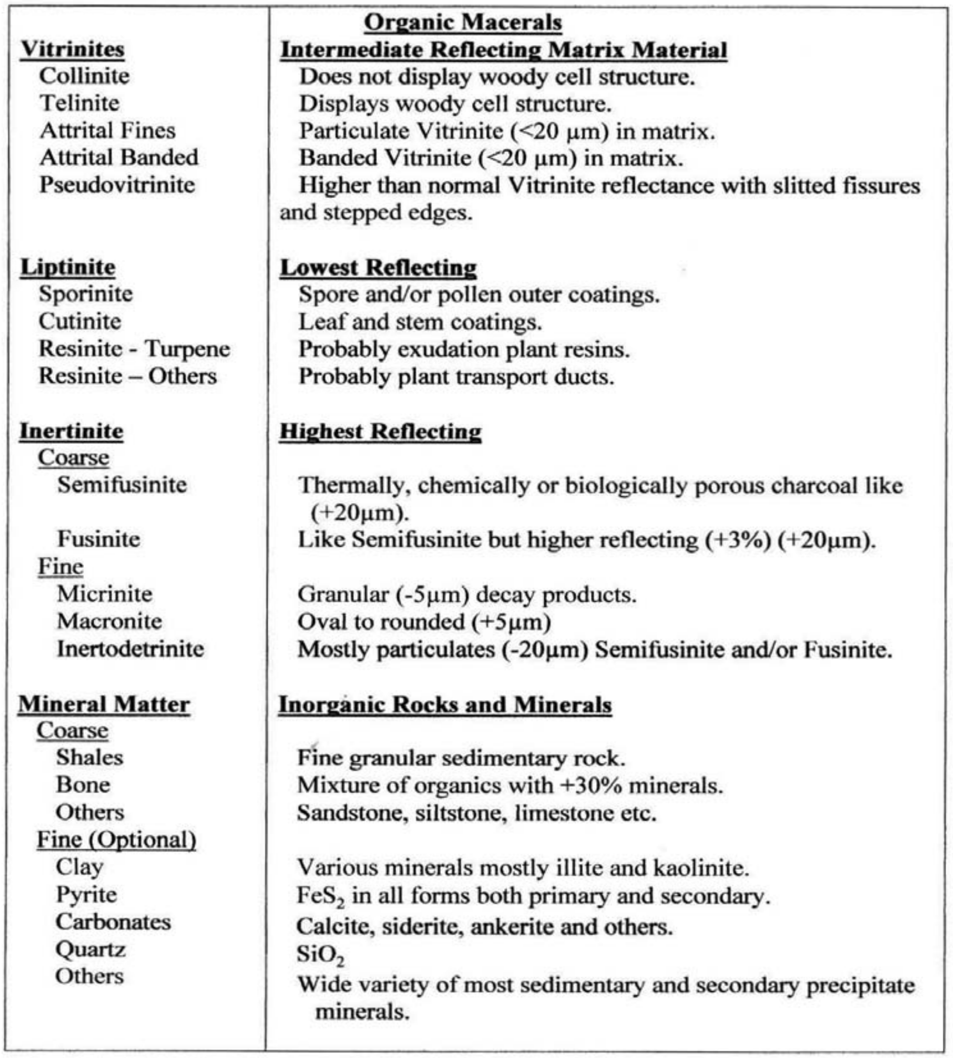
Figure 16. TGA analysis of feed coal, extract, and residue.

5.3 Optical Microscopy of Coal and Solvent Extracted Products: Observations and Comments

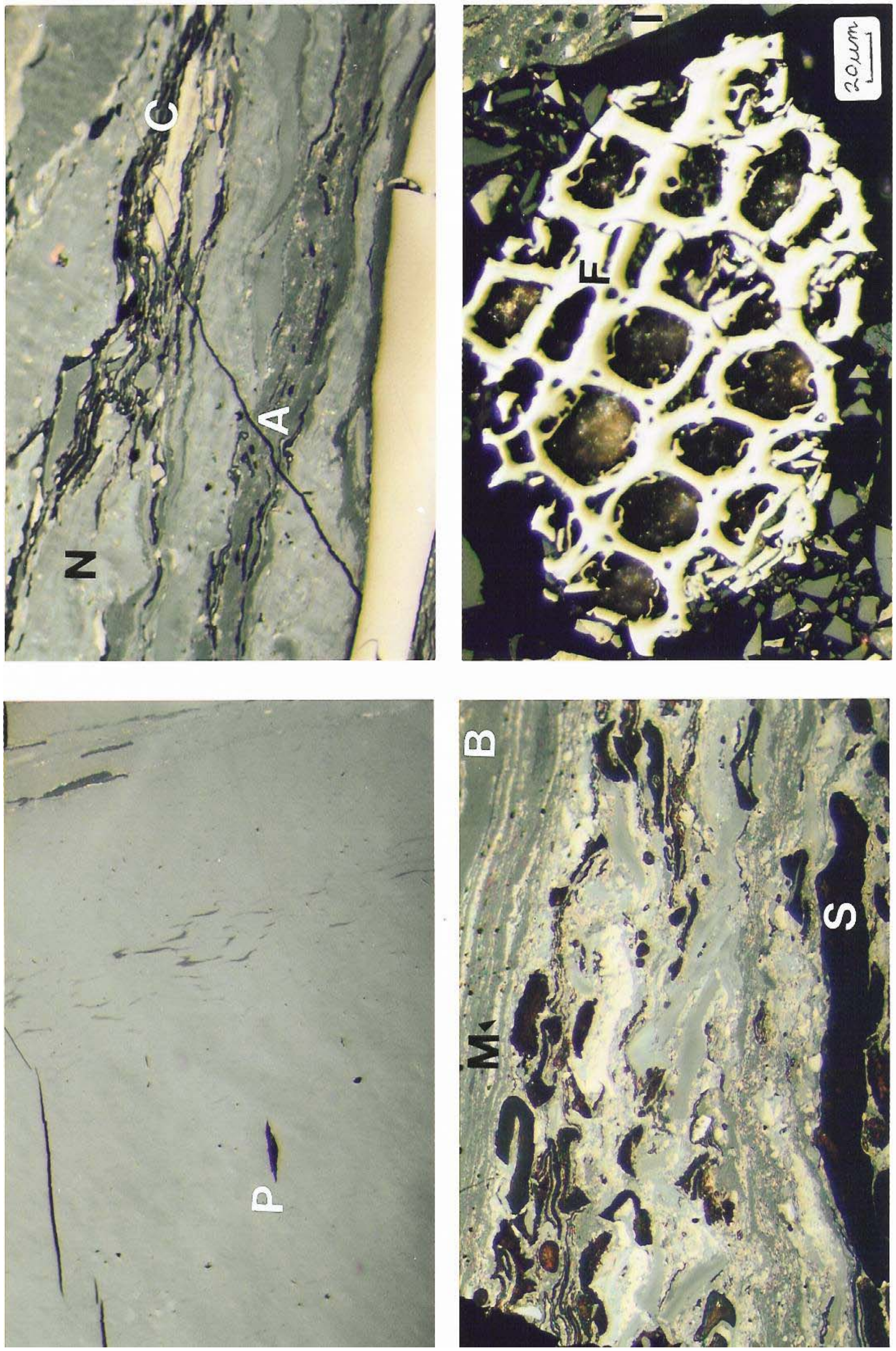
For this research project, the petrographic maceral and mineral classification terminology used in the descriptive and quantitative analysis of coal and insoluble residue is shown in Table 7. The vitrinite group as used in this report consists of several subgroup macerals. Telinite displays wood structure and collinite appears structureless together with attrital vitrinite. Pseudovitrinite is slightly higher in reflectance than collinite and exhibits distinct fracture patterns. Attrite vitrinite was divided into particulate and layered categories.

Collinite, appearing structureless, is homogeneous and is a major component of most coals. The collinite is commonly used in ranking coal by determining its reflectance. The reflectance values are reported as V-types, which represent reflectance categories. The distribution of V-types determined for the feed coals, extracts, and residues are provided in Appendices 8, 9, and 10, respectively. Telovitrinite consists of vitrinite that displays cellular plant structures. Pseudovitrinite is a variety of telinite that commonly is higher reflecting than other vitrinites and displays remnant cell structure, fusiform slits, and stepped particle edges. Attrital vitrinite frequently occurs as fine size bands (attrital banded) and/or particles (attrital fine) in attrital coal. Figure 17 shows some of the macerals identified in one of the bituminous coals used in this project.

Table 7. Petrographic maceral and mineral classification.



	Organic Macerals
Vitrinites	**Intermediate Reflecting Matrix Material**
Collinite	Does not display woody cell structure.
Telinite	Displays woody cell structure.
Attrital Fines	Particulate Vitrinite (<20 μm) in matrix.
Attrital Banded	Banded Vitrinite (<20 μm) in matrix.
Pseudovitrinite	Higher than normal Vitrinite reflectance with slitted fissures and stepped edges.
Liptinite	**Lowest Reflecting**
Sporinite	Spore and/or pollen outer coatings.
Cutinite	Leaf and stem coatings.
Resinite - Turpene	Probably exudation plant resins.
Resinite – Others	Probably plant transport ducts.
Inertinite	**Highest Reflecting**
Coarse	
Semifusinite	Thermally, chemically or biologically porous charcoal like (+20μm).
Fusinite	Like Semifusinite but higher reflecting (+3%) (+20μm).
Fine	
Micrinite	Granular (-5μm) decay products.
Macronite	Oval to rounded (+5μm)
Inertodetrinite	Mostly particulates (-20μm) Semifusinite and/or Fusinite.
Mineral Matter	**Inorganic Rocks and Minerals**
Coarse	
Shales	Fine granular sedimentary rock.
Bone	Mixture of organics with +30% minerals.
Others	Sandstone, siltstone, limestone etc.
Fine (Optional)	
Clay	Various minerals mostly illite and kaolinite.
Pyrite	FeS_2 in all forms both primary and secondary.
Carbonates	Calcite, siderite, ankerite and others.
Quartz	SiO_2
Others	Wide variety of most sedimentary and secondary precipitate minerals.



Photomicrographs of Macerals in Arch Minerals Mingo Logan Coal, West Virginia Showing;
P=Pseudovitrininte, A=Fine Attrital Vitrinite, B=Banded Attrital Vitrinite, S=Sporinite, C=Cutinite,
M=Micrinite, N=Macrinite, I=Inertodetrinite and F=Fusinite. Reflected Light In Oil, X600.

Figure 17. Features of maceral components in bituminous coal.

Liptinite in bituminous coals has lower reflectance than the other group macerals. It consists of sporinite, cutinite, and resinite (lights and darks). Sporinite is commonly derived from the protective coatings of the reproductive organs of plants. Cutinite is derived from the protective coatings on leaves and stems. Liptinite is high in hydrogen and volatile matter. Resinous materials may be excretion products and/or duct and cell filling.

The inertinite maceral group consists of high carbon and relatively high oxygen materials with low volatility. It generally has the highest reflectance of all the other macerals. Inertinite has limited plasticity when heated. It is hard and tends to exhibit relief in polished samples. The inertinite macerals consist of fusinite, semifusinite, inertodetrinite, micrinite, macrinite, secretinite and funginite. Fusinite selectively exhibits cell wall preservation like charcoal. Semifusinite also exhibits distinct charcoal-like wall structures but is lower in reflectance than fusinite but higher in reflectance than vitrinite. Inertodetrinite consists of less than 50μm fragments of the other inertinite macerals, such as semifusinite and fusinite fragments. Micrinite occurs as relatively amorphous <10μm specks in vitrinite. Macrinite may be round or irregular in shape and exceeds 10μm in size but seldom exceeds 50μm. Funginite is frequently rounded to oval and less than 30μm in size. It commonly contains a network of walls and windows that are evidence of its fungal origin. Secretinite is commonly rounded to oval but can also be rod-like in shape. Secretinite may be the products of oxidation of resinite ducts in plants.

