Cryo preparation of brittle particles for SEM-EDX analysis

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Modern detergent powders are brittle, dissolve rapidly, have a large specific surface and a viscous liquid component as glue. All have to be retained during microstructural investigation. A controlled cross fracture or section through the granules is difficult as a result of the brittleness of the granules, the porosity and the presence of crystalline particles. Good cross sectioning is only possible using an ultra-microtome on fully embedded particles. The liquid organic component however dictates a cryogenic approach to prevent redistribution of the organic solvents during embedding and curing but also to minimise smearing during sectioning.

Lowicryl HM20 was used for embedding the detergent granules at -40°C [1]. Processing, infiltration and curing of the resin is slow but Lowicryl, being a low viscosity resin, infiltrates into all pores and small crevasses in the sample; resulting in complete embedding of the particle. The embedded granules are easily sectioned and imaged on the block face. Drawbacks of the method were the poor Z-contrast between the Lowicryl embedding medium and the organic components present in the sample, and also the inability to differentiate between Lowicryl and the organic liquids when using EDX analysis (figure 1).

As a second approach, the granules were embedded at low temperature frozen in 2.3 M Sucrose and cryo planed at very low temperatures [2,4], typically -160°C (figure 2, 3). The lack of Z-contrast between the organic liquids and the sucrose was overcome by dissolving the Sucrose in a saturated lead nitrate solution and imaged using the BSE detector. This mixture slowly vitrifies in the cold microtome chamber [3] and has a high BSE yield by the presence of the Pb ions. Differentiation between the organic liquids and the lead-sucrose is now easily demonstrated based on the Z-contrast of the BSE images; EDX spectroscopy confirms the position of the lead-sucrose embedding material and the organic components of the granules when lead is used as marker for mapping the embedding medium.

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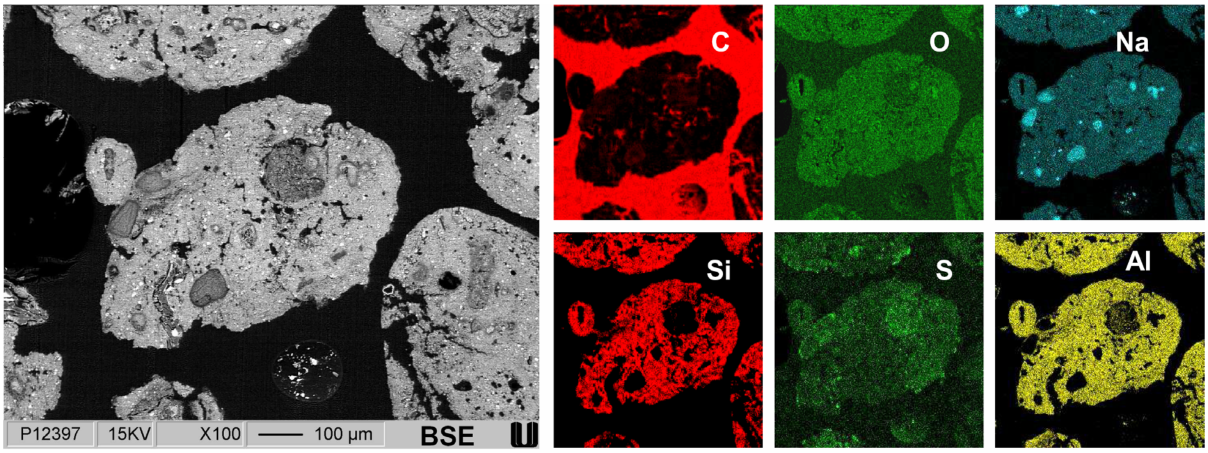

Figure 1. Typical data set of embedded and cross-fractured particles showing high contrast in BSE imaging mode. The low viscosity of the embedding medium fills all pores and crevasses of the detergent granules. The liquid organic compound is not displaced by the embedding procedures. The accompanying elemental maps display the different elements present in the sample. No significant differences in Z-contrast are present between the Lowicryl embedding resin and the organic components of the detergent granule.

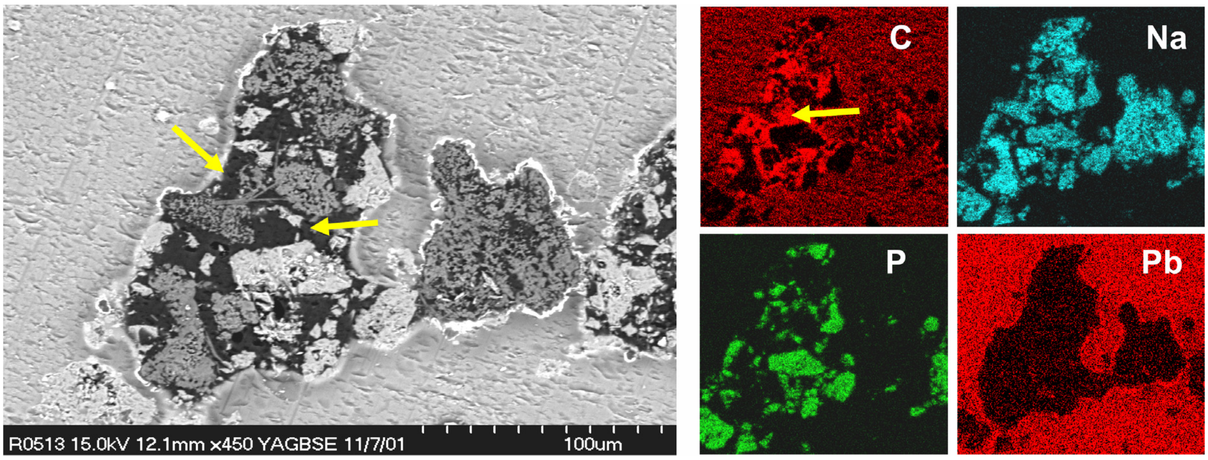

Figure 2. BSE image and corresponding element maps of the detergent granules after embedding and cryo-planing in the lead-sucrose mixture. The organic liquid components contrast in the BSE images with the higher Z yield of the lead present in the sucrose. The corresponding EDX maps of the carbon and lead signals can be used to further discriminate between the embedding medium and the various components the granule.

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