

RETORTING POTENTIAL OF LIGNITE OVERBURDEN FROM CLAY MINING  
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**Keywords:** Lignite, Pyrolysis, Oil Yields

Lignite is found in the Eocene formations in the Gulf Coast Province from Texas north to Kentucky and east to Georgia. Commercial development of the lignite as an energy resource is currently confined to Texas and Louisiana where mine-mouth power plants are large consumers of lignite. The lignite in other Gulf Coast Province states is not mined as a product at the present time.

Lignite is present in the eight-county Jackson Purchase region of Kentucky at the northern end of the Mississippi embayment. The Claiborne Formation, the primary lignite-bearing formation in Kentucky, is dominated by clay deposits with associated lignites and sands. The lignite and clay bodies were deposited as narrow elongate deposits, which resulted from the filling of oxbow lakes over periods of 500 to 1500 years. The axis of the fluvial depositional system migrated east then back to the west over the course of the Claiborne deposition.

The lignite sample was obtained from the Eocene Claiborne Formation, Milburn 7½ minute quadrangle, Carlisle County, Kentucky. Lignite is removed as overburden in the mining of ball clay from the Eocene of Kentucky's Jackson Purchase region, but is not currently mined as an energy resource but rather is a waste product. About 160 kt of clay was mined in 1991 (Kentucky Department of Mines and Minerals) with an estimated 40 kt of lignite moved in the process. The high moisture, low heating value lignite has not been competitive as an energy resource due to its proximity to the higher quality bituminous coals in the nearby Illinois Basin and, more recently, to the import of Powder River Basin coals into the lower Ohio River Valley. In this study, we examine potential uses of the lignite which could provide the opportunity to develop it as an added resource along with the clay.

## Results and Discussion

### *Petrography of the Lignite*

The petrography of the lignite is rather typical of other lignites from the region in having a high percentage of detrital macerals: humodetrinite and liptodetrinite (Table 1). Petrographic analysis of those macerals is difficult owing to their fine (generally less than 10 µm) size and consequent difficulty of resolution in either white-light or blue-light microscopy. Blue-light excitation is an aid in resolving some liptinite macerals in complex mixes. Liptinite macerals

in this and other Kentucky lignite samples evolve mobile constituents in the form of the maceral fluorinite. The reflectance of lignite is typically measured on ulminite. The low percentage of ulminite in this lignite limited the number of measurements. Ulminite reflectance was determined to be 0.21%, consistent with previous samples from the same mine. The petrology of the Eocene lignites in the Jackson Purchase region was discussed by Hower et al. (1990).

#### *Pyrolysis of the Lignite Sample*

The lignite sample was retorted in N<sub>2</sub>-swept fixed bed experiments. The production of oil from a waste overburden, such as the present lignite, would make the economics of clay mining in this region even more attractive. Accordingly, the lignite sample was first ground to a grain size of 18 x 20 mesh and fed into a retort to establish the lignite's oil yield potential. The N<sub>2</sub>-swept fixed bed experiments were performed in a 1.5 inch stainless steel reactor. Oil vapors and sweep gas passed through a 100 micron filter at the base of the reactor. Heating rate was 4° C/minute with a maximum bed temperature of 550 °C. The reactor conditions are discussed in detail by Rubel and Coburn (1981). The objective of this part of the research was to obtain the oil yields of the lignite sample. Results of the retorting experiments indicate that 11.7 wt % oil may be obtained during pyrolysis.

The oils derived from the lignite are very waxy. Large amounts of water were also captured during the retorting of the lignite sample. Because most of the water was released before the sample was heated to above 150 °C it may be concluded that only small amounts of structural water were released by clay minerals which occur as ash components within the lignite sample.

The processed lignite sample was analyzed for carbon, hydrogen, nitrogen, and sulfur using automated LECO elemental analyzers. Ultimate and proximate analyses of the retorted lignite sample are illustrated in Table 2. The majority of the moisture was released during pyrolysis. After pyrolysis the retorted lignite sample was found to have very high fixed carbon values [Table 2].

#### *Heating Stage Microscopic Examination of the Lignite Sample.*

The heating stage microscope (HSM) apparatus utilized in this study consists of a Zeiss Universal polarized light microscope, a heating stage, and temperature and gas flow controllers. A polished shale sample was positioned in the heating chamber and could be observed at any time using a polarized light microscope. The sample chamber consists of three quartz plates which help to channel the preheated N<sub>2</sub>-gas flow above and below the sample for optimum temperature control. The hot stage was equipped with a monitor and video camera for real time observations. Most importantly, the HSM -apparatus allowed the *in situ* observation of the pyrolysis process of the lignite sample.

