

High resolution structural characterization of formulations using Cryo Focused Ion Beam Scanning Electron Microscopy (Cryo FIB-SEM) and Energy **Dispersive X-ray Spectroscopy (EDX)** Laidlaw, Fraser H. J.¹; Li, Ngai Ying Denise²; Moore, David J.^{1,2*}

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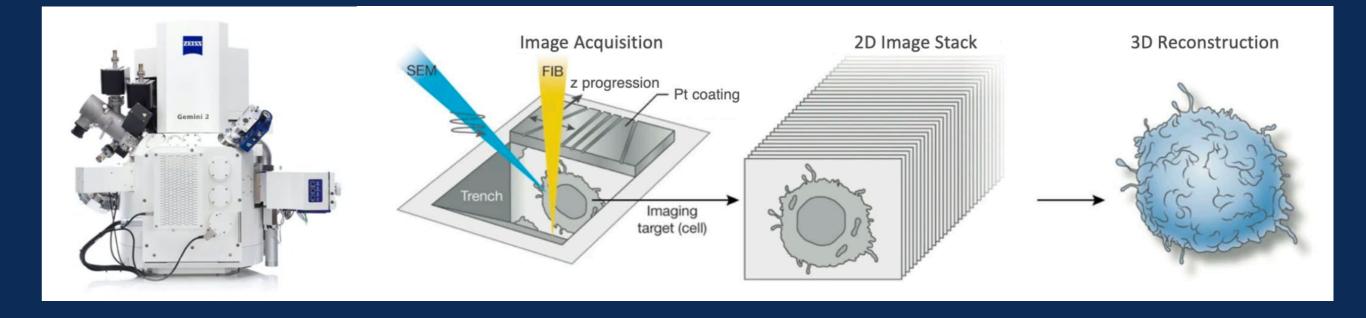


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Introduction

The microstructure of formulations determines a range of key performance properties including flow, texture and feel. Whether a formulation feels gritty, grainy or smooth is influenced by the size and shape of particles while properties such as flow, spreadability, and stability can be determined by particle-particle interactions.

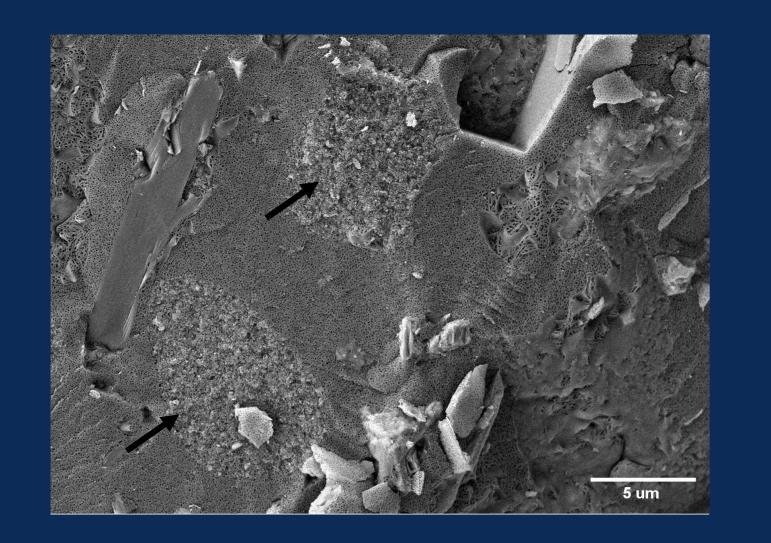
Cryo FIB SEM provides nanometre resolution images of semi-solid samples such as cosmetic formulations. 3D visualisation of formulations is accomplished by slicing and sectioning the samples.

