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Using Cryo FIB-SEM and Confocal Microscopy to Visualise the Microstructure of Skincare Formulations

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Introduction

- The texture or 'feel' of a cosmetic product is a key for consumer experience
- The 'feel' of a product can also be associated to the efficacy of a product
- Most products are sheared down to microscopic films upon application, which induces a change in the microstructure
- The aim of this study was to characterise the microstructure of a commercial product and track how this changes upon application

Figure 1: Image of a person rubbing in a skin cream.

Sample

- The product studied was a commercial SPF 30 sunscreen
- The formulation is an oil-in-water emulsion containing both organic and inorganic UV absorbers
- The initial microstructure was imaged using Cryo Focused Ion Beam Scanning Electron Microscope (Cryo FIB-SEM) and the evolution of microstructure was tracked using Confocal Microscopy
- Soft samples can be frozen to preserve the sample structure close to its natural state and allow it to withstand the high vacuum inside the measurement chamber of the Cryo FIB-SEM

Characterising the Microstructure

- All samples were imaged using a Zeiss Crossbeam 550 (FIB-SEM) microscope while Elemental analysis was conducted using an Oxford Instruments XMax^N 150 EDX detector
- Samples were frozen in slush nitrogen prior to imaging
- The microscope can image soft solid materials up to nanometer resolution

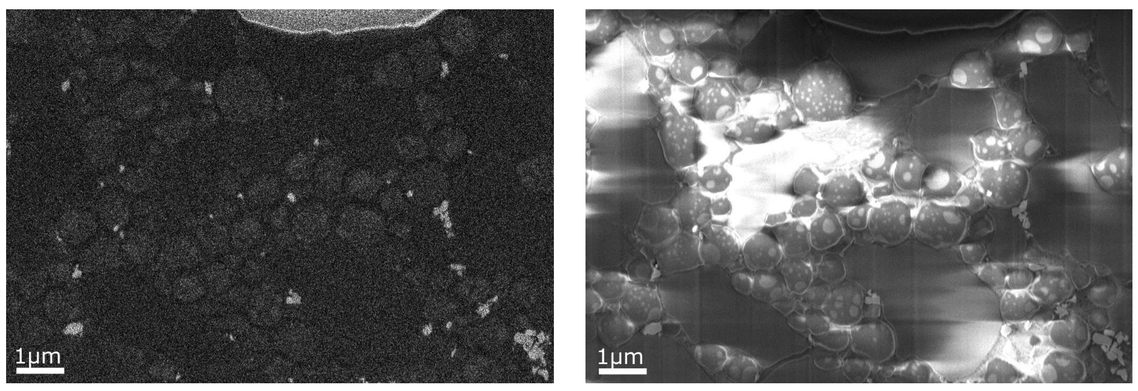

Figure 2: High magnification images of the formulation where the ion beam has been used to mill into the sample revealing the the cross-section. The image on the left is produced by backscattering of electrons (BSE), where heavier elements appear brighter revealing the presence of small particles. The image on the right is produced by secondary electrons (SE) emitted when interacting with the surface, revealing higher order structures possibly a double emulsion.

- The BSE image (left) shows the distribution of particles and indicates the droplets in the SE image (right), contains elements heavier than the continuous phase
- The droplets shown on the SE image (right) seem to to have droplets of a different phase within them, such as a double emulsion