5.3.1 Petrographic Analysis of Feed Coals and Insoluble Residues

The basic coal petrographic data can be broken down into reactives and inerts as shown in Table 8. The reactives are more sensitive to oxidation and thermal changes.

The reactives consist mostly of vitrinite, which determines most of the coal's characteristics. The liptinite portion of the reactives does not respond well to NMP extraction while the vitrinites are the most amenable to extraction. The inerts remain unchanged during NMP extraction and concentrate more in the insoluble residue.

Table 8. Summary of petrographic analysis of coal and insoluble residue.

	Coal		Residue		Extraction Yield, wt%	
Coals	Reactives	Inerts	Reactives	Inerts	Extract	Residue
Ky #9	78.2 (7.5)*	21.8 (5.6)**	76.6 (9.0)*	23.4 (7.6)**	30.3	67.7
Shoemaker	79.2 (7.0)	20.8 (4.2)	68.8 (7.2)	31.2 (7.4)	34.1	65.9
Bailey	80.2 (6.5)	19.8 (3.4)	74.5 (7.6)	25.5 (6.1)	30.9	69.1
Mingo-Lo.	67.1(13.5)	32.9 (2.9)	60.7 (72.9)	39.3 (4.3)	32.4	67.6
Marfork	71.7 (6.5)	28.3 (4.4)	52.0 (13.9)	48.0 (6.9)	45.3	54.7
Kingwood	79.6 (5.0)	19.4 (4.3)	63.1 (11.4)	36.9 (16.4)	66.7	33.3
Litwar	76.9 (6.3)	23.1 (2.6)	65.7 (4.3)	34.8 (5.1)	40.6	59.4
Jewell	79.4 (2.9)	20.6 (4.4)	74.3 (2.9)	25.1 (5.6)	30.0	70.0
Poca #12	80.6 (1.2)	19.4 (3.1)	80.0 (1.1)	20.0 (2.3)	5.0	95.0

* (0.0) Liptinite
**(0.0) Mineral Matter

The mineral portion of the inerts is determined by volume (area) in the petrographic analysis. Thus, it has a disproportionate impact on the residue weight since the mineral weight is about twice that of macerals per unit volume. The summary petrographic data in Table 8 shows the inerts, liptinite, and mineral matter are almost all higher in the insoluble residue than in the parent coal. The most NMP extractable coals show the greatest increase in inerts, liptinite, and mineral matter in the residues. From other data it is also evident that particulate attrital vitrinite is the least amenable vitrinite in NMP extraction. Thus vitrinite, with the partial exception of particulate attrital vitrinite, is the NMP extractable portion of bituminous coals.

The composition of the feed coals and insoluble residues were calculated petrographically and converted from a volume to a weight basis. The results of the calculations are tabulated in Appendix 14 and 15, respectively. If in the solvent extraction only vitrinite is dissolved, then it is possible to estimate the amount of vitrinite remaining in the insoluble residue from mass balances. Comparison of the petrographic and mass balance estimations in Figure 18, shows a reasonable relationship. However, vitrinite content by mass balance underestimates that by petrography. This could because non-vitrinite material passed through the filter during product work-up and/or the assumptions associated with petrographic analysis.

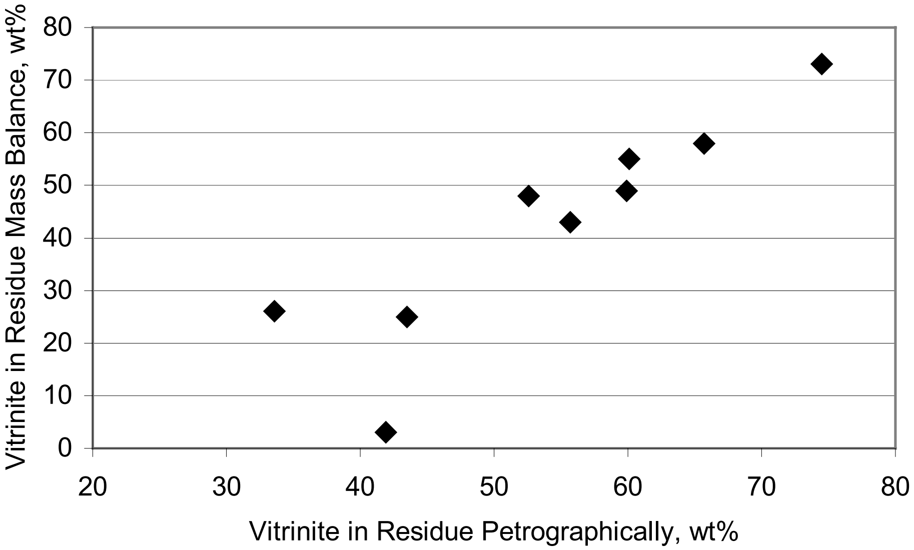


Figure 18. Estimated vitrinite content in insoluble residues.

Table 9. Classification of non-maceral microstructures.

Microstructure Categories	**Subdivisions of Microstructure Categories**
Normal Coal	Vitrinite coarse, +50µm Semifusinite coarse +50µ Fusinite coarse +50µm. Bright attrital >75% Vitrinite. Intermediate attrital <75% and <50% Vitrinite. Dull attrital <50% Vitrinite.
Fine Coal <5 microns	Angular Rounded Indistinct
Pseudovitrinite	All relatively high reflectance Slitted Remnant cellular texture Homogeneous Combinations of above
Microbrecciated	Fine Intermediate Coarse Faults or folds
Oxidized (Weathered)	Slight Moderate Bad
Oxidized (Thermal)	Rounded Bright rims Porous Semicoked Coked
Coarse Mineral Matter	Clay Shale Pyrite Carbonate Quartz Bone Others
Cenospheres	Semicoke Coked
Contamination	High-volatile coal Medium volatile Low volatile Others designated

5.3.2 Coal Types and Non-Maceral Microstructures

Some of the criteria used in the microscopic identification and counting of coal non-macerals and minerals are listed in Table 9. A normal coal is divided into bright, intermediate, and dull based on vitrinite content. This serves to separate coal into the rock types and is an indicator of the degree of homogeneity. Coarse vitrinite, pseudovitrinite, and fusinite are also counted.

There are many important coal characteristics that are not determined in a normal maceral analysis. Such features as oxidation are determined as well as brecciation faulting and folding and other occurrences such as cenospheres. Each of the determined microstructures has a specific significance. Non-maceral microstructure analysis is also useful in determining the extent to which a coal has or can be beneficiated based on mineral form and association. It is also very useful in determining contamination where coals of different ranks or other materials have been mixed either inadvertently or intentionally with the subject coal.

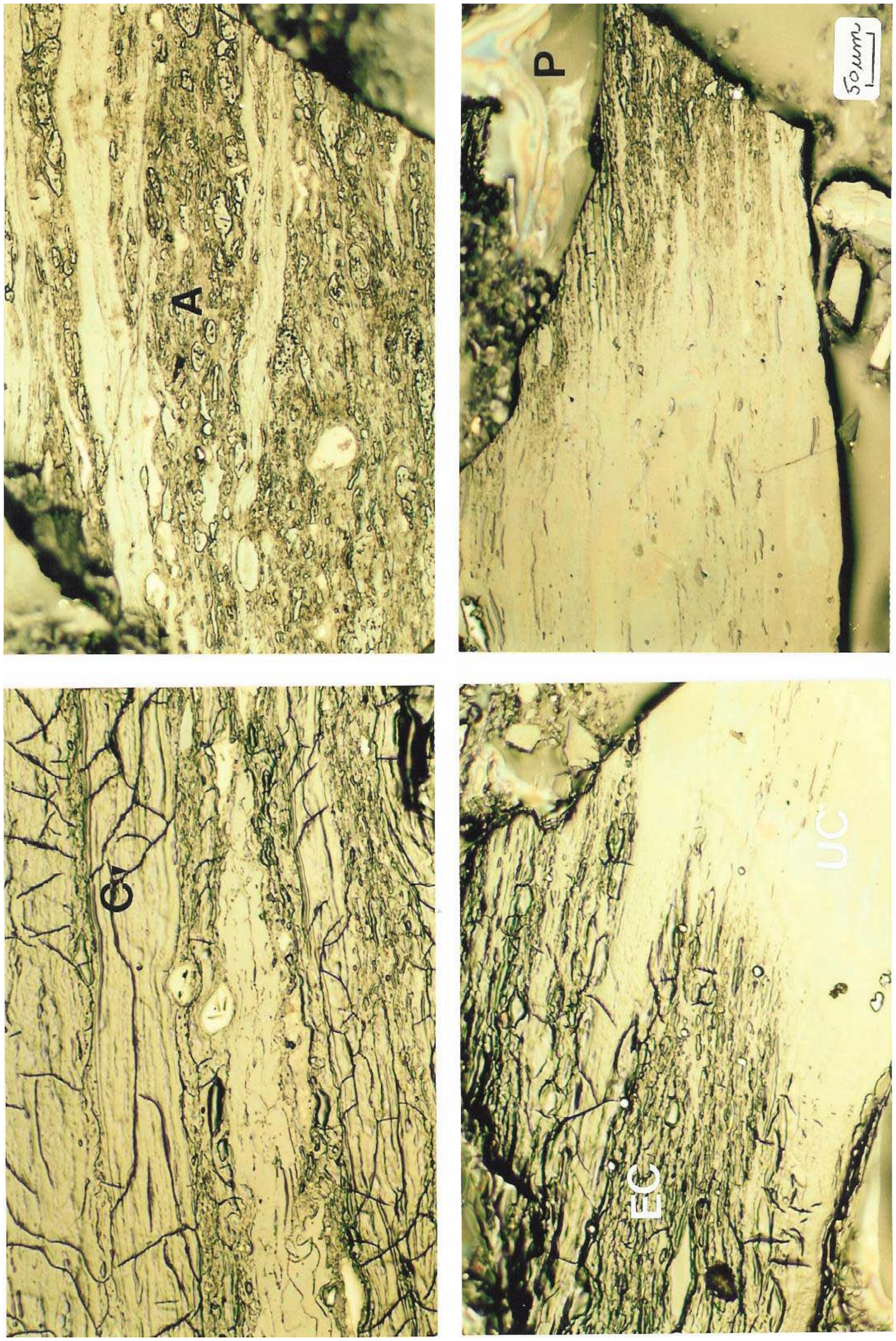
The non-maceral petrographic data show that there are only minor oxidation, contamination, or brecciation in the coals. The coarse mineral matter varied but is not unusually high in any of the coals. The sum of bright and intermediate attrital plus coarse vitrinite and pseudovitrinite, shown in Table 9, range from 61.9 o 83.3% by volume. None of the non-maceral data showed any correlation with NMP extractability.

