

NanoMin; Sub Micrometer Mineral Mapping to Identify Diagenetic and Depositional History in Shale

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ABSTRACT

Spatially resolved chemistry, phase (mineralogical) identification, and quantification are essential to many fields of scientific enquiry, particularly at the critical nanoscale at which many natural environmental processes occur. Sub micron scale mineral growths within shale can determine the viability of a hydrocarbon reservoir by occluding porosity and influencing the composition of hydrocarbons generated from primary organic particles by catalytic reactions. Studies of coarse-grained materials routinely depend on the determination of minerals and their distribution using simple optical petrography or spatially resolved electron probe data such as QEMSCAN (Quantitative Evaluation of Minerals by SCANNing electron microscopy) or MLA (Mineral Liberation Analyser) systems. However, spatially resolved mineral identification has long proven a challenge in fine-grained materials, whether engineered, geological or biological in origin, where individual mineral grains are commonly smaller than the resolving power or analytical volumes of the techniques used. This broad class of material is most commonly studied using bulk characterisation approaches such as X-ray diffractometry and FTIR spectroscopy of powders. The loss of spatial and contextual information leads to inferences of importance based simply on abundance, which is of particular concern with these techniques because of their poor detection limits.

Here we apply a new system called NanoMin capable of sub J..lm quantitative mineral mapping based on FEG-SEM-EDS (Field Emission Gun Scanning Electron Microscope - Energy Dispersive X-ray spectrometry). At present, the most advanced commercial SEM-based mineral mapping systems (QEMSCAN and MLA) are fundamentally limited in their characterisation of mixed phase X-ray spectra. Mixed spectra are obtained when multiple phases (minerals) are presented in the electron-beam excitation volume. QEMSCAN and MLA can only assign a single mineral to each 5 J..lm pixel area. NanoMin quantitatively resolves multiple minerals within the excitation volume of every single pixel allowing for multiple phase determinations. The spectral deconvolution engine and standard mineral spectra database that form part of NanoMin allow each analysed micro-spot to be decomposed into up to three component minerals, an approach which explicitly acknowledges the common presence of mixed mineralogies in each analysed volume/spot. This capability opens up analysis of complex fine-grained materials in which quantitative mineral mapping has not previously been possible, resulting in greater accuracy in mineral identification, spatial understanding of trace minerals, quantification and analysis of spatial relationships between phases.

NanoMin data are compared between two late Permian aged shale reservoirs in South Australia; the lacustrine REM interval of the Cooper Basin and adjacent marine Stuart Range Formation in the Arckaringa Basin. Both show systematic distributions of early carbonate cements. The REM lacustrine depositional environment resulted in early diagenetic lowS, high Fe cements (siderite) that were preferentially formed in coarser grained portions of laminae. These cements parse the reservoir by restricting migrating hydrocarbons to finer grain size intervals. By contrast, S availability in the marine Stuart Range Fm. led to sequestration of Fe in pyrite prohibiting the formation of siderite. Excess S resulted in sulfurization reactions that preserved lipid-rich type II organic matter. Pyritic intervals dominated by type II kerogen alternated with

Mn-carbonate cemented intervals dominated by type III (refractory terrestrial) OM in varved intervals with the distribution controlled by oscillations in basinal redox conditions. The dominance of one cycle over the other influences hydrocarbon potential as well as brittleness and reservoir compartmentalization where Mn-carbonate intervals increase. While Mn-carbonate and siderite were present in trace amounts in many of the samples analysed by powder X-ray diffraction, the spatial data from the in situ technique provided the environmental significance and the ability to better understand basinal trends in source, reservoir, and rock properties.