Application Note: XRF

Silicone Distribution on Consumer Paper Products by Micro-EDXRF

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Introduction:

Consumer paper products such as facial tissues or paper towels are often chemically treated with silicones to enhance the performance and/or manufacturability of the product. Properties enhanced or modified by silicone treatments include liquid absorbency, softness, wet and dry strength, processing wear resistance, antistatic qualities and dust and lint reduction during use. The distribution of the silicone treatment, whether it is uniform or patterned for example, also impacts the effectivity of the treatment; however, the silicone treatment doesn't always distribute on the paper product as intended. Scientists and manufacturing engineers are interested to characterize the silicone distribution in order to correlate distribution with optimal product performance and manufacturing properties.

In this application note, the EDAX Orbis micro-EDXRF (Energy-Dispersive X-ray Fluorescence) spectrometer was used to characterize the distribution of silicone applied to a facial tissue. The Orbis uses a low-power X-ray tube and collimating or focusing optics to generate an X-ray beam. Samples are mounted on an XYZ stage and elemental and full spectral maps are created by rastering the sample under the excitation/detection system. The X-ray detector is sealed with a Be window which allows for an elemental detection range from Na to U. The Orbis is an ideal tool for measuring silicone distribution because:

- XRF is non-destructive
- Little or no sample preparation is required
- A simple, unattended, simultaneous multi-element mapping analysis is possible

Experimental:

For this study, the Orbis configuration was as follows:

Mo tube (40kV, 40W)
Polycapillary lens (40 µm spot size FWHM @ Mo(K_α); Note: Spot size at Si(K_α) is roughly 70 to 80 µm FWHM due primarily to beam divergence at lower X-ray energies)
30 mm^2 Si(Li) detector
Software options: Vision Spectral Mapping
Orbis Image and Display

The tissue sample (measured silicone coverage = 1.4 wt% Si) was fixed to a metal frame and a strip of double-sided carbon tape was used to mask a small portion of the tissue in order to demonstrate how a Si-free area of the X-ray map would be displayed (see Figure 1). The metal frame was then mounted approximately 1" above the XYZ stage to minimize background X-ray scatter.

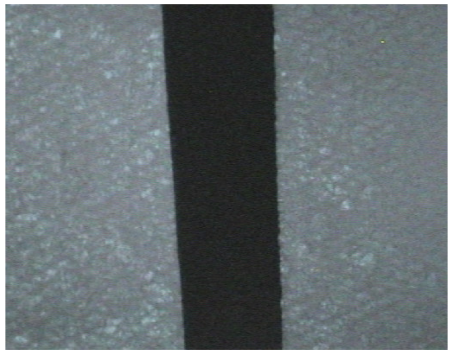

Figure 1: Facial tissue masked with carbon tape

The tissue was mapped under the following conditions:

Tube power = 25 kV, 25 W
Chamber atmosphere: Vacuum
Map area = 15.6 mm x 11.3 mm
Map resolution = 128 x 100 pixels
Dwell time = 600 ms
Map type = ROI (i.e. no background subtraction)
Total map time ~ 4 hours
(Note: The target acquisition time was set to map 6 samples per day. To date, no attempt has been made to minimize acquisition time further.)

Results and Discussion:

The Si map is displayed in Figure 2 using a green grey-scale palette with the brightest green representing the highest Si(K) intensities.

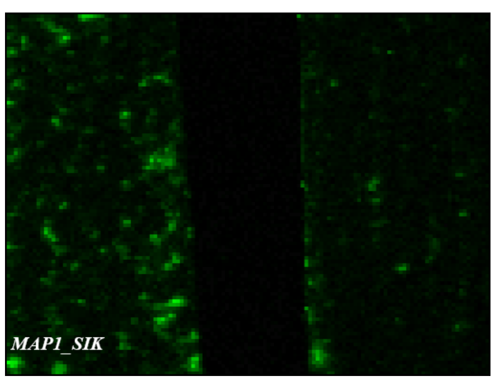

Figure 2: Si(K) ROI map from facial tissue

The Si map clearly shows hot spots of Si intensity and the blank area masked by the carbon tape. From the Si map, spectra associated with each mapping pixel can be recalled or a series of spectra can be summed from a specified region of the map. In

Figures 3a and 3b, three regions of the Si map have been marked and the spectra within the marked regions summed together: hot spot (red), blank (blue) and low to no intensity area of tissue (yellow).

