Primary and Secondary Pores of Kerogen and Bitumen By Reflected Light and Scanning Electron Microscopy

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ABSTRACT

Macerals, inclusive of kerogen and bitumen, are recognized and distinguished in reflected white and fluorescent light at magnifications of 200-625x using 20-50x oil immersion objectives and I 0-12.5x oculars. Recent applications of scanning electron microscopy (SEM) to shale resource plays at magnifications of <500-80,000x have reported nanoporosity in organic matter, with limited interpretations of organic matter type. The objectives of this study are to evaluate the extent to which macerals may be recognized in SEM, which macerals contain primary and secondary porosity, and at what thermal maturity levels secondary porosity develops.

Since coals are organic rich with a better chance of identifying maceral types than in shales, humic and sapropelic (boghead and cannel) coal samples ranging in rank from high volatile bituminous to semianthracite were examined in reflected white light. In order to better view the organic matter microporosity by maceral type, coal samples were also viewed on ion-milled samples in SEM. The two basic signals utilized in SEM are secondary electron and backscattered electron microscopy. Secondary electron (SE) microscopy images high resolution sample topography. Backscattered electron (BSE) microscopy reveals organic matter by atomic number contrast.

SEM analysis in BSE mode is the best method to observe nanoporosity in organic matter; however, organic matter types are not routinely distinguishable at low accelerating voltages of 1-2 kV. Initial studies of coal samples at standard low voltage SEM settings resulted in all macerals having the same gray level with no contrast. Manipulation of the accelerating voltage to I 0 kV in BSE mode of a high volatile bituminous humic coal durain lithotype sample revealed a contrast between maceral groups (vitrinite, inertinite, liptinite). Vitrinite maceral subgroups telovitrinite and detrovitrinite are distinguished based on occurrence as bands or groundmass, respectively. Sporinite and cutinite are distinguished based on dark gray level and shape. Semifusinite and fusinite are recognized by bogen structure but not distinguishable separately. Lacking bogen structure, macrinite/semimacrinite are weakly distinguishable from vitrinite. Fusinite and semifusinite macerals in shale may be recognized as inertinite by bogen structure but are otherwise indistinguishable from vitrinite.

A digital camera attached to a reflecting light microscope is more sensitive than the human eye. Manipulation of the exposure and contrast of the digital camera revealed pits (initially interpreted to be microporosity) in vitrinite and some inertinite macerals at 500x magnification. However, examination of the same samples prepared for SEM by ion milling did not contain the same microporosity. Coal in general, and vitrinite in particular, is brittle. In addition to incorporating scratches on the surface during pellet polishing, pits are also introduced which may be erroneously interpreted to be pores. Even though porosity is revealed at high magnification in BSE, too high of a magnification (> 15,000x) prohibits recognition of maceral types. The best approach is to examine samples at a lower magnification in BSE (e.g., 650x), identifY the maceral type, and then go to higher magnification to look for porosity.

Microporosity observed in macerals at low rank (high volatile C bituminous) are determined to be primary. Primary macroporosity occurs as woody cell lumens in semifusinite and fusinite macerals observed in both reflected light and SEM. Slitted structure in pseudovitrinite is an early oxidation product and is considered to be primary porosity since it is not related to coalification. During low light exposures, some inertinite clasts are observed to contain slitted structures that are considered primary porosity from oxidation. Secondary nanoporosity, observed in SEM, develops in a post-oil solid bitumen network in shale beginning at the peak of the oil window. Additional observations on the development of secondary nanoporosity by maceral type with rank will be discussed.

Knowledge of organic matter porosity distribution by maceral type and development by thermal maturity provides insight for coalbed methane and shale gas and oil production potential.