5.3.3 Coal Etching in NMP

The nine coals were etched to determine if extract yield correlated with some microscopic feature of the etched coal. Dried polished coal pellets (ground and polished coal mounted in plastic) were used for etching. Following exposure to NMP and treatment in the microwave oven for 30sec, the water rinse appeared to remove all of the NMP leaving coal particles that contained areas with various degrees of extraction (relief and fracturing). When an alcohol rinse was used, the relief and fracturing that differed for various coal macerals and coal types were retained but staining was added. Liptinite and inertinite did not show any evidence of change because of NMP etching. The vitrinite, particularly the coarser layers or bands, showed significant alteration. The Ky # 9 showed the greatest relief with the telinite cell walls showing less alteration than the cell fillings. Staining differences were also observed. The Shoemaker coal developed some distinct crack patterns in addition to relief changes. The staining consisted of a variety of colors, particularly blue with some green. A larger portion of what appeared to be collinite in the unetched coal developed a very distinct cellular pattern after etching: this was very prominent in the Shoemaker, Bailey, and Mingo Logan samples. The staining cellular structures became very distinct in the Marfork coal. The vitrinite staining appeared less intense in the Kingwood sample and distinct conchoidal fractures developed around the coal particles and many of the coal particles were reacted to the extent that they were plucked out of the etched Kingwood surface. The Litwar had less staining, the colors were more yellow to brown than blue, and the edges of particles had very distinct cracks. The etching caused the cellular structure of some vitrinite to become very distinct. The Jewell coal showed very little staining but the conchoidal cracks around particles became very distinct. The Poca #12 when etched did not develop stains but showed the most extensive fracturing around the coal particles.

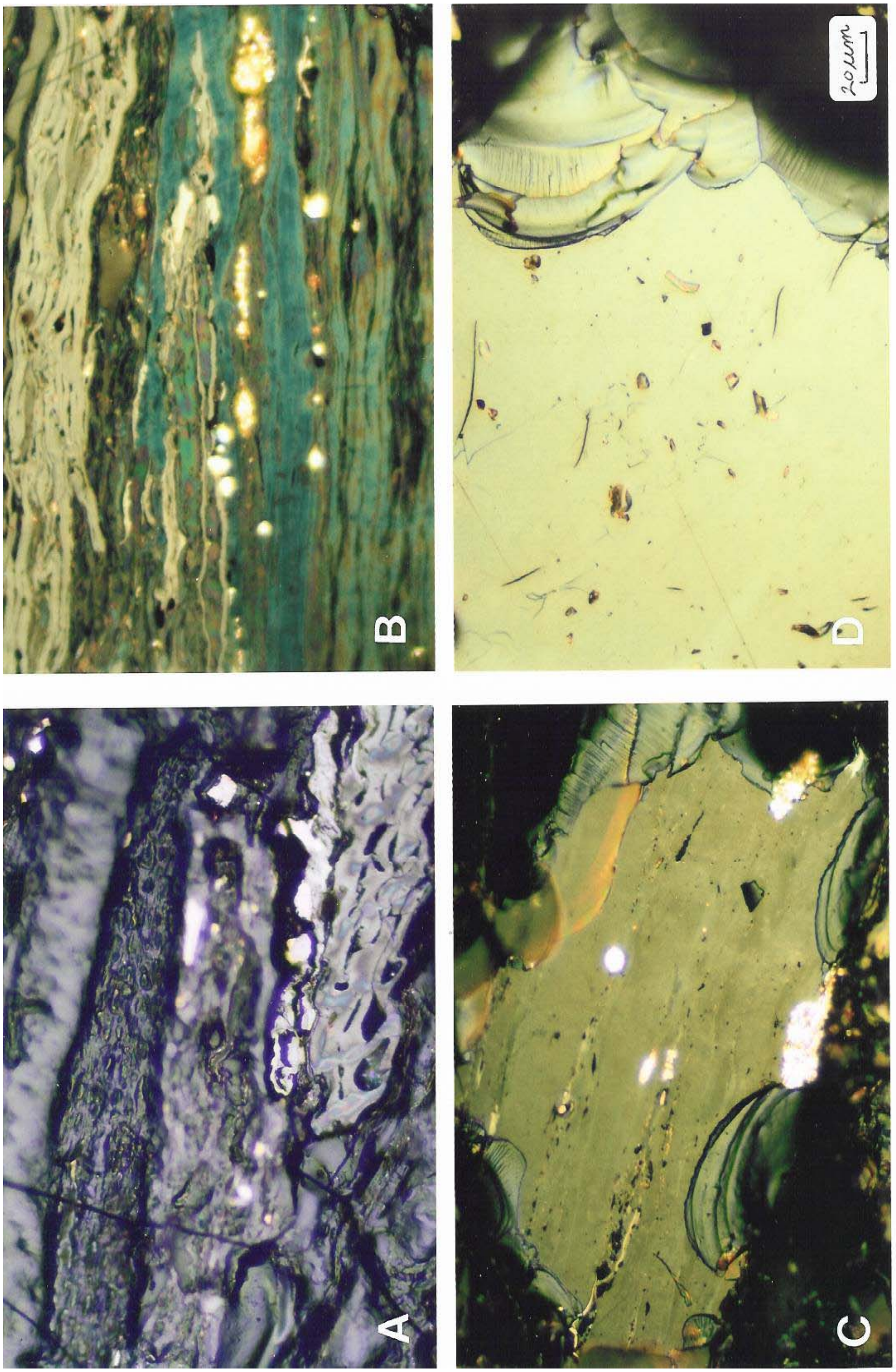
The lower rank coals, including Ky #9, Shoemaker, and Bailey, showed greatest changes due to NMP etching. The Mingo Logan surface developed very distinct conchoidal cracks. The Litwar sample had very little staining and the colors shifted from blue to yellow and brown, with pronounced particle cracking around the edges. The Jewell and Poca #12 had very little staining and were most unchanged except for conchoidal cracks. The coal staining might be because NMP remains on the surface after the alcohol rinse. The internal crack patterns are probably due to shrinkage while the conchoidal edge fractures may be due to a pressure difference between the coal and mounting media.

As expected, the liptinite, inertinite, and mineral matter did not change because of etching. The staining appears to be rank related. All of the vitrinites except particulate attrital vitrinite stained in all but the higher rank coals. The kind and amounts of fractures also appear to be related to vitrinite maturity. A quantitative relationship between etching characteristics and extractability was not established. Examples of the solvent-etch coal surfaces are shown in Figures 19, 20, and 21.



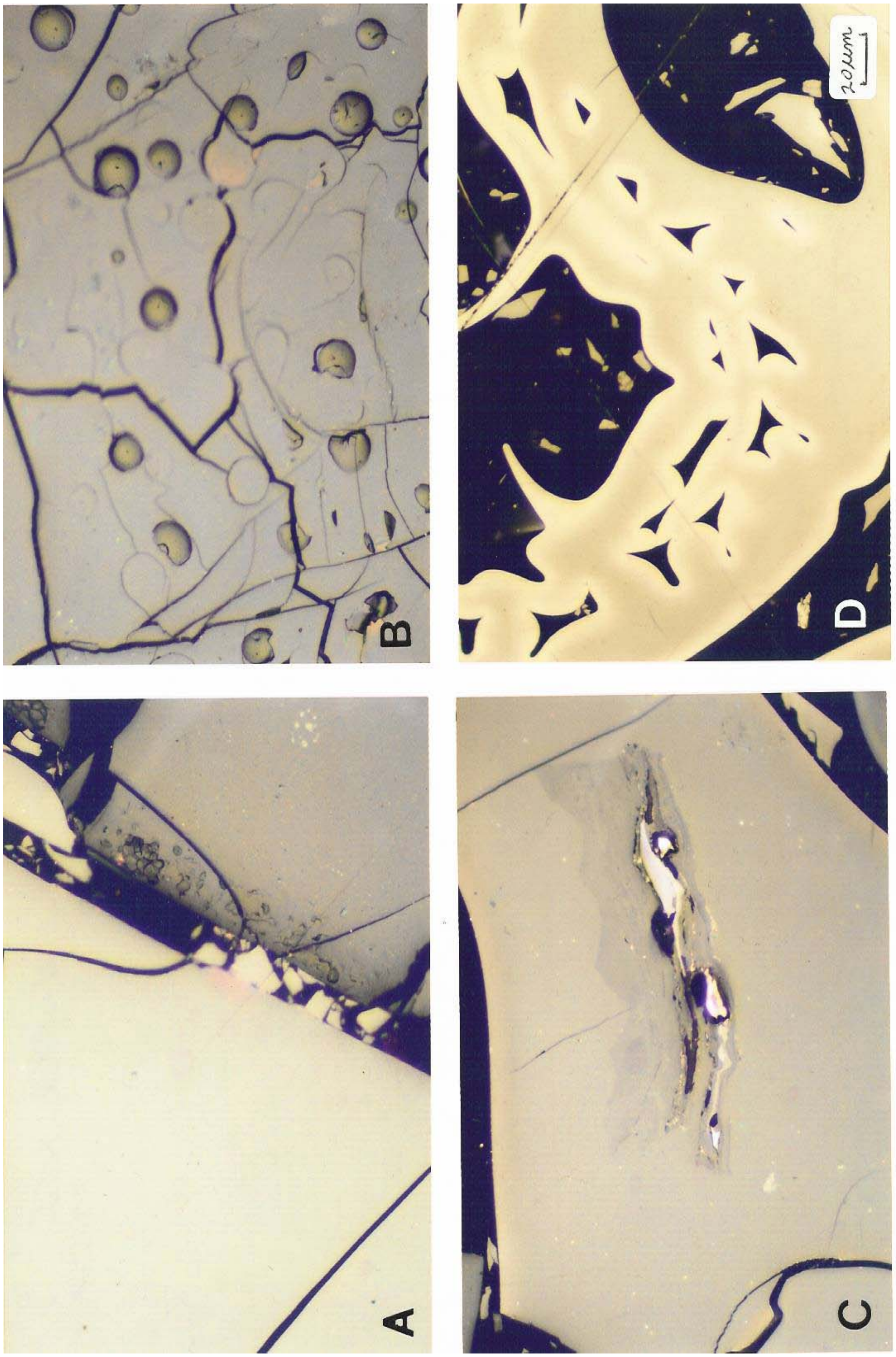
Photomicrographs of NMP Etched Pittsburgh Coal From Shoemaker Mine, Marshall County, West Virginia Showing; A=Etched Attrital Coal, EC=Etched Collinite and UE=Unetched Collinite, C=Cracks and P=Plastic. Reflected Light In Air, X160.

Figure 19. Optical microscopy of NMP-etched coal.



Photomicrographs of NMP Etched Coals Showing; A=Various Shades of Dark Blues Stains In Deeply Etched Kentucky No. 9; B=Bright Blue and Green Stains in Banded Bailey Coal; C=Olive Stains In Kingwood Coal and Concoidal Fractures an Particle Edges; and D=Unstained Poca 12 Coal With Concoidal Cracks. A Through D Arranged In Order of Inceasing Rank. Reflected Light In Oil, X600.

Figure 20. Optical microscopy of NMP-etched coal.



Photomicrographs of NMP Extracted Pitch Showing; A - Has White (most Altered) and Gray (Less Altered) Particles; B - Showing Circular Areas of NMP Entrapment; C - Shows Partially Altered Coal; and D - Shows Flow Patterns With Lighter (Oxidized) Surfaces. Reflected Light In Oil, X600.

Figure 21. Optical microscopy of NMP-etched coal.

5.3.4 Microscopic Properties of NMP-Soluble Extracts

The extracts were heterogeneous in terms of their degree of alteration and flow characteristics, and NMP, coal, and oxidized content, as shown in Table 10. The extracts contained areas that ranged from white (bright) to various shades of gray (dark). This indicated differences in treatment of the extract. There are flow shapes which are also related to how the extracts were processed. Several of the extracts have NMP inclusions. All of the extracts have traces of coal, with small amounts of coal in Shoemaker and Bailey, and a large amount in the Poca #12, as summarized in Table 10. All of the extracts have some oxidized material. It is most abundant in the Shoemaker, Bailey, Kingwood, and Litwar coals, which were prepared for microscopy at the same time.