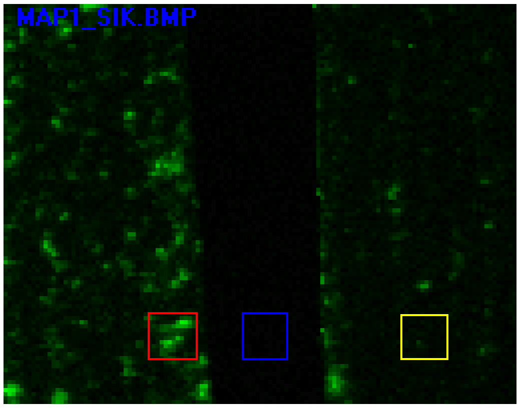

Figure 3a: Marked Si(K) map

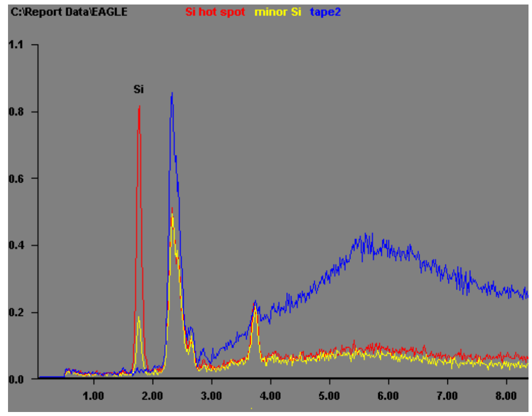

Figure 3b: Summed spectral overlay from marked areas in Figure 3a

Clearly, the area within the yellow box contains a low, but significant, level of Si intensity indicating the presence of silicone. However, due to the influence of high Si intensity in several spots on the relative scaling of the map, the intensity in the low-level areas is scaled to a similar brightness level as the masked area.

In this case, a 3-logarithmic band color scaling, developed by researchers at the National Institute of Standards and Technology (Washington DC, USA) was applied to the Si map in an attempt to better highlight the silicone distribution. This scaling, which is available in the Orbis Image and Display software, takes the normal intensity scaling and creates three separate logarithmic intensity scales: 0 to 1% relative intensity (blue); 1 to 10% relative intensity (green); 10 to 100% relative intensity (pink). The resulting 3-band Si map clearly displays all levels of the Silicone distribution as shown in Figure 4. The mapped area of the tissue is largely covered by a low-level of silicone (green). About 20% of the mapped area is covered by a high-level of silicone (pink). {Note however, in this example, because the sample was masked with carbon tape, the calculated fractional area is not truly representative of the actual silicone distribution.} Any fractional area of the map can be calculated by selecting the desired section of the intensity histogram shown at the right of Figure 4. The blue area of the map corresponds to the masked area of the tissue.

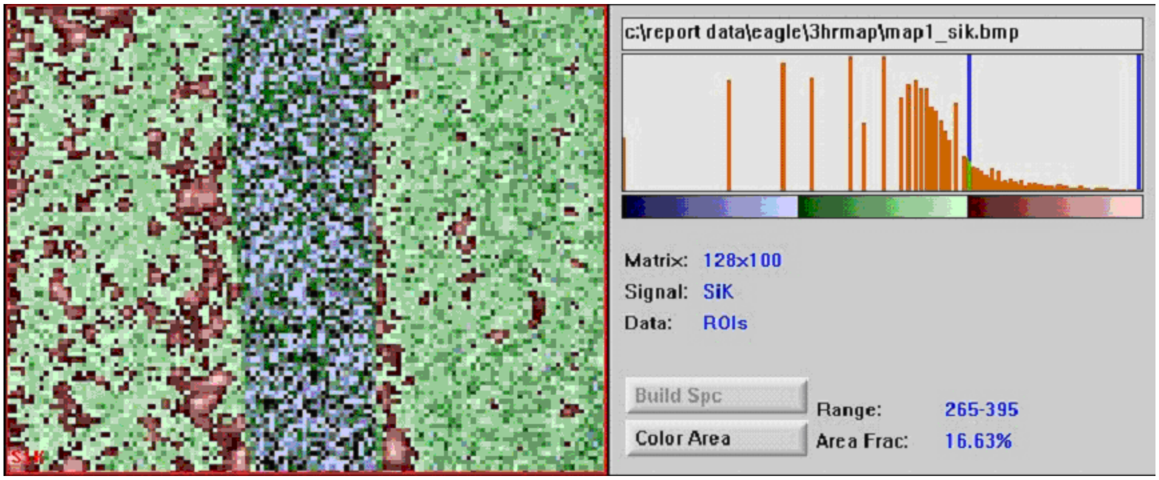

Figure 4: 3-logarithmic color band scaling of Si(K) ROI map

In this example, the intended distribution was to have the silicone in discrete spots on the tissue. This was confirmed by the hot Si spots (Figure 4 – pink) in the elemental map. The low level distribution of silicone (Figure 4 – green) over the majority of the tissue was unintentional.

Conclusions:

The Silicone distribution on a consumer paper product was successfully mapped using the Orbis with an X-ray Polycapillary Lens and a Mo anode tube. A monochromatic grey-scale representation of the Silicone distribution contains hot spots of Si intensity which obscure the overall Silicone distribution. Application of a 3-logarithmic band color scaling to the Si intensity map displays all levels of the Silicone distribution present in the sample simultaneously. This logarithmic scaling highlights an inadvertent low-level Silicone distribution over a majority of the mapped area.