Because the HSM-study permitted the use of larger lignite particles than those used in the reactor, the retorted parts were big enough to preserve the spatial relationships between the charred macerals, mineral matter and macro-pores that were generated during oil evolution.

Observations of the process induced macroporosity in the retorted (spent) lignite sample were made possible using a scanning electron microscope ( SEM; Hitachi 2700-S), which allowed a magnification of the sample by  $\times 10^4$  times. Structural changes in the retorted lignite sample are documented in Figure 1 and Figure 2. The SEM results indicate that volatilization of macerals causes a complex network of macropores to develop which typically have the shape and size of the macerals in the raw lignite. A multitude of cracks also formed throughout the lignite sample (Figure 1), which probably aid in the escape of the oil vapors. Prior to pyrolysis, cracks were not observed in the lignite sample (Figure 1).

Not all of the macerals contribute to the oil phase when heated in the retort. The pyrolysis residue was primarily composed of uniformly reflecting isotropic semi-coke and unreacted inertinite macerals, primarily sclerotinite. Semi-coke with the texture of the huminite maceral textinite was observed. Other huminite macerals, and perhaps a portion of the liptinite component, are preserved as a porous semi-coke with little preservation of the original morphology. Anisotropic semi-coke, perhaps as a retrograde product of evolved liquids, was a rare component. Pyrrhotite was observed, but silicates are the more common mineral form in the residue.

#### **Summary and Conclusions**

Lignite is presently removed as overburden in the mining of ball clay in the eight-county Jackson Purchase region of Kentucky rather than mined as an energy resource. Although this is mainly due to the fact that higher quality bituminous coals are mined locally, the removal of the lignite causes both environmental and economical concerns. This led to the objective of this study to examine potential uses of the lignite to be utilized as an added resource along with the clay. Liquid product recovery after pyrolysis of the lignite indicates that this waste product contains 11.7 wt % of oil. Furthermore, a carbon-rich solid remains after pyrolysis. Upon retorting the lignite was observed to have formed an intricate network consisting of variously sized pores and charred carbonaceous material. Heating stage microscope (HSM) observations helped to establish the spatial relationships between process induced pores and char. In addition, the HSM-experiments allowed the precise determination of the boiling temperature when constituents in the macerals start to vaporize. The development of a porous structure in the carbon-rich pyrolysis residue of the lignite under investigation may indicate the lignite's potential as source material for the production of adsorbent carbons. Utilization of the lignite in the production of liquid fuels, and possibly as carbon precursor, may help in the regulation of a waste material.

**Acknowledgements** The Authors would like to thank G. Thomas, M. Moore, and M. Spears for analytical and technical support.

## References

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**Table 1** Maceral Composition of the Lignite

Maceral	Sample 91999 (W. KY)
Textinite	0.7
Ulminite	3.5
Humodetrinite	35.1
Gelinite	0.4
Corpohuminite	5.5
Fusinite	3.3
Semifusinite	0.3
Sclerotinite	1.1
Inertodetrinite	0
Exinite	6.3
Resinite	2.3
Suberinite	1.6
Liptodetrinite	39.1
Alginite	0.8

**Table 2** Ultimate and Proximate Analyses of Raw and Pyrolyzed Lignite Sample

	91999	91999	91999	91999p	91999p	91999p
	as rec.	dry	daf	as rec.	dry	daf
Moisture	10.97					
Ash	19.11	21.16		3.21		
Volatile Matter	51.30	57.62	73.36	35.53	36.71	
Fixed Carbon	18.60	20.89	26.64	21.64	22.36	35.33
Carbon	50.00	56.16	71.50	39.60	40.91	64.67
Hydrogen	6.03	5.39	6.86	56.11	57.97	91.59
Nitrogen	0.96	1.08	1.37	2.15	1.85	2.92
Oxygen	11.85	14.70	18.73	1.07	1.11	1.75
Sulfur	1.08	1.21	1.54	0.52	0.90	1.43
Heating value (Btu/lb)	8690			1.41	1.46	2.31
Heating value (MJ/kg)	20.26					

91999 = lignite raw sample

91999p = pyrolyzed lignite sample



**Figure 1** SEM-microphotograph of a polished section of the lignite sample. The insert presents the raw lignite sample before the heating stage microscope (HSM) experiment. The polished lignite surface shows only minimal signs of cracks or pores. The main photo shows the surface of the lignite sample after pyrolysis in the HSM. The surface is characterized by abundant cracks of various sizes and large amounts of elongated macropores.



**Figure 2** Magnification of lignite sample shown in SEM-microphotograph of Figure 1. Macropores develop during pyrolysis. The spatial relationship among macropores, and that of the macrostructure stabilizing char and mineral matter are preserved during the HSM experiments.