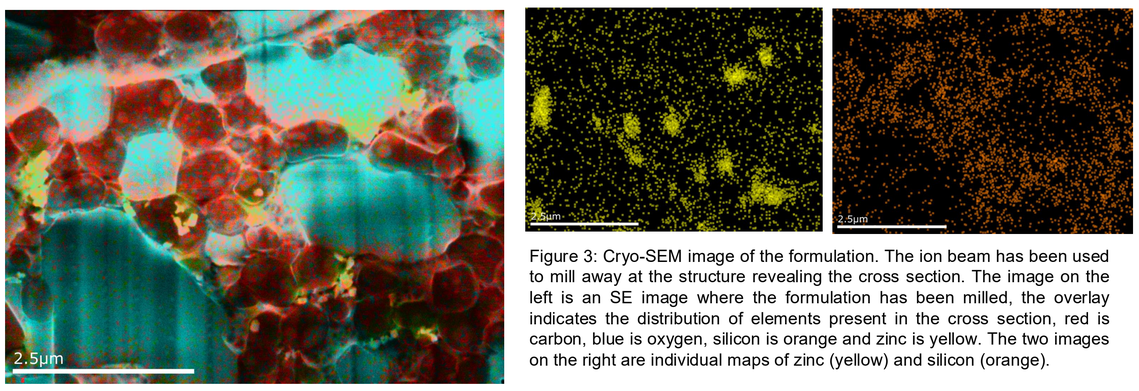

Figure 3: Cryo-SEM image of the formulation. The ion beam has been used to mill away at the structure revealing the cross section. The image on the left is an SE image where the formulation has been milled, the overlay indicates the distribution of elements present in the cross section, red is carbon, blue is oxygen, silicon is orange and zinc is yellow. The two images on the right are individual maps of zinc (yellow) and silicon (orange).

- Mapping the distribution of the X-rays can help with interpretation of the microstructure of the sample
- The maps of the carbon X-rays reveals that the droplets observed are composed mainly of carbon, while the bright particles observed are confirmed as containing zinc
- The silicon seems to be dispersed throughout the oil droplets, possibly being the other component of the double emulsions

Thin Film Analysis

- All samples were imaged using a Zeiss LSM700 laser scanning microscope
- Small aliquots of product were dyed with Nile Red before being imaged, as the Nile Red will bind to the oil [1]
- Imaging was done using a x 63 oil objective and using a 488nm laser to excite the Nile red dye
- The formulation was drawn down to thin films (<100 microns) and imaged at as a function of time to observe the drying behaviour of the films

Figure 4: Photo of the Zeiss LSM700 laser scanning microscope.

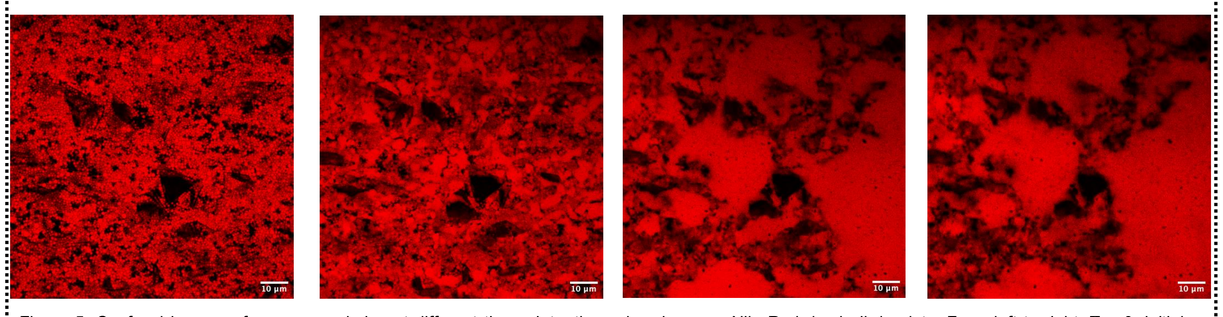

Figure 5: Confocal images of sunscreen drying at different timepoints, the red regions are Nile Red dyed oil droplets. From left to right: T = 0, initial deposition; T = 6 minutes, partial coalescence; T = 10 minutes, near full coalescence and T = 12 minutes, arrested coalescence.

- The images in Figure 5 shows how the microstructure changes as the formulation dries
- Coalescence at the later timepoints reveals pools of oil with dark spots within them, comparable the structures found in the Cryo FIB-SEM image

Conclusions

- We have demonstrated that both cryo-FIB SEM and confocal microscopy can provide key details of the microstructure of formulations and how this changes upon drying
- Confocal microscopy can be used to track the evolution of the microstructure of formulations, which complementing the cryo-SEM which can be used to image the initial microstructure in much higher resolution than optical techniques
- The deployment of such techniques could help to establish a framework for assessing how the microstructure of cosmetics changes when in use

REFERENCES: [1] Halim, Ronald, and Paul A. Webley. "Nile red staining for oil determination in microalgal cells: A new insight through statistical modelling." *International Journal of Chemical Engineering* 2015.1 (2015): 695061.

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