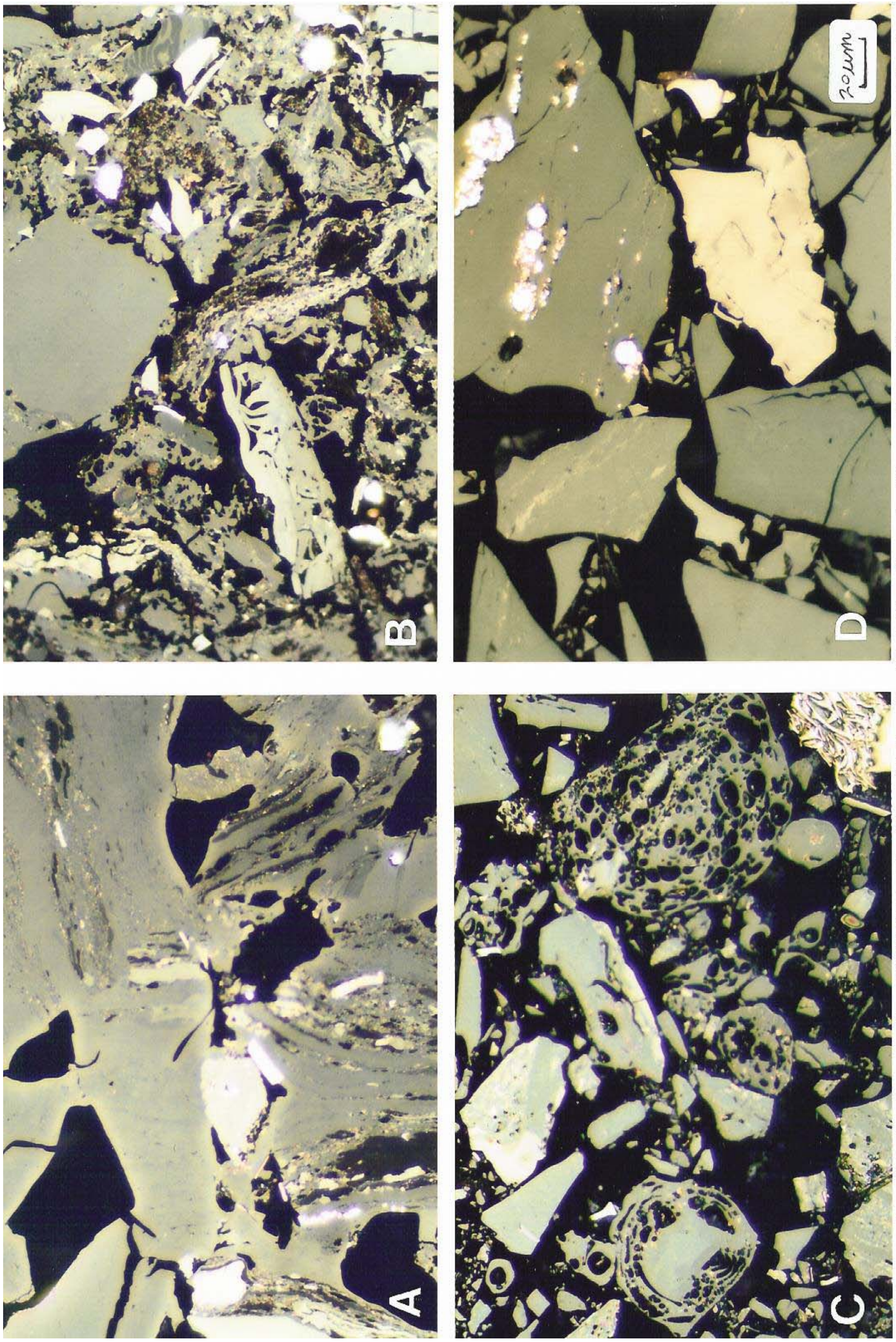
Table 10. Microscopic properties of NMP-soluble extracts

Decreasing Reflectance ⟶								
Coal	White	Light Gray	Darker Gray	Darkest Gray	Flow Shapes	NMP	Coal	Oxidized
Ky #9	81.7	14.0	--	--	3.3	0.3	--	0.7
Shoemaker	62.5	20.0	1.0	6.7	1.3	1.3	0.7	6.5
Bailey	50.5	34.6	4.3	--	1.0	T	0.3	9.3
Mingo Lo.	80.7	9.6	--	0.3	4.7	0.7	T	4.0
Marfork	87.6	3.7	--	--	7.7	--	T	1.0
Kingwood	62.7	18.0	2.7	--	4.3	0.3	T	12.1
Litwar	62.4	20.7	1.0	--	3.3	--	T	12.2
Jewell	73.5	3.3	1.3	--	17.6	1.3	T	3.1
Poca #12	6.0	57.5	3.0	0.3	2.7	2.7	26.5	1.4

T= Occurred in count area but not in count.

5.3.5 Microstructures in NMP-Insoluble Residues

The insoluble residues contained a variety of relatively distinct microstructures that range from unaltered or rounded particles to particles with altered edge surfaces. In addition, all of the samples had some fused agglomeration. Some particles showed distinct pore development and all of the samples displayed oxidation, Figure 22 and Table 11.



Photomicrographs of NMP Residue From Extraction Test Showing; A – Shows Coarse Coal Agglomerate With Oxidized Particle Borders From Extracted Bailey Coal; B- Shows Loose Aggregates of Fine Size High Inert Coal From Kingwood Residue; C – Shows Spherical Porous Coal Alteration Products in the Jewell Extraction Residue and D – Shows Unaltered Coal Particles From the Poca #12 Extraction Residue. Reflected Light In Oil, X600.

Figure 22. Insoluble residue from NMP extraction.

Table 11. Microstructures in NMP-insoluble residues.

			Reacted	Agglomerates			
Coal	Unaltered	Rounded	Edges	Coarse	Fine	Pores	Oxidized
Ky #9	14.8	6.8	13.6	56.4	7.6	0.0	0.8
Shoemaker	9.6	5.6	0.0	78.4	0.0	0.0	6.4
Bailey	12.8	6.4	0.0	72.8	0.0	0.0	8.0
Mingo Lo.	44.4	13.6	10.4	29.6	0.0	0.0	2.0
Marfork	17.6	4.0	9.2	60.4	8.0	0.0	0.8
Kingwood	11.8	15.6	4.8	28.4	23.6	14.4	1.4
Litwar	14.2	7.0	9.6	39.6	20.8	6.4	2.4
Jewell	30.0	18.8	17.6	1.2	2.0	26.8	3.6
Poca #12	61.6	20.4	14.0	1.2	0.0	0.0	2.8

The coal with the most liptinite and inerts, Mingo Logan, had a large amount of unaltered coal. The highest rank coals, Jewell and Poca #12, also had significant amounts of unaltered coal. All of the coals but the higher rank Jewell and Poca #12 had large amounts of fused agglomerates or aggregates. The Kingwood, Litwar, and Jewell had particles with distinct pore development. All of the residues had some oxidized coal particles particularly Bailey and Shoemaker. The Ky #9, Shoemaker, and Bailey had large amounts of aggregates which may cause filtering or separation problems. This effect was borne out in the laboratory extraction studies.

5.3.6 Reflectance Characteristics of Coal, Extract, Residue, FSI Coke

The vitrinite reflectance of coals increases with coal rank as shown in column one in Table 12. The reflectance of the vitrinite in the extracts and residues has a similar pattern but differs in the actual volumes. The Shoemaker, Bailey, Kingwood, and Litwar all have significant amounts of oxidized materials in the insoluble residues and extracts. The estimated R_o from the coal FSI coke parallels that of the coal reflectances, except for the Kingwood estimated reflectances which are high. The estimated reflectances from the extract FSI show the same rank trends as that of the coal, but the Kingwood is higher in the FSI coke. The estimated reflectance from the insolubles shows no trends with the other reflectance data. This may be due to significant oxidation of the residue used to make the FSI button.

The vitrinite reflectances of the extracts were generally lower than the vitrinite reflectances of the feed coals. The Ky #9 extract sample appeared relatively uniform in reflectance (0.59% R_o). The Bailey extract is similar to Shoemaker, but contains less NMP and more evidence of oxidation. The Shoemaker extract appeared variable in reflectance averaging 0.71 %R_o . In addition to cracks, some particles had round cavities that appear to be due to entrapped NMP. Most of the samples also contained oxidized coal and a small amount of undissolved attrital coal.

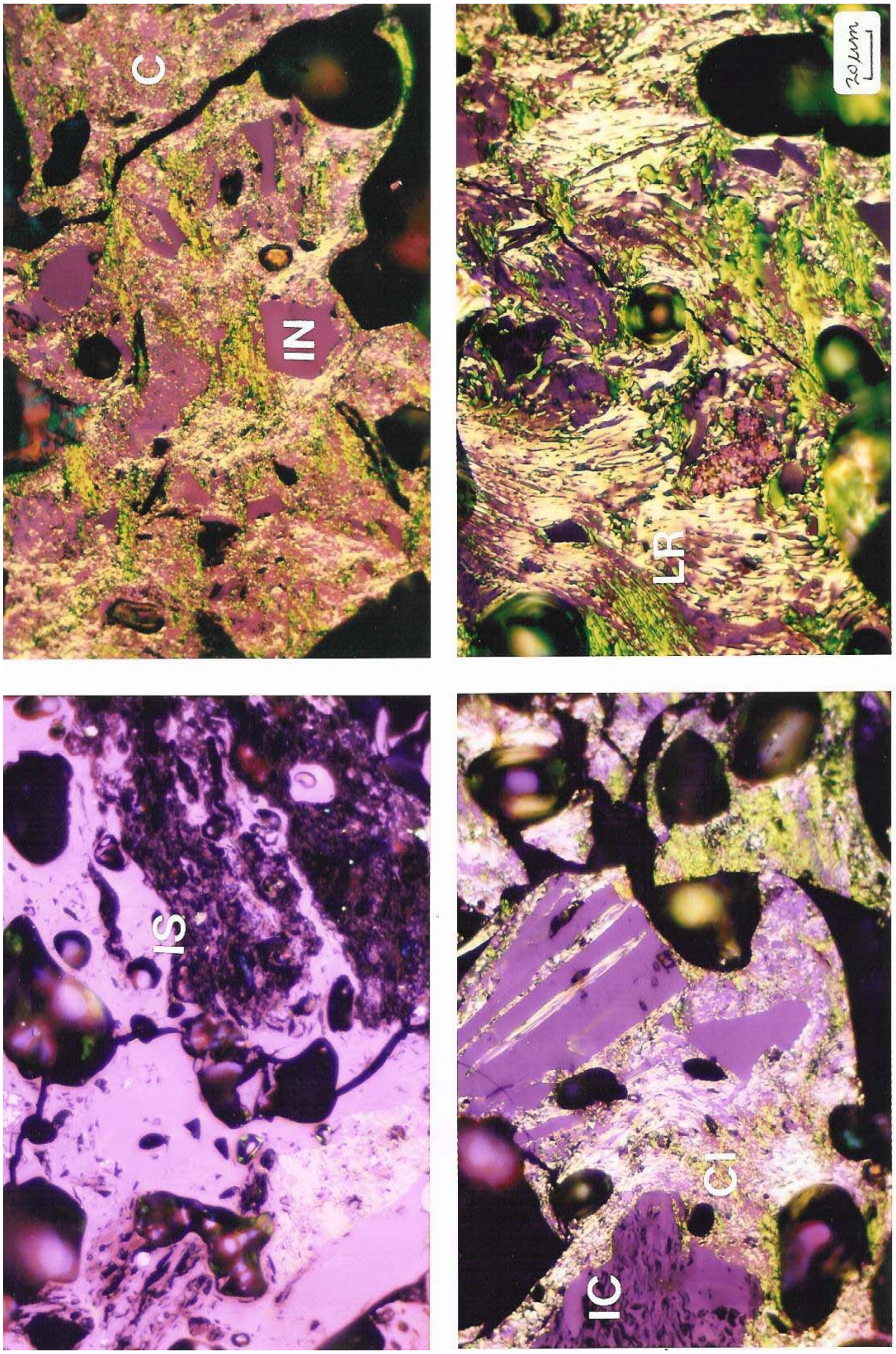
Table 12. Reflectance of indicated samples in the order of increasing rank.

Coal	Coal Vitrinite Mean Max., $\%R_o$	Vitrinite In Extract, $\%R_o$	Vitrinite In Residue, $\%R_o$	Estimated $\%R_o$ From Coal FSI	Estimated $\%R_o$ From Residue FSI	Estimated $\%R_o$ From Extract FSI
Ky #9	0.53	1.59	0.57	1.60	0.60	0.66
Shoemaker	0.77	0.71	0.66	0.90	0.60	0.86
Bailey	0.83	0.76	0.73	0.90	0.60	0.89
Mingo Logan	0.98	0.83	0.86	0.99	0.79	1.07
Marfork	1.02	0.85	0.96	1.02	0.80	1.07
Kingwood	1.08	1.00	1.00	1.24	0.65	1.21
Litwar	1.23	1.09	1.14	1.25	0.68	1.26
Jewell	1.36	1.13	1.38	1.30	1.25	1.38
Poca #12	1.53	1.04	1.56	1.50	1.48	1.46

The Mingo Logan extract averages 0.83% R_o. In addition to cracks, some particles have traces of what appears to be NMP as well as partially dissolved coal. The Marfork extract has only slightly variable reflectance averaging 0.85% R_o . The Kingwood extract is homogeneous and has an average reflectance of 1.0% R_o and contains some normal cracks. In addition, this extract has a significant level of unusual features that appear to be due to plastic deformation and flow. There are traces of coal and a relatively large amount of oxidized surfaces. The Litwar extract has variable gray levels and an average reflectance of 1.09% R_o . It has some cracks and distinct plastic deformation features. It also has some thermal oxidation around the particles and cracks. The Jewell extract is relatively uniform in gray levels and has an average reflectance of 1.13% R_o . It contains the normal compression cracks in addition to some fusiform voids and a significant amount of flow structures and some oxidized coal. The Poca #12 extract has an unusually low reflectance of 1.04% R_o . The extract sample is different than all of the previous extracts. It consists of a continuous matrix binder phase with relatively distinct coal inclusions. Most of the coal inclusions are angular and consist of coal inerts and/or dull coal particles with attrital vitrinite, liptinite, and inertinite.

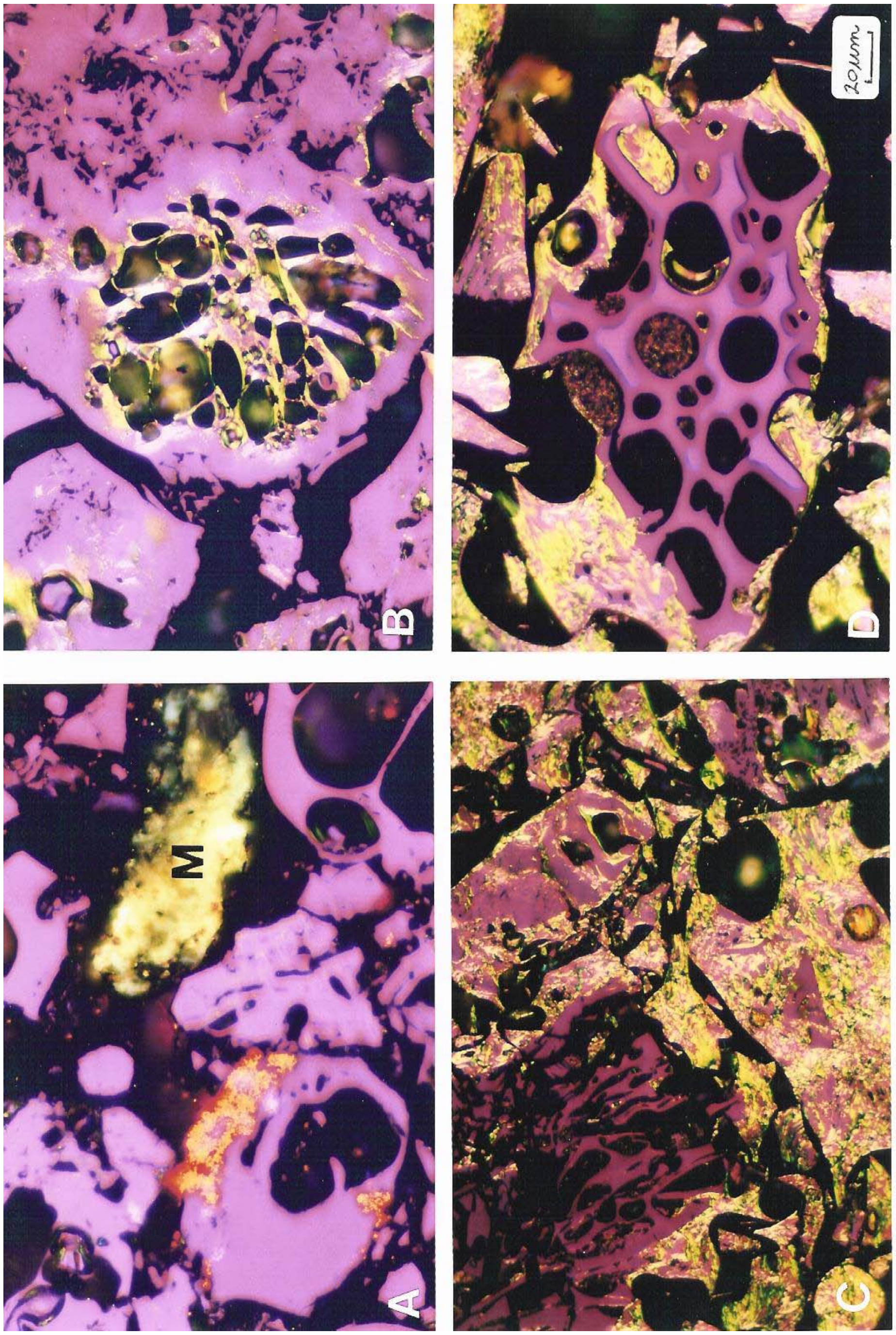
5.3.7 Petrographic Analysis of FSI Coke from Coal, Extract, Residue

The binder phase in cokes is produced from the reactive macerals and the filler phase is produced from incorporated inerts. During carbonization, the development of anisotropy increases upon moving up in rank from low rank bituminous to low volatile bituminous coals. The organic inerts are isotropic in all cokes while the binder phase carbon forms from the reactive macerals. A measure of anisotropy is expressed by an anisotropic index; the higher the index the more anisotropic the carbon. The feed coals, extracts, and residues produced in the FSI determinations were analyzed petrographically Figures 23, 24, and 25, respectively, and the anisotropic index calculated, Table13 .



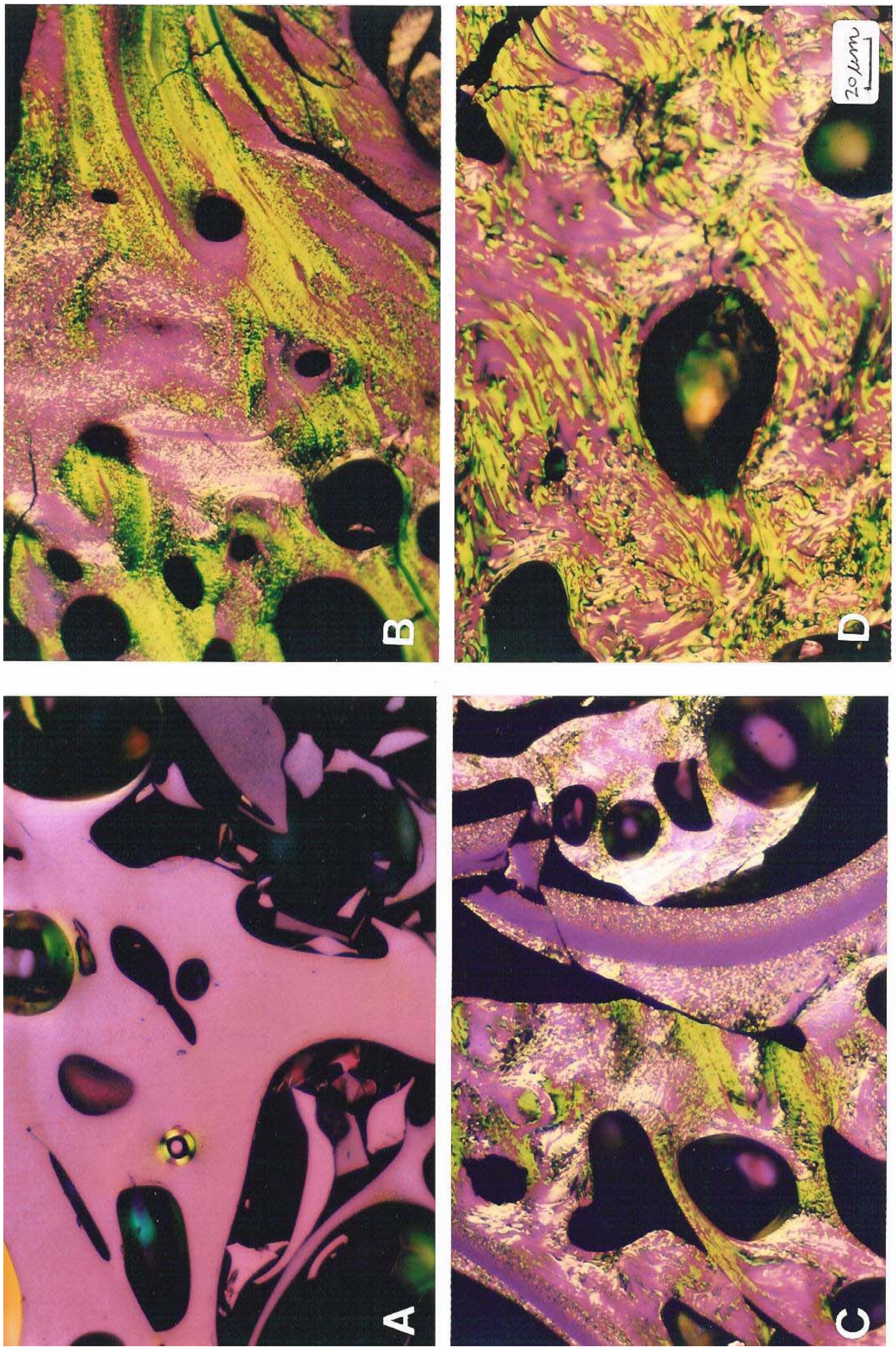
Photomicrographs of FSI Cokes From the NMP Test Coals Showing Coke Microstructures; IS=Isotropic and Mineral Matter In KY #9 FSI, C=Circular Anisotropic Carbons With Fine Size Microtexture, IN=Issotropic Inerts From Marfork FSI, CI=Coarse Circular to Fine Size Lenticular Anisotropic Binder Phase With IC=Coarse Isotropic Inert Fller Phase and LR=Lenticular to Ribbon Anisotropic Domains in FSI Coke From Poca #12 Coal. Reflected Polarized Light In Oil, X600.

