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Image-based microstructural analysis: A BIB-SEM study on Upper Visean shales from the Dniepr-Donets Basin (Ukraine)

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Great improvement in the understanding of shale microstructures arose from the implementation of broad ion beam – scanning electron microscopy (BIB-SEM) into petroleum geosciences. BIB-SEM allows the investigation of relatively large, undisturbed specimen surfaces, for which high magnifications (over 100kx) can be achieved. Nevertheless, a crucial part in BIB-SEM studies is related to image processing, as single high-resolution images need to be combined into a mosaic, in order to obtain a large area map. This study aims on the quantification of organic matter (OM)- and mineral-hosted porosity, the assessment of pore geometry factors, as well as the relative importance of drying cracks and other core alteration phenomena as artificial contributors to the apparent total porosity. Furthermore, the validity of a total organic carbon (TOC) estimation based on grey scale raster data was tested at multiple scales. To do so, BIB-SEM maps at 312x, 1250x, 10kx and 20kx magnifications were acquired for 17 Upper Visean black shale samples from the Ukrainian Dniepr-Donets Basin, covering a broad maturity interval of 0.65 – 2.7 %Rr (vitrinite reflectance). OM-hosted porosity was obtained by combining 10kx backscattered electron (BSE) and 20kx secondary electron (SE) maps, allowing for the automated segmentation of pore structures within the organic matter, which shows a similar grey scale as pores in BSE images. In general, total porosity in the SEM-visible scale ($\sim$30 nm of equivalent diameter at 20kx magnification) is limited ($\leq$ 2 %), but it is likely that a considerable fraction of the total porosity is too small to be visualized by this method. Clay-rich samples at advanced maturity (1.0 – 2.7 %Rr) mostly show true SEM-visible porosity values < 0.2 %. A significant number of SEM-visible pores (up to 50 %) were classified as artefacts, mainly drying cracks and pores within gypsum, a product of core alteration. A good correlation was found for the amount of total porosity and OM-hosted porosity, and OM-hosted porosity makes up a significant proportion (10-60 %) of total visible porosity in most samples. However, the ratio of OM-hosted porosity versus total visible porosity does not correlate with the total amount of OM-hosted porosity, arguing for multiple factors being responsible for the distribution of pore types, e.g. the increase of solid bitumen porosity in samples where large intergranular pores allow solid bitumen to accumulate. A correlation was furthermore found for circularity and aspect ratio of total visible pores versus OM-hosted pores, which suggests a compaction control rather than generally different pore shapes in organic matter and mineral matrix. A good TOC estimation could be achieved from grey scale mosaics at 1250x magnification (R2 $\sim$ 0.8), while a weak correlation was found at 10kx magnification (R2 $\sim$ 0.5). Hence, the representative area for a valid organic matter quantification and spatial statistics ranges at 1-2 mm2 for the investigated samples, which is considerably larger than proposed for mineral matrix porosity and mineralogy of a typical mudstone. The relative error is strongly related to size distribution and geometry of organic matter particles in the individual sample.