



INTRODUCTION

The microstructure of detergent products determines to a large extent the physical and textural properties of these products. Developing a proper understanding of the microstructure, particular the spatial distribution and interaction of chemical components is a key tool in developing products with desired properties. Microscopy is an indispensable tool to elucidate the microstructure and properties of these products. For detergents commonly the following microscopic techniques are used: conventional light microscopy (LM), confocal scanning light microscopy (CSLM), transmission electron microscopy (TEM), scanning electron microscopy with energy dispersive X-ray analysis (SEM-EDX), X-ray microtomography (XRT), Fourier transform Infrared (FTIR) microscopy. Each method has its own advantage and limitations. The 'Holy Grail' of microscopy is to reach real time three-dimensional imaging of large spatial volumes of samples with high spatial resolution, high chemical specificity (contrast) under environmental conditions without sample disturbance. All these goals can not yet be reached with either of the used techniques. Correlative microscopy offers the possibility to combine the advantages of several techniques by reconciling images of the same sample volume. In this study the real three-dimensional structural information obtained by XRT with high spatial resolution of SEM and high chemical specificity of FTIR. The success of correlative microscopy imposes special demands on the sample preparation. The prepared specimen should be suitable for each imaging technique. The method was tested on a spray dried detergent base powder.

METHODOLOGY

The detergent granules were embedded in polyacrylate (Lowicrylic HM20) at -40°C followed by UV polymerisation. (figure 1). The embedding block was imaged by XRT using the Skyscan 1072 desktop XRT system. XRT produces two-dimensional images of projections of the sample. A set of flat cross sections (1024 * 1024 pixels) was obtained after tomographical reconstruction of images acquired under different rotations over 180 degrees with a step size of 0.45 degrees. For SEM and FTIR imaging the embedding block was fractured. The embedding block was glued onto a standard SEM stub using two-component glue and trimmed in a microtome using a razor blade and glass knife for the initial rough trimming. For the final fracture a freshly made glass knife was used to obtain the best possible surface. A field emission scanning electron microscope (SEM, Jeol JSM6301F) was used to image the remaining fractured embedding block. The surface of the block was coated with a Au layer to prevent electrical charging during the observation. Observations were made at low accelerating voltage of the primary electron beam (15 keV). For subsequent FTIR imaging the Au layer was removed by trimming the block in the microtome. This resulted in a difference of about $10\ \mu\text{m}$ between the surfaces imaged by SEM and FTIR (trimming can be prevented by using a C coating for SEM). For FTIR imaging the Perkin Elmer Spotlight with Attenuated Total Reflection (ATR) was used whereby the sample is in close contact with the ATR crystal (image size: $128*128$ pixels). 3D visualisation and transformations in 3D space were performed using the Amira Resolve RT software from Mercury.

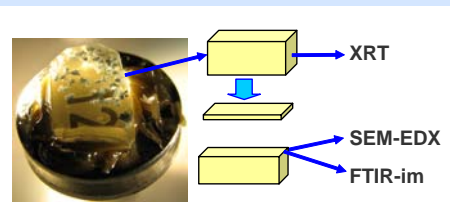


Figure 1. Detergent particles embedded in polyacrylate resin indicating which parts of the embedding block were used for imaging

RESULTS

Figure 2 shows the combination of XRT with SEM. A good correlation was found between XRT and SEM. The 3D information obtained by XRT is supplemented by high resolution information obtained by SEM together with the element specific information obtained by EDX. In the XRT and SEM images a thick and dense wall is visible around the powder particles. EDX revealed that they contained a high amount of sulphur and sodium. Furthermore clusters of zeolite (high Al and Si levels) were detected by EDX.

