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DAAS Deutscher Arbeitskreis für Analytische Spektroskopie

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Nonaqueous Suspensions in Sample Preparation forTotal Reflection X-Ray Fluorescence Analysis

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Total Reflection X-ray Fluorescence Analysis (TXRF) is a modern technique for elemental composition determination. The main advantages of TXRF are high sensitivity, wide dynamic ranges of concentrations, multi-element analysis and very low influence of matrix effects. Method is capable to analyze samples in liquid (after drying) and solid states. The last feature is very useful, because it makes possible to reduce analysis time and complexity significantly by refusing the stage of sample digestion.

The most convenient approach to analysis of solids is sample preparation in suspensions. An important factor responsible for the correctness of the results of analysis is the stability of suspensions. In case of particles with sizes higher 1 μm stable colloids could not be achieved. It is enough if suspension remains stable during preparation and sampling time. The negative effect of sedimentation is often observed for samples with an inhomogeneous matrix. In this case, nonuniform sedimentation of particles is observed, which leads to the error in measurements and decreases the repeatability.

In this work we have suggested a new approach to the enhancement of the sedimentation stability of suspensions (coal and coke) using ethylene glycol or glycerol as the dispersion media. These substances are readily available, can be completely removed by evaporation, and have a high viscosity (1490 mPa·s for glycerol and 16.1 mPa·s for ethylene glycol against 0.89 mPa·s for water).

The experiments were carried out with coal coke samples. Grounded samples with 9 μm average particle sizes were weighed on analytical balances with an accuracy of 0.1 mg and a portion of 8-10 mg was transferred to a 1.5 ml vial. Then, 1 ml of a surfactant solution (0.01 % Triton X 100 in water), 1 ml of ethylene glycol and 1 ml of glycerol, and 3 5 μl of an internal standard solution (gallium, 1 g/l) were added into the test tube. The mixture was blended to homogeneity and allowed to stand for a specified period of time. Then 2 μl of the suspension was transferred to a sample career. Aqueous suspensions were dried in a vacuum desiccator, and suspensions in ethylene glycol and glycerol were dried on an electrical hot plate at 70 80 °C. X-ray fluorescence spectra were recorded using Bruker Nano S2 PICOFOX spectrometer (Germany). Monochromatic Mo Kα radiation (17.44 keV) was used for fluorescence excitation. The spectrum acquisition time was 650 s.

It was found that within 5 min of sedimentation the suspended analyte concentration changes by 10–15 rel. % in nonaqueous media and by up to 50 rel. % in aqueous media which demonstrate sufficient stability improvement. For the analysis purposes, 5 min is excessive time, but this result allows us to expect that nonaqueous media suspensions will be suitable for analysis of less stable suspensions composed of larger and denser particles. It has been also determined that the repeatability of measurements in nonