Determination of compositional patterns in glasses and sands: multivariate approach.

Sergey Mamedov, Ph.D

Horiba Scientific, 3880 Park Ave, Edison, New Jersey 08820, USA

X-Ray Fluorescence (XRF) spectroscopy is useful tool for identification 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 spectra of small glass particles (as small as 50-100 microns) but also hyper-spectral image of any object with high spatial resolution, and collect average spectrum over certain area. 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 features after the measurements have been made. Multivariate analysis (MVA) can produce chemical distributions of elements and/or material classification based on Principal Component Analysis (PCA), Partial Least Square Discriminative Analysis (PLS-DA), in particular, with association between elements that can aid in identification of bonded phases. For example, analysis of micro-XRF data of car window glasses can be used to locate the make, model, and year of car or it can differentiate sands, for example from East and West coasts, based on analysis of similarly in spectra of the sands taken at different location. This presentation will provide practical insights into the application of the micro-XRF to the analysis of glasses and sands and application of PCA and PLS-DA to data analysis.

The XGT-7200V XRF analytical microscope was used in this study. This desktop unit utilizes a portable 50W X-ray source, two switchable monocapillaries, and the capability to work in vacuum, in partial vacuum, and under ambient conditions.

X-ray fluorescence spectrum of the materials strongly depends on X-ray optical system, sensitivity of the detector and accelerating voltage. In addition of that, background from the substrate will contribute to the spectrum of the small pieces because excitation X-ray penetrates through the material and interacts with substrate. This effect becomes very importance for the 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 concentration profile of the sample (different composition). We developed method which allows one to minimize this effect or take it into consideration. In this presentation we’ll show examples of the spectra from bulk material, small pieces and powder.

We collected and analyzed spectra of the glasses from several cars manufactures and commercial glasses and sands from different location in US and Europe in the range of 1.00-40.96 keV (<400 spectra). Because the only 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 oxides in all samples. This set of concentration was used to build a data set for PCA and PLS-DA. Correlation between classification based on spectral analysis and concentration analysis will be shown. The data shows that MVA allows differentiate samples which have very similar spectra features (concentration profiles) and this approach may be useful for forensic investigation.