



Visualising the 3D Structure of Fine-Grained Estuarine Sediments; Preliminary Interpretations of a Novel Dataset Obtained via Volume Electron Microscopy

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Accurate measurement of the physical characteristics of sediment are critical to determining sediment transport behaviour and the stability of settled deposits. The properties (e.g. particle size, density, and settling velocity) of coarse-grained sediments ($> 63 \mu\text{m} \varphi$) can be easily characterised, hence their behaviour is relatively simple to predict and model. However, due to their small size and tendency to interact with their surrounding medium, the characteristics of fine sediments ($< 63 \mu\text{m} \varphi$) and their behaviour during transportation, deposition and consolidation is poorly understood.

Recent studies have used correlative microscopy, a multi-method technique combining scanning confocal laser microscopy (SCLM), conventional optical microscopy (COM), and transmission electron microscopy (TEM), to characterise fine sediments at both the gross ($> 1 \mu\text{m}$) and sub-micron scale (Droppo et al., 1996). Whilst this technique has proven insightful, the measurement of geometric properties (e.g. the shape of primary particles and their spatial arrangement) can only be achieved by three-dimensional (3D) analysis and the scale of observation for e.g. TEM does not overlap with those techniques used to characterise sediments at larger scales (100s to 1000s microns) (e.g. video analysis). Volume electron microscopy [or focused ion beam scanning electron microscopy (FIB-SEM)] provides 3D analysis at scales of 10s to 1000s microns and though widely used in cell biology, has not been used to observe sediment. FIB-SEM requires samples that are vacuum stable and a key challenge will be to capture fragile, hydrated sediment samples whilst preserving their structural integrity. The aims of this work are therefore: 1) to modify preparation techniques currently used in cell biology for the stabilization of sedimentary materials; 2) to acquire 3D datasets for both fragile suspended sediments (flocs) and consolidated bed sediments and 3) to interpret the 3D structure of these samples.

In order to reduce alteration of the samples caused by dehydration, samples were first 'fixed' in 2.5% glutaraldehyde/2% formaldehyde before embedding in Durcupan, a hydrophobic epoxy resin. Pore water was then replaced by exposing the sample to a series of solutions containing increasing concentrations of ethanol, and then anhydrous acetone. Samples were stained using heavy metals to improve contrast, particularly for organic content. Selected regions of the prepared sample were sequentially milled and imaged using a FEI Quanta 3D FIB-SEM either in backscattered or secondary electron imaging mode. Image processing was initially conducted using Amira 5.5, images were then exported into Drishti 2.0 in which pixel intensity thresholding allowed particle-matrix boundaries to be defined and 3D models generated.

Preliminary results demonstrate the complex 3-dimensional, non-fractal nature of aquatic sediments with samples composed of multiple 'bundles' of primary particles held loosely together by bridges composed of both organic and inorganic matter. There is minimal evidence to suggest that structure has been altered by the stabilization process and individual flocs and samples were observed bridging the gap between the submicron and gross scale. Volumetric microscopy offers potential to examine 3D structure of sediments in a range of environmental settings and provide valuable insight into their behaviour and transport.