A112   Determination of Unique Compositional Patterns in Glasses by Micro-XRF: Multivariate Approach and Statistical Analysis

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After attending this presentation, attendees will learn about application of micro-XRF to glass and soil analysis.

This presentation will impact the forensic science community by providing practical information about application of micro-XRF to chemical analysis.

X-ray Fluorescence (XRF) spectroscopy is a useful tool for identification of substances and confirming their identity with little or no sample preparation. New capabilities of the energy dispersive XRF analytical microscope (micro-XRF) enable the recording not only of spectra from small glass particles (as small as 50-100 microns) but also the hyper-spectral image of any object with high spatial resolution (<10 micrometers). Hyper-spectral image is a set of the data which contain information about position of the point along with full XRF spectrum at this point. This means that the data can be mined for unsuspected elements after the measurements have been made, and that statistical methods (multivariate analysis) can produce chemical distributions of the elements (Image analysis) and/or material classification based on Principal Component Analysis, in particular, with association between elements that can aid in identification of bonded phases. For example, statistical analysis of micro-XRF data for glasses can be used to locate the make, model, and year of car by analyzing a glass chip. This presentation will provide practical insights into the application of the micro-XRF to the analysis of glasses and soil.

An XRF analytical microscope was used in this study. This desktop unit utilizes a portable 50W X-ray source for excitation, two switchable (as small as 10 microns) monocapillaries for different spatial resolution, and the unique capability to work in vacuum (enhanced sensitivity for light elements), in partial vacuum, and under ambient conditions. In addition to the XRF spectrum/image, the XRF microscope provides a micro-transmission image of the material. Standard software package includes quantification using fundamental parameters method, basic statistical analysis for multi-point measurements, image analysis, and image processing.

X-ray fluorescence spectrum of the glass strongly depends on X-ray optical system, sensitivity of the detector, and accelerating voltage. In addition, background from the substrate will contribute to the spectrum of the small glass pieces because excitation X-ray penetrates through the glass and interacts with substrate. This effect becomes very important for particle size of 300 microns (or less) or powder. The change in the spectrum due to the shape or size will lead to the different quantification of the sample (different composition). A method which allows one to minimize this effect or take it into consideration was developed. In this presentation, examples of spectra from bulk material, small glass pieces, and powder will be shown.

Spectra of glass from several car manufacturers and commercial glasses (microscope slides, window glasses, fuse glass) in the range of 1.00-40.96 keV (<400 spectra) were collected and analyzed. Because only a few spectra have an additional features in the energy range above 15 keV, spectra were truncated and analysis was done in spectral range of 1.00-15 keV. Standard FPM algorithm without any correction and/or calibration was used to calculate concentration of Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, TiO_2, MnO_2, Fe_2O_3, As_2O_5, and CeO_2 in all samples. This set of concentration to build a data set for PCA was used. All spectra and concentration data sets were scaled before Principal Component Analysis was applied. Correlation between classification based on spectral analysis and concentration analysis will be shown.

Glass, XRF, Analysis