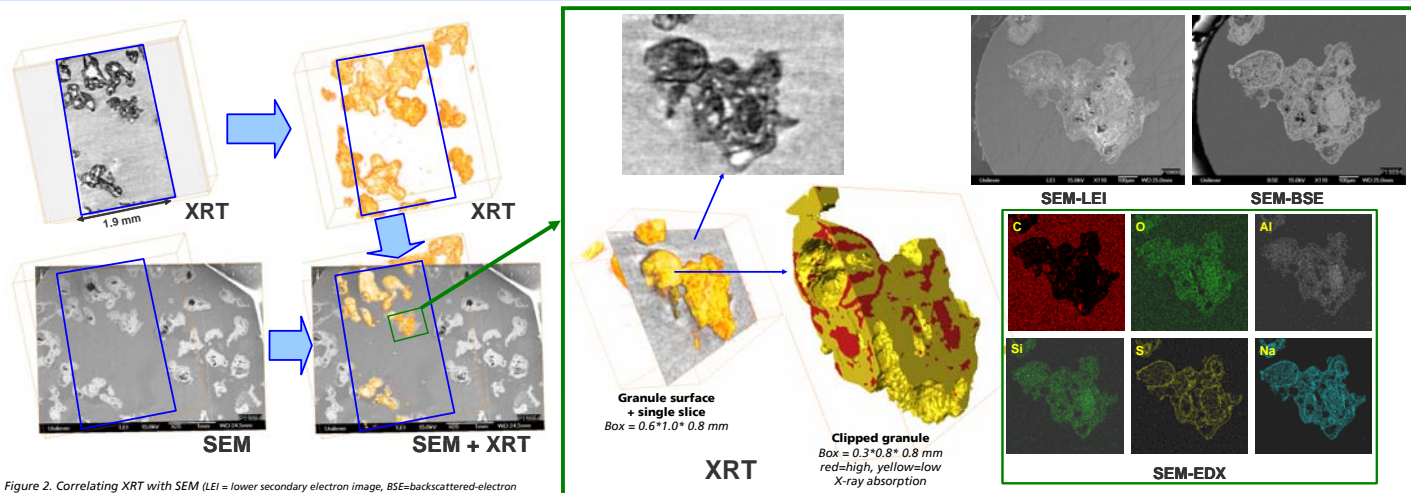
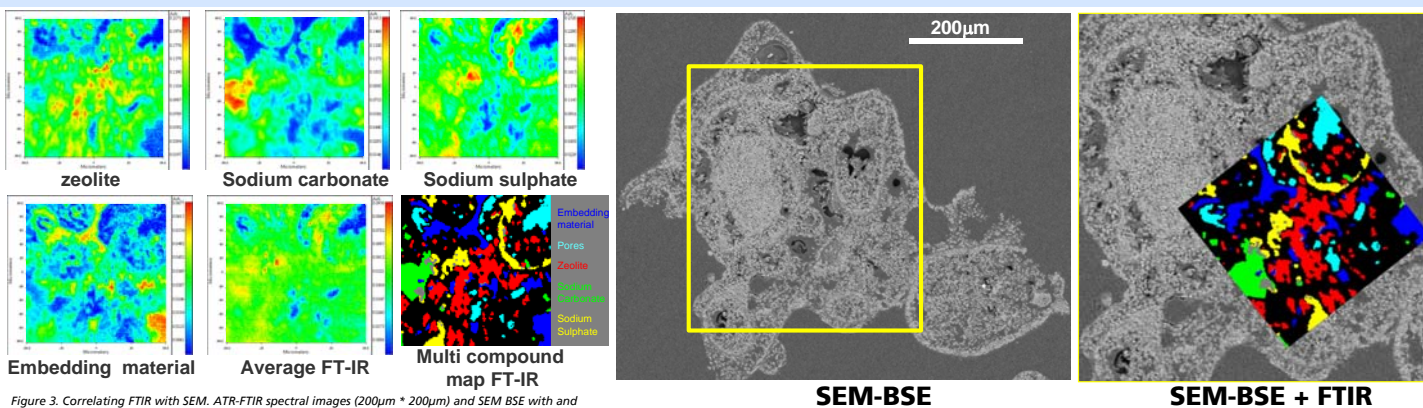


Figure 3 shows the combination of SEM with FTIR imaging. FTIR imaging records an entire FTIR spectrum for each pixel. From these spectra, functional group images were generated. With FTIR the following components could be identified: zeolite, sodium carbonate, sodium sulphate and the polyacrylate embedding material. FTIR images were obtained at low spectral resolution ($16\ \text{cm}^{-1}$). At higher resolution ($8\ \text{cm}^{-1}$) also surfactant LAS could be detected. The wall around the powder particles was identified as sodium sulphate. A good correlation was found between SEM and FTIR.



CONCLUSION

Correlative microscopy combines microscopic techniques in an optimal way by imaging the same sub-sample on the same location. It bridges the gap between 3D imaging, high resolution imaging and spectral imaging. This study showed that images of XRT, SEM-EDX and FTIR of a detergent granule can be correlated providing additional information which can not be obtained by using the techniques separately. The 3D internal and external structure of detergent granules can be investigated from milli - to nano scale with detailed spatial information about the components present. This will generate knowledge how to design optimal microstructures for laundry products to obtain product properties demanded by the market.

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