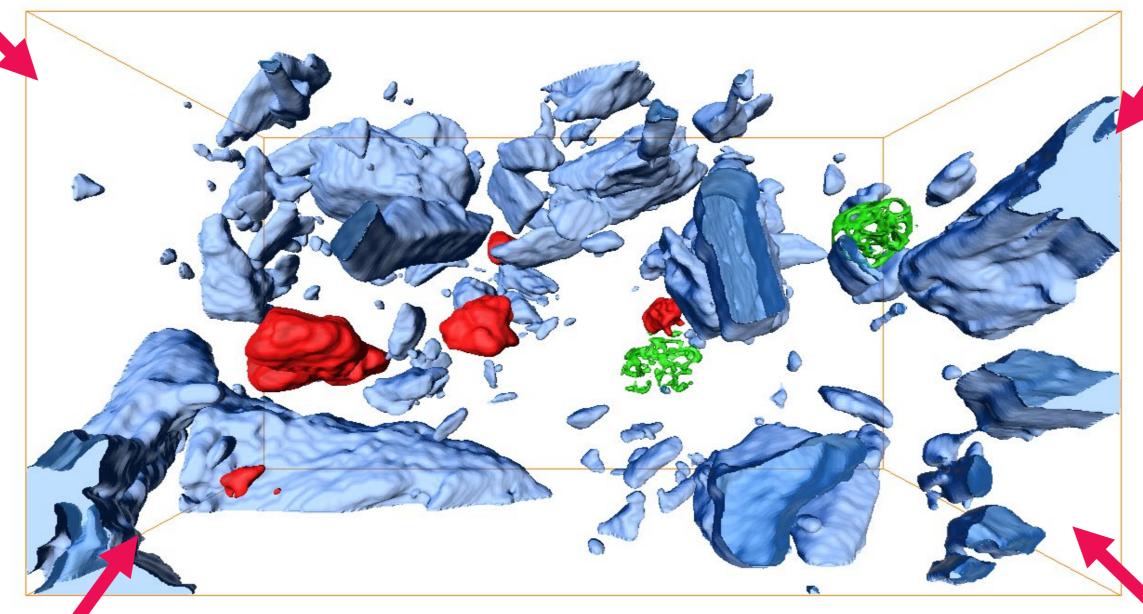


Results

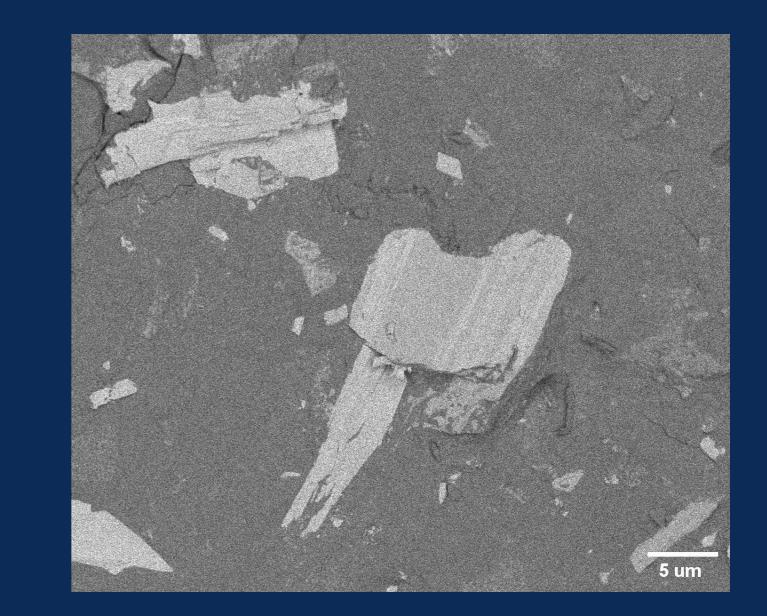
The different detectors on the SEM provide complementary information. By combining this information with slicing and imaging a detailed 3D visualisation of the formulation microstructure can be constructed complete with molecular identification: dicalcium phosphate dihydrate particles (blue), hydrated silica (red) and magnesium carbonate (green). The volume of the reconstructed volume is $17.2 \times 9.2 \times 9.8 \mu m$.

1. The secondary electron detector provides a highresolution image of the sample's surface; a gel network containing various particles and clusters of hydrated silica (arrow indicated).





2. The back scatter electron detector reveals differences in the average atomic weights; the large, crystalline particles being brighter.



3. Energy dispersive X-ray spectroscopy provides direct elemental identification at a given location within a formulation sample: different elements are then colour coded. Ca

P

4. The focused ion beam reveals the internal structure of the sample. A 3D visualisation of the formulation's microstructure is generated by stitching together a continuous series of 2D images.



34° Congress IFSCC Brazil | 14-17 October | Iguazu Falls **Biodiversity and Cosmetics: Reaching Sustainable Technology**



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