Bridging the Gap Between Optical and Electron Microscopy: The Importance of Correlative Microscopy for Understanding Dispersed Organic Matter

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ABSTRACT

In recent years, there has been an increase in the use of scanning electron microscopy (SEM) to improve our understanding of both conventional and continuous resources by characterizing dispersed organic matter (OM) in shales and other geologic materials at the submicron scale. Traditional organic petrographic identification of OM uses optical microscopy, where reflectance (R, %), form, relief, and fluorescence can be used to discern OM types (e.g., vitrinite, solid bitumen, inertinite, alginite, etc.). Unfortunately, SEM's do not give petrographers the same information as optical microscopy. In electron microscopy, the information garnered can provide details as to relief(secondary electron image, SE) of the sample surface and contrasts in atomic weight (backscatter electron image, BSE) of the sample matrix. Due to the low atomic weight of carbon, all OM appears black in SEM regardless of differences in thermal maturity, which are readily observed via optical microscopy (e.g., by Ro or fluorescence). The lack of contrasting properties between OM types with SEM also can lead to misidentification or to the idea that all OM (i.e., kerogen and bitumen) is the same.

In order to improve the accuracy of identifying dispersed OM, this study uses both optical and SEM techniques by taking images in the same field of view (500x) under white light, blue light, SE, and BSE conditions. Samples (n=7) of varying thermal maturities from immature to over mature (Green River Fm., Bakken Fm., Tuscaloosa Group, Eagle Ford Fm., Barnett Fm., Haynesville Fm., Woodford Shale) were mounted in a thermoplastic resin and polished following ASTM D2797. Using an automated stage and the mosaic function of the FOSSIL software by Hilgers Technisches Buero, we captured sample maps of the entire pellet surface (100x) as well as smaller-scale regional maps (500x) to aid in navigation when using both microscopes. Under the optical microscope (500x, oil immersion) fields of view that exhibited multiple OM types were captured using three conditions: 1) a gray-scale image showing measured reflectance of OM types under white light conditions; 2) a color image under white light conditions; and 3) a color image under blue light fluorescence. After cleaning the pellet surface and coating it with carbon, the same field of view was captured using a Hitachi SU-5000 field emission SEM at 500x (5kV, spot intensity 10, working distance 5 mm) under SE and BSE conditions.

The image sets demonstrate the importance of correlative microscopy by showing how easily OM can be misidentified or misinterpreted. For example, the Bakken Fm. samples under both SE and BSE modes show a prominent network of void-filling OM, which was interpreted as solid bitumen. Even though the optical light images agree with the solid bitumen identification, what could not be differentiated via SEM was that there were two forms of solid bitumen found in the organic network: 1) low-reflecting (0.32%), void-filling, narrow stringers of solid bitumen; and 2) higher-reflecting (0.59%), larger isolated solid bitumen. If this difference in OM is not distinguished, interpretations and data generated on properties such as organic porosity could give misleading results when trying to determine how or where pores have developed.

The image sets collected in this work will be added the USGS Organic Petrology Photomicrograph Atlas to aid petrographers in the identification and interpretation of OM in shales and other geologic materials. Further work will focus on differences related to sample preparation, as organic petrographers typically use crushed, epoxy/thermoplastic mounted samples (viewed in random orientations) and SEM petrographers use ion-milled cored chips (viewed either parallel or perpendicular to bedding).