Figure 23. Photomicrographs of FSI cokes from feed coals.



Photomicrographs of FSI Cokes From the NMP Extraction Residues Showing; Photo A – Isotropic Carbon With M=Mineral Matter In Shoemaker FSI, Photo – B Shows Anisotropic Sphere in Isotropic Carbon From Litwar FSI, Photo – C Shows Coarse and Fine Size Isotropic Filler Carbon In Anisotropic Binder Phase In Jewell FSI and Photo – D Shows Coarse Isotropic Inert Filler Phase In Anisotropic Carbon From Poca #12 FSI Coke. Reflected Polarized Light In Oil With Gypsum Tint Plate, X600.

Figure 24. Photomicrographs of FSI cokes from insoluble residues.



Photomicrographs of FSI Cokes From the NMP Pitch Extracts Showing; Photo A – Isotropic Carbon from KY #9, Photo B – Shows Circular Anisotropic Carbon With Strain Patterns From Mingo Logan, Photo C – Shows Circular to Lenticular Carbon With Isotropic Oxidized Carbon From Kingwood and Photo D – Shows Lenticular to Ribbon Anisotropic Microtextures From Jewell FSI Coke. Reflected Polarized Light In Oil With Gypsum Tint Plate, X600.

Figure 25. Photomicrographs of FSI cokes from extracts.

All of the FSI cokes from coals have relatively high amounts of binder phase from bright (high reactives) coals. The coke binder phase ranges from 67.2 to 78.4 percent by volume. FSI coke from extracts has only traces of coal inerts except the Poca #12 , which contain significant amounts of coal inerts. The anisotropic index ranges from 0.29 to 7.63 and increases with increased rank. The FSI coke from residues have anisotropic index values that range from 0.01 to 7.80, but also contain significantly more isotropic carbon than either the feed coal or extract.

Table 13. Summary petrographic analysis of FSI coke from coal, extract, and residue

Coal	Coal FSI Coke			Residue FSI Coke			Extract FSI Coke	
	Binder Phase	Filler Phase	AI	Binder Phase	Filler Phase	AI	Binder Phase	AI
Ky #9	73.3	26.7	0.02	54.2	45.8	0.01	100	0.29
Shoemaker	70.9	29.1	2.03	55.2	44.8	0.00	100	1.64
Bailey	78.4	21.6	2.05	62.8	37.2	0.01	100	1.86
Mingo Lo.	67.2	32.8	2.94	50.8	49.2	1.13	100	3.69
Marfork	71.2	28.8	3.87	33.9	66.1	1.16	100	3.74
Kingwood	78.0	22.0	5.41	24.6	75.4	0.33	100	5.06
Litwar	70.2	29.8	5.49	52.4	47.6	.054	100	5.63
Jewell	78.1	21.4	5.99	53.6	46.2	5.46	100	6.76
Poca #12	77.6	22.4	7.98	77.9	22.1	7.80	100	7.63

AI = anisotropic index

5.4 Results of WVU Coal Extraction Pilot Plant Runs

Based on the results of the research, the Kingwood coal was chosen for processing in the WVU coal extraction pilot plant. About 22lb of ¼ inch coal were added to 100L of NMP in the continuously stirred extractor. The coal was added slowly using a scoop while the reactor was stirring. The mixture was then heated to about 190°C and held at this temperature for 20 minutes. Experience with other coals showed that most of the coal is extracted at this temperature and time. A process flow diagram is shown in Figure 26. After the extraction was complete, the mixture was then pumped (Pump 1) through a basket filter to remove any large undissolved particles before being sent to the centrifuge. The filter is a 15-gallon chrome plated vessel with a removable stainless steel 60 mesh screen filter basket.

The filtered mixture was then pumped (Pump 2) to the centrifuge to remove the remaining undissolved coal and mineral matter, and then sent to a holding tank before being sent to the Ross unit.

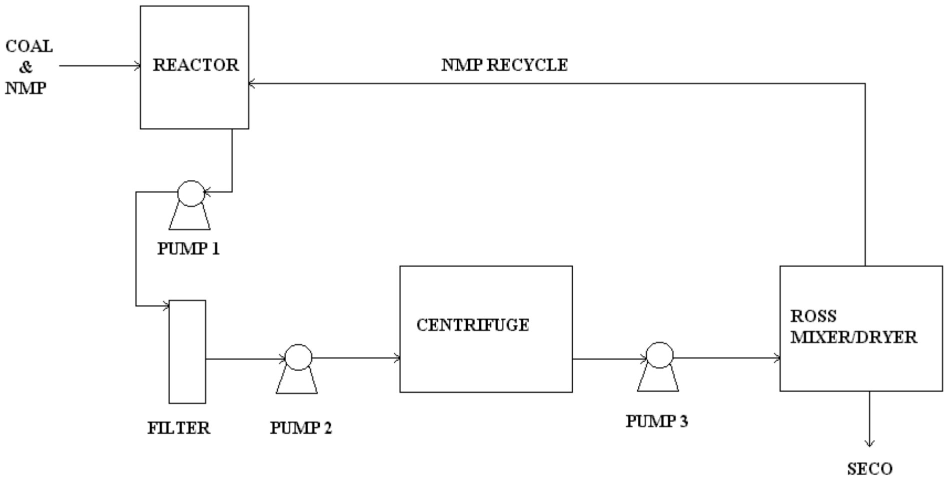


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Figure 26 Flow diagram of the WVU coal extraction pilot plant

The liquid mixture is then pumped (Pump 3) into the Ross drier where the solvent is stripped and recycled leaving a dry, powdered coal extract. Mass balance showed that about 61wt% of the coal was extracted (about 67wt% in laboratory trials) but contained over 2wt% ash. It was felt that the system was contaminated from previous runs. Thus another attempted to extract the Kingwood coal was conducted under identical conditions. Again, extraction yields were over 60wt% but with an extract ash content of about 0.2wt%. The Kingwood coal underwent processing through the pilot scale system without any difficulty whatsoever and as one of the engineers stated is a “joy” with which to work.

6 CONCLUSIONS

The results of this research show that several methods can be used to aid in the selection of bituminous coals for solvent extraction, particularly under mild conditions where coal composition is especially important. Some of the more effective tests involve the determination of thermoplasitc behavior in which Gieseler plasticity and Audibert-Arnu dilatometry are helpful. Proximate, FTIR, and elemental analyses are also useful in defining rank parameters and in predicting the chemical nature of the coal and extract. Petrographic examination and analysis are especially necessary to the successful operation of the process in determining the quantity, quality, and susceptibility of vitrinite to extraction.

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APPENDIX 1
Feed Coals
Basic Analyses

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell/WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Mean Max Vitrinite Reflectance R_o, %	*0.53*	*0.77*	*0.83*	*0.98*	*1.02*	*1.08*	*1.23*	*1.36*	*1.53*
Sulfur, %dry	3.26	3.12	1.91	0.76	1.06	1.02	0.74	0.92	1.11
Proximate, %dry									
Volatile Matter	36.65	38.06	36.22	35.00	33.45	33.17	27.42	22.77	19.63
Fixed Carbon	53.52	51.92	56.11	59.27	60.05	57.91	68.09	70.63	76.63
Ash	9.83	10.02	7.67	5.73	6.50	8.92	4.49	6.60	3.83
Elemental Analysis, wt%									
C	68.94	72.37	74.74	78.08	80.52	77.44	82.57	84.6	85.96
H	5.13	5.1	5.25	5.16	5.21	4.95	5.02	4.76	4.68
N	1.34	1.22	1.18	1.12	1.13	1.18	1.32	0.93	1.06
S	2.72	2.59	1.56	0.66	0.72	1.58	0.6	0.7	0.99
O by difference	21.87	18.72	17.27	15.64	12.42	14.85	10.49	9.01	7.31
C/H atomic ratio	1.124	1.176	1.190	1.266	1.30	1.515	1.538	1.538	1.531

APPENDIX 2

Feed Coals and Residues
Surface and Bulk Properties

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell/WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Real Density, g/cm^3									
Feed Coal	1.374	1.330	1.370	1.361	1.364	1.404	1.356	1.394	1.369
Insoluble Residue	1.432	1.426	1.402	1.365	1.992	1.649	1.417	1.398	1.359
BET Multipoint Surface Area, m^2/g									
Feed Coal	18.209	0.664	0.514	0.472	0.559	0.429	0.531	0.664	0.532
Insoluble Residue	0.243	0.538	0.451	0.481	0.484	0.992	0.959	0.655	0.291
Mercury Porosimetry									
Feed Coals									
Median Pore Diameter (Volume), µm	ND	14.149	11.820	ND	ND	12.383	10.982	ND	ND
Median Pore Diameter (Area), µm	ND	0.0052	0.0046	ND	ND	0.0033	0.0045	ND	ND
Apparent Density, g/cm^3	ND	1.320	1.343	ND	ND	1.267	1.315	ND	ND
Insoluble Residues									
Median Pore Diameter (V), µm	ND	32.216	24.290	ND	ND	24.099	17.579	ND	ND
Median Pore Diameter (A), µm	ND	0.0064	0.0050	ND	ND	0.0051	5.043	ND	ND
Apparent Density, g/cm^3	ND	1.408	1.427	ND	ND	1.697	1.186	ND	ND

ND = not determined

APPENDIX 3
Feed Coals
Thermoplastic Properties

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell/ WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Mean Max Vitrinite Reflectance R_o, %	0.53	0.77	0.83	0.98	1.02	1.08	1.23	1.36	1.53
Yield NMP Solubles, wt%	30.3	34.1	30.9	32.4	45.3	66.7	40.6	30	5
Yield NMP Solubles, daf%	33.6	37.9	33.5	34.4	48.4	73.2	42.5	32.1	5.2
Free Swelling Index, FSI									
FSI	4.0	7.0	7.5	7.5	7.5	9.0	8.5	9.0	9.0
Gieseler Plastometry									
Max ddpm	9	12019	771	20246	29999	30000	11009	1914	551
Temp Max ddpm, °C	422	429	440	441	453	456	456	471	476
Temp Softening, °C	382	382	400	394	391	369	397	409	424
Temp Soldification, °C	443	465	471	483	489	500	499	509	506
Plastic Range, °C	61	83	71	89	98	121	102	100	82
Arnu Dilatometry									
Max Contraction, %	-22	-28	-30	-24	-29	-32	-30	-25	-29
Max Dilatation, %	-35	185	73	149	300	300	271	182	114
Init Softening Point, °C	356	347	360	354	349	346	362	393	399
Init Dilatation Temp, °C	403	390	400	406	395	382	403	418	434
Max Dilation Temp, °C	427	441	433	452	478	466	476	480	479

APPENDIX 4

Thermoplastic Properties
Feed Coals, Extracts, and Residues

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Feed Coals									
Free Swelling Index, FSI									
FSI	4.0	7.0	7.5	7.5	7.5	9.0	8.5	9.0	9.0
Gieseler Plastometry									
Max ddpm	9	12019	771	20246	29999	30000	11009	1914	551
Temp Max ddpm, °C	422	429	440	441	453	456	456	471	476
Temp Softening, °C	382	382	400	394	391	369	397	409	424
Temp Soldification, °C	443	465	471	483	489	500	499	509	506
Plastic Range, °C	61	83	71	89	98	121	102	100	82
Arnu Dilatometry									
Max Contraction, %	-22	-28	-30	-24	-29	-32	-30	-25	-29
Max Dilatation, %	-35	185	73	149	300	300	271	182	114
Init Softening Point, °C	356	347	360	354	349	346	362	393	399
Init Dilatation Temp, °C	403	390	400	406	395	382	403	418	434
Max Dilation Temp, °C	427	441	433	452	478	466	476	480	479
Residues									
Free Swelling Index, FSI									
FSI	0.5	0.5	0.0	1.0	1.0	0.5	0.0	7.5	9
Gieseler Plastometry									
Max ddpm	0	0	0	12	4	0	0	3	95
Temp Max ddpm, °C	na	na	na	449	457	na	na	479	480
Temp Softening, °C	na	na	na	436	436	na	na	459	437
Temp Soldification, °C	na	na	na	470	475	na	na	499	511
Plastic Range, °C	na	na	na	34	39	na	na	40	74
Max ddpm	0	0	0	12	4	0	0	3	95
Extracts									
Free Swelling Index, FSI									
FSI	9.0	9.0	9.0	9.0	9.0	9.0	9.0	9.0	9.0
Gieseler Plastometry									
Max ddpm	2438	5500	6522	30000	29967	17782	11089	30000	30000
Temp Max ddpm, °C	408	428	429	422	431	445	444	435	350
Temp Softening, °C	333	333	351	318	337	354	342	308	<300
Temp Soldification, °C	454	474	474	490	489	494	495	512	507
Plastic Range, °C	121	141	123	172	152	140	153	204	>207
Max ddpm	2438	5500	6522	30000	29967	17782	11089	30000	30000

APPENDIX 5
Feed Coals, Residues, and Extracts
FTIR Analysis

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell/ WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Mean Max Vitrinite Reflectance R_o, %	0.53	0.77	0.83	0.98	1.02	1.08	1.23	1.36	1.53
Yield NMP Solubles, wt%	30.3	34.1	30.9	32.4	45.3	66.7	40.6	30	5
Yield NMP Solubles, daf%	33.6	37.9	33.5	34.4	48.4	73.2	42.5	32.1	5.2
Feed Coal									
Hal[1]	29.856	39.236	42.948	36.229	53.396	46.604	30.325	40.8605	NA
Har[2]	0.487	0.684	3.724	2.5275	5.5495	5.481	6.219	7.5625	NA
Har/Hal	0.016311	0.017433	0.08671	0.069765	0.103931	0.117608	0.205078	0.185081	NA
Insoluble Residue									
Hal[1]	25.0845	NA	NA	22.9815	22.493	3.156	10.193	44.0625	NA
Har[2]	0.329	NA	NA	0.585	1.5365	0.599	1.34	8.888	NA
Har/Hal	0.013116	NA	NA	0.025455	0.06831	0.189797	0.131463	0.201713	NA
Extracts									
Hal[1]	53.0045	31.363	50.724	76.7235	59.316	40.686	58.32	52.7065	NA
Har[2]	0.978	0.465	1.748	7.191	7.0345	3.399	7.869	9.231	NA
Har/Hal	0.018451	0.014826	0.034461	0.093726	0.118594	0.083542	0.134928	0.149037	NA

[1] Integrated IR intensity associated with aliphatic C-H stretching mode

[2] Integrated IR intensity associated with aromatic C-H stretching mode
NA = not applicable or available

APPENDIX 6

Maceral Analysis-Feed Coal
Volume Percent

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Mean Max Vitrinite Reflectance R_o, %	0.53	0.77	0.83	0.98	1.02	1.08	1.23	1.36	1.53
Vitrinite Group									
Collinite	41.1	37.8	38.3	24.6	28.1	39.8	31.0	39.5	53.9
Telinite	13.9	5.8	3.4	4.0	5.2	9.5	1.1	0.0	1.0
Attrital Fine	7.5	10.1	15.6	14.4	15.9	12.9	18.7	9.0	8.1
Attrital Band	6.8	15.3	10.0	7.6	12.5	10.3	16.5	9.4	7.2
Pseudovitrinite	1.4	3.2	6.4	3.0	3.5	2.1	3.3	18.6	9.2
Total Vitrinite Group	70.7	72.2	73.7	53.6	65.2	74.6	70.6	76.5	79.4
Liptinite Group									
Sporinite	4.7	5.0	5.1	9.6	4.5	2.8	4.8	2.3	0.7
Cutinite	0.5	0.2	0.1	0.8	0.4	0.7	0.2	0.2	0.1
Resinite-Light	2.1	1.4	1.1	2.8	0.8	1.3	1.3	0.4	0.3
Resinite-Dark	0.2	0.4	0.2	0.3	0.8	0.2	0.0	0.0	0.1
Total Liptinite Group	7.5	7.0	6.5	13.5	6.5	5.0	6.3	2.9	1.2
Inertinite Group									
Semifusinite	4.7	3.8	5.7	6.6	7.5	3.2	2.7	5.4	5.3
Micrinite-Granular	5.4	3.8	4.7	11.1	6.7	6.9	8.5	5.2	4.9
Micrinite-Macrinite	0.4	1.8	0.3	2.0	1.3	0.3	0.7	0.5	0.8
Micrinite-Inertodetrinite	2.8	3.8	2.3	6.4	3.1	2.4	4.5	2.5	3.3
Fusinite	2.9	3.4	3.4	3.9	5.3	2.3	4.1	2.6	2.0
Total Inertinite Group	16.2	16.6	16.4	30.0	23.9	16.1	20.5	16.2	16.3
Mineral Matter									
Pyrite	1.1	1.4	0.8	0.5	0.6	0.9	0.3	0.6	0.9
Clay	1.8	1.0	1.4	1.5	2.0	0.4	1.5	2.2	1.8
Carbonates	0.0	Trace	0.0	0.1	0.0	1.1	0.2	0.0	0.1
Calcite	0.4	0.2	0.0	0.0	0.0	1.2	0.1	0.2	0.0
Quartz	0.5	0.8	1.0	0.3	0.7	0.3	0.1	0.0	0.2
Shale	0.2	0.6	0.2	0.3	0.2	0.4	0.3	0.0	Trace
Bone	1.6	0.2	0.0	0.2	0.9	0.0	0.1	1.4	0.1
Rust	0.0	Trace	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total Mineral Matter	5.6	4.2	3.4	2.9	4.4	4.3	2.6	4.4	3.1
Total All Groups	100	100	100	100	100	100	100	100	100

APPENDIX 7

Maceral Analysis-Insoluble Residue
Volume Percent

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Vitrinite Group									
Collinite	30.2	44.6	50.5	20.9	5.4	36.5	39.3	47.4	54.6
Telinite	2.9	2.0	0.8	2.2	0.5	0.0	0.0	0.7	0.6
Attrital Fine	14.6	5.1	3.4	14.2	18.6	6.3	10.8	12.6	8.0
Attrital Band	17.6	5.8	7.7	6.2	12.1	3.0	7.9	6.2	12.4
Pseudovitrinite	2.3	3.6	4.5	4.3	1.5	5.9	3.4	5.1	3.3
Total Vitrinite Group	67.6	61.1	66.9	47.8	38.1	51.7	61.4	72.0	78.9
Liptinite Group									
Sporinite	6.1	5.9	6.1	9.4	9.2	9.9	3.7	2.7	0.9
Cutinite	0.5	0.7	0.7	0.5	0.5	0.6	0.1	0.0	0.1
Resinite-Light	2.2	0.6	0.8	2.8	4.0	0.9	0.5	0.2	0.1
Resinite-Dark	0.2	0.0	0.0	0.2	0.2	0.0	0.0	0.0	0.0
Total Liptinite Group	9.0	7.2	7.6	12.9	13.9	11.4	4.3	2.9	1.1
Inertinite Group									
Semifusinite	3.6	6.1	4.6	8.6	12.2	6.5	7.2	4.7	6.6
Micrinite-Granular	3.5	4.8	4.1	13.1	13.6	2.8	9.3	5.9	4.0
Micrinite-Macrinite	0.9	1.6	0.5	1.6	1.7	1.5	1.6	0.9	0.9
Micrinite-Inertodetrinite	5.4	7.6	6.0	6.9	10.0	6.2	7.8	3.8	3.2
Fusinite	2.4	4.2	4.2	4.8	3.6	3.5	3.3	4.2	3.0
Total Inertinite Group	15.8	24.3	19.4	35.0	41.1	20.5	29.2	19.5	17.7
Mineral Matter									
Pyrite	1.7	2.7	1.2	0.6	0.7	2.1	0.5	0.4	0.5
Clay	4.3	2.3	2.4	2.2	4.3	8.9	2.9	2.3	1.0
Carbonates	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.0	0.0
Calcite	0.0	0.2	0.2	0.2	0.1	0.2	0.2	0.4	0.0
Quartz	0.6	0.3	0.2	0.3	0.3	0.7	0.2	0.5	0.1
Shale	0.1	1.1	0.7	0.2	0.1	0.9	0.5	0.4	0.2
Bone	0.9	0.8	1.4	0.8	1.3	3.6	0.8	1.6	0.5
Rust	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total Mineral Matter	7.6	7.4	6.1	4.3	6.9	16.4	5.1	5.6	2.3
Total Inertinite + MM	23.4	31.7	25.5	39.3	48.0	36.9	34.3	25.1	20.0
Total All Groups	100	100	100	100	100	100	100	100	100

APPENDIX 8
Feed Coals
Vitrinoid-Type Distribution
Reflectance Analysis

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell/ WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Reflectance Analysis, %									
V4	37.0								
V5	51.0								
V6	12.0	4.0	1.0						
V7		74.0	21.0						
V8		22.0	72.0	6.0	8.5	1.0			
V9			6.0	56.0	33.0	13.0			
V10				38.0	36.5	46.0			
V11					20.5	38.0	33.0		
V12					1.5	2.0	57.0	9.5	
V13							10.0	75.5	1.0
V14								13.0	25.0
V15								2.0	67.5
V16									6.0
V17									0.5
Total	100	100	100	100	100	100	100	100	100

APPENDIX 9

NMP-Soluble Extract
Vitrinoid-Type Distribution
Reflectance Analysis

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell/ WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Reflectance Analysis, %									
V5	64.0	4.0							
V6	36.0	36.0							
V7		56.0	90.0	24.0					
V8		4.0	10.	76.0	100.0				
V9						48.0			10.0
V10						52.0	64.0	18.0	88.0
V11							36.0	80.0	2.0
V12								2.0	
V13									
V14									
V15									
V16									
V17									
Total	100	100	100	100	100	100	100	100	100
Mean Max Reflec Ext.	0.59	0.71	0.76	0.83	0.85	1.00	1.09	1.13	1.04
Microscopic Observations									
White Binder Phase	81.7	62.5	50.5	80.7	87.6	62.7	62.4	73.5	6.0
Light Gray Binder Ph	14.0	20.0	34.6	9.6	3.7	18.0	20.7	3.3	57.5
Darker Gray Bind Ph	0.0	1.0	4.3	0.0	0.0	2.7	1.0	1.3	3.0
Darkest Gray Bind Ph	0.0	6.7	0.0	0.3	0.0	0.0	0.0	0.0	0.3
Flow Shapes	3.3	1.3	1.0	4.7	7.7	4.3	3.3	17.6	2.7
NMP	0.3	1.3	Trace	0.7	0.0	0.3	0.0	1.3	2.7
Coal	0.0	0.7	0.3	Trace	Trace	Trace	Trace	Trace	26.5
Oxidized	0.7	6.5	9.3	4.0	1.0	12.0	12.6	3.0	1.3
Total, %	100	100	100	100	100	100	100	100	100

APPENDIX 10
Insoluble Residue
Vitrinoid-Type Distribution
Reflectance Analysis

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell/ WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Reflectance Analysis, %									
V4	2.0								
V5	78.0	10.0							
V6	20.0	74.0	32.0						
V7		12.0	58.0	12.0					
V8		4.0	10.0	66.0	26.0	2.0			
V9				22.0	42.0	46.0	2.0		
V10					26.0	50.0	16.0		
V11					6.0	2.0	70.0		
V12							12.0	6.0	
V13								62.0	
V14								30.0	8.0
V15								2.0	70.0
V16									22.0
V17									
Total	100	100	100	100	100	100	100	100	100
Mn Max Refl Residue	0.57	0.66	0.73	0.86	0.96	1.00	1.14	1.38	1.56

APPENDIX 11

Non-Maceral Microstructures-Feed Coal
Volume Percent

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Normal-Bright	48.8	45.5	45.1	28.2	35.6	50.3	44.0	33.8	47.4
Normal-Intermediate	8.2	13.8	16.7	17.0	15.8	10.8	12.5	10.4	6.9
Normal-Dull	5.4	9.6	9.8	21.0	13.3	7.8	9.1	9.1	4.3
Normal-Coarse Pure Vitrinite	19.5	14.8	11.4	10.3	10.9	9.6	12.2	9.3	21.3
Normal-Semifusinite-Isolated or >50µm	2.8	2.4	1.6	5.8	6.0	0.9	2.4	2.2	2.5
Normal-Fusinite-Isolated or >50µm	2.3	2.2	2.3	2.2	3.1	1.6	3.9	2.7	1.7
Ultrafine Coal <5µm	4.0	3.1	5.4	5.2	5.6	5.2	7.5	8.0	4.5
Pseudovitrinite	1.7	2.9	3.8	6.4	5.4	7.3	4.7	17.5	7.7
Oxidized Slight	0.2	0.2	0.2	0.6	0.6	0.3	0.5	0.5	0.5
Oxidized Moderate	0.3	0.1	0.0	0.2	0.2	0.0	0.0	0.0	0.1
Oxidized Severe	0.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Microbrecciation Fine	0.5	0.2	0.0	0.2	0.8	0.2	0.6	0.2	0.3
Microbrecciation Inter	0.2	0.3	0.4	0.4	0.0	0.3	0.5	0.2	0.2
Microbrecciation Coarse	0.2	0.0	0.2	0.0	0.0	0.2	0.0	0.0	0.0
Coarse Mineral Matter-Clay	1.8	0.6	0.9	0.5	0.4	0.9	0.6	2.4	0.5
Coarse Mineral Matter-Bone Coal	1.4	0.8	0.3	0.5	1.3	1.9	0.5	1.3	1.1
Coarse Mineral Mattter-Carbon Shale	0.3	0.0	0.0	0.2	0.0	0.1	0.2	0.0	0.1
Coarse Mineral Matter-Shale	0.0	1.3	0.3	0.0	0.2	0.8	0.3	0.5	0.1
Coarse Mineral Matter-Pyrite	1.3	1.9	1.3	0.2	0.6	0.9	0.2	0.8	0.5
Coarse Mineral Matter-Carbonates	0.7	0.1	0.1	0.2	0.0	0.9	0.1	0.2	0.0
Coarse Mineral Mattter-Quartz	0.3	0.2	0.0	0.2	0.2	0.0	0.1	0.0	0.3
Miscellaneous Features									
Contamination-Mill Scale	0.0	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.0
Contamination-rank	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.9	0.0
Contamination-Coke Dust	0.0	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.0
Cenosheres	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

APPENDIX 12
Feed Coal
Coke Petrography
FSI Button

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Binder Phase Carbon									
Isotropic	71.7	0.6	1.5	0.3	0.0				
Incipient	1.5	9.5	10.3	0.5	0.7				
Circular Anisotropy-Fine	0.0	49.2	52.4	19.6	2.1		0.1		
Circular Anisotropy-Interm	0.1	10.4	12.1	34.2	17.4	0.5	0.1		
Circular Anisotropy-Coarse		1.0	1.0	9.9	37.1	7.3	0.4	1.1	
Lentcular Anisotrop-Fine		0.1	0.9	1.3	13.4	40.3	36.7	19.6	
Lenticular Anisotrop-Interm		0.1	0.2	0.9	0.3	22.6	30.7	41.9	1.9
Lenticular Anisotrop-Coarse				0.4	0.1	5.1	2.2	10.0	7.7
Ribbon Anisotrop-Fine				0.1	0.1	1.7		5.5	58.8
Ribbon Anisotrop-Intermediate						0.5			9.0
Ribbon Anisotrop-Coarse									0.2
Total Binder Phase	73.3	70.9	78.4	67.2	71.2	78.0	70.2	78.1	77.6
Filler Phase Carbon									
Organic Inerts <50µm	8.8	13.5	9.0	19.8	15.2	10.0	17.8	10.5	10.0
Organic Inerts ≥50µm	3.8	5.3	5.8	6.0	4.0	4.0	8.4	4.9	4.1
Inorganic Inerts-Fine	5.6	4.5	3.4	4.0	5.2	2.8	2.7	3.3	4.9
Inorganic Inerts-Coarse	5.6	3.4	2.0	1.8	2.6	4.0	0.6	2.1	1.7
Misc. Org. Inerts (Oxid.)	0.0	0.1	0.0	0.4	1.2	0.0	0.0	0.2	0.5
Pyrite	2.9	2.3	1.4	0.8	0.6	1.2	0.3	0.4	1.2
Total Filler Phase	26.7	29.1	21.6	32.8	28.8	22.0	29.8	21.4	22.4
Pyrolytic Carbon	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.4	0.0
Non-Coal Related MM	0.0	0.0	0.0	0.0	0.0		0.0	0.1	0.0
Binder Reflectance-Estimated	0.6	0.90	0.90	0.99	1.02	1.24	1.25	1.30	1.50
Feed Coal Reflectance	0.53	0.77	0.83	0.98	1.02	1.08	1.23	1.36	1.53
Anisotropic Index	0.02	2.03	2.05	2.94	3.87	5.41	5.49	5.99	7.98

APPENDIX 13
NMP-Soluble Extract
Coke Petrography
FSI Button

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Binder Phase Carbon									
Isotropic	71.7	4.1		1.8	0.9			0.6	
Incipient	27.9	36.4	30.5	0.2	2.8		0.9		
Circular Anisotropy-Fine	0.4	52.6	59.8	7.2	10.6	3.9	1.9		
Circular Anisotropy-Interm		5.5	6.2	31.6	26.1	9.6	4.2		
Circular Anisotropy-Coarse		1.2	1.3	42.2	30.2	19.1	11.8	1.4	
Lentcular Anisotrop-Fine		0.2	1.4	14.4	25.5	40.6	19.1	4.8	2.9
Lenticular Anisotrop-Interm			0.8	0.8	3.9	10.3	39.6	31.6	10.2
Lenticular Anisotrop-Coarse				0.8		4.2	14.8	36.4	25.7
Ribbon Anisotrop-Fine				1.0		12.3	7.7	24.8	43.6
Ribbon Anisotrop-Intermediate								0.4	17.6
Ribbon Anisotrop-Coarse									
Total Binder Phase	100	100	100	100	100	100	100	100	100
Filler Phase Carbon									
Organic Inerts <50µm									
Organic Inerts ≥50µm									
Inorganic Inerts-Fine									
Inorganic Inerts-Coarse									
Misc. Org. Inerts (Oxid.)									
Pyrite									
Total Filler Phase	0	0	0	0	0	0	0	0	0
Oxidation Themal?	?	8.4	7.5	?	?	8.6	9.6	?	?
Non-Coal Related MM									
Binder Reflectance-Estimated	0.89	0.86	0.89	1.07	1.07	1.21	1.26	1.38	1.46
Feed Coal Reflectance	0.83	0.77	0.83	0.98	1.02	1.08	1.23	1.36	1.53
Anisotropic Index	1.86	1.64	1.86	3.69	3.74	5.06	5.63	6.76	7.63

APPENDIX 14
Feed Coal
Maceral Analysis
Weight Percent

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell/ WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Mean Max Vitrinite Reflectance R_o, %	0.53	0.77	0.83	0.98	1.02	1.08	1.23	1.36	1.53
Maceral Analysis									
Total Vitrinite Group	64.3	66.1	68.7	49.8	59.7	67.5	66.4	70.9	74.1
Total Liptinite Group	6.4	6.0	5.7	11.8	5.6	4.2	5.6	2.5	1.0
Semifusinite	4.7	3.8	5.8	6.7	7.5	2.3	2.8	5.5	5.4
Total Micrinite	8.9	9.7	7.7	20.5	11.5	9.8	14.6	8.6	9.5
Fusinite	3.5	4.1	4.2	4.8	6.4	1.8	5.1	3.2	2.5
Mineral Matter									
Pyrite	3.9	5.0	2.9	1.8	2.1	3.2	1.1	2.2	3.3
Clay	3.3	1.9	2.7	2.8	3.7	5.7	2.9	4.1	3.4
Carbonates	0.0	0.0	0.0	0.2	0.0	2.0	0.4	0.0	0.2
Calcite	0.7	0.4	0.0	0.0	0.0	2.2	0.2	0.4	0.0
Quartz	0.9	1.5	1.9	0.6	1.3	0.6	0.2	0.0	0.4
Shale	0.4	1.1	0.4	0.6	0.4	0.7	0.6	0.0	0.0
Bone	3.0	0.4	0.0	0.4	1.7	0.0	0.2	2.6	0.2
Rust	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total All Groups	100	100	100	100	100	100	100	100	100

APPENDIX 15
Insoluble Residue
Maceral Analysis
Weight Percent

Coal Name	Ky #9	Shoemaker	Bailey	Mingo Logan	Marfork	Kingwood	Litwar	Jewell-Tiller	Poca #12
Seam	No. 9	Pittsburgh	Pittsburgh		70/30 Pow/CG	Kittanning	Litwar	Tiller	McDowell/ WV
County/State	KY	Marshall/WV	PA	WV	WV	Preston/WV	WV	Buch./VA	WV
Mean Max Vitrinite Reflectance R_o, %	0.53	0.77	0.83	0.98	1.02	1.08	1.23	1.36	1.53
Maceral Analysis									
Total Vitrinite Group	59.9	52.6	60.1	43.5	33.6	41.9	55.7	65.7	74.5
Total Liptinite Group	7.5	5.8	6.4	11.0	11.5	8.7	3.7	2.5	1.0
Semifusinite	3.5	5.7	4.5	8.6	11.8	5.8	7.1	4.7	6.8
Total Micrinite	9.8	13.7	10.8	22.2	25.3	9.6	19.2	11.0	8.7
Fusinite	2.8	4.8	5.0	5.8	4.2	3.8	4.0	5.1	3.8
Insol Residue Refle %	0.57	0.66	0.73	0.86	0.96	1.00	1.14	1.38	1.56
Mineral Matter									
Pyrite	5.9	9.1	4.2	2.1	2.4	6.7	1.8	1.4	1.8
Clay	7.7	4.0	4.4	4.1	7.7	14.7	5.3	4.3	1.9
Carbonates	0.0	0.0	0.0	0.0	0.2	0.0	0.0	0.0	0.0
Calcite	0.0	0.4	0.4	0.4	0.2	0.3	0.4	0.7	0.0
Quartz	1.1	0.5	0.4	0.6	0.5	1.2	0.4	0.9	0.2
Shale	0.2	1.9	1.3	0.4	0.2	1.5	0.9	0.7	0.4
Bone	1.6	1.4	2.6	1.5	2.3	5.9	1.5	3.0	1.0
Rust	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total All Groups	100	100	100	100	100	100	100	100	100