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Microstructural Analysis of the Transformation of Organic Matter during Artificial Thermal Maturation of the Upper Cretaceous Boquillas (Eagle Ford) Formation, Texas, USA

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A dynamic heating experiment using an field emission-environmental scanning electron microscope (FE-ESEM) equipped with a programmable heating stage was designed following the work of Dahl et al. [1] to observe microstructure changes during artificial thermal maturation of an organic-rich shale (petroleum source rock). This experiment differs from prior studies in that this is the first study designed to compare the transformation of a variety of organic matter types identified by optical organic petrology from the same area of the same sample, before and after heating.

An outcrop sample of the Boquillas Formation (Eagle Ford Formation equivalent) was collected near Comstock, Texas. The sample is a black, organic-rich (5-7 wt% total organic carbon), laminated mudstone, composed of 60 wt% calcite, 26 wt% quartz, and 10 wt% clay (XRD). The outcrop sample is thermally immature with a mean vitrinite reflectance of 0.50 %Ro and 422 ^{o}C Tmax. The high hydrogen index of 603 mg HC/g TOC and high S2 of 35.5 mg HC/g rock indicates that the sample is a potential source rock capable of generating petroleum.

Samples were prepared from 25 mm (one inch) diameter plugs cut perpendicular to bedding, and polished to a smooth surface using a large area Ar-ion mill. The organic composition and vitrinite reflectance were analyzed using an incident light microscope equipped with a photometer following standardized petrographic procedures (ASTM Standard Test Method D 7708). Four areas of interest (AOI's) were selected for SEM examination. White and ultraviolet light photomicrograph, and FE-SEM image mosaics were prepared to document the pre- and post-heating microstructure of the organic matter. Two 3 mm x 1.4 mm microplugs were cut over the AOI's to accommodate the 5 mm diameter ceramic crucible of the heating stage FE-ESEM.

The sample was successfully heated as programmed to 500 ^{o}C at rate of 1 ^{o}C/min. The FE-ESEM chamber vapor pressure was held constant at 2.0 Torr (266 Pa). Static and video secondary electron SEM images were captured at 300x magnification using a heat compatible gaseous secondary electron (GSE) detector at 30 kV accelerating voltage. Unfortunately, the images acquired were unsuitable to detect any noticeable changes during heating up to 2500x magnification.

Several types of organic matter were identified, including: 1) structured particles of vitrinite, inertinite (semifusinite), liptinite, and vitrinite-mineral aggregates; 2) diffuse amorphous organic matter (AOM); and 3) solid bitumen. The liptinite occurred as fluorescent filamentous lamalginite and *Tasmanites*-like alginite. The bitumen was weakly fluorescent, filling preserved voids in foraminifera chambers and present as discontinuous lenses.

Comparisons of the pre- and post-heating SEM image mosaics revealed no detectable microsctructural changes within the various organic matter types except for the alginite macerals (Figure 1). The post-heating SEM images revealed that portions of the alginites were no longer present, leaving behind void spaces within the mineral matrix (Figure 1B). Presumably the evacuated organic matter represents portions of the original kerogen that was converted to oil during heating. The post-heating SEM images also revealed variations in backscatter electron (BSE) intensity within the alginites that was not observed in the pre-heating FE-SEM images. The variation in BSE intensity may reflect subtle compositional variation as a result of nonuniform chemical and structural changes in the algal kerogen during thermal maturation.

The incomplete and variable conversion of the oil-prone organic matter (liptinite and AOM) probably reflect variations in the thermal reaction kinetics between the two types of organic matter. The limited conversion of the alginite may also reflect the relatively short duration (5 hrs) of the heating experiment and limited availability of water in the gaseous FE-ESEM environment.

The remnant voids (pores) between the converted alginite macerals and mineral matrix of the heated sample are not typical of the characteristic organic matter pores observed in SEM images from subsurface samples of the of age-equivalent Eagle Ford shale reservoirs in the Maverick basin (or other unconventional shale reservoirs) such as compiled by Camp et al. [2]. Such slot-like pores are unlikely to be preserved at depth due to overburden pressure.

References:

[1] Dahl, J. *et al*, Making movies of oil generation (abs.), Unconventional Resources Technology Conference Paper # 2152075, (2015), 5 p.
[2] Camp, W.K., E. Diaz, and B. Wawak (eds.), Electron microscopy of shale hydrocarbon reservoirs: American Association of Petroleum Geologists Memoir 102, 260 p.

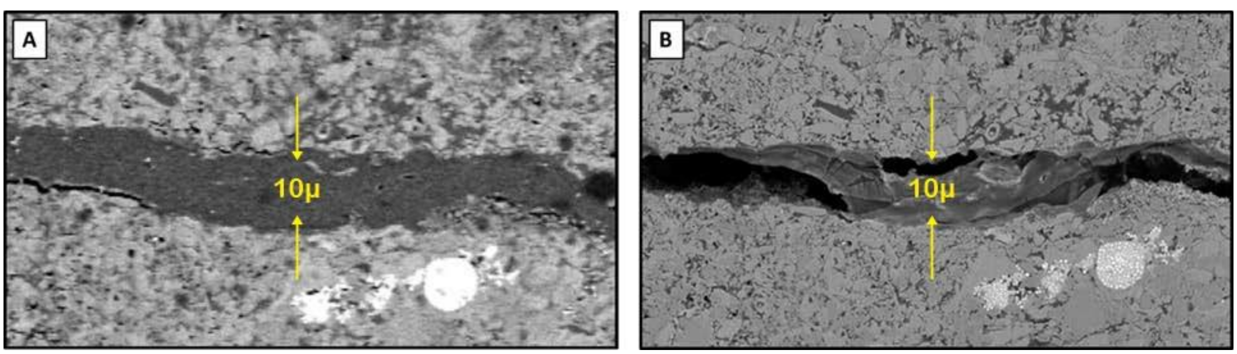

Figure 1. Backscattered electron FE-SEM images comparing filamentous algal organic maceral (dark gray) before (A) and after (B) heating to 500 ^{o}C in the heating stage FE-ESEM. The void space (black) partially surrounding the alginite in the post-heating image (B) presumably represents regions of the original kerogen that was converted to petroleum. Note the lack of apparent change in the other smaller particulate and amorphous organic matter (dark gray) in the adjacent mineral matrix. Width of the alginite maceral is approximately 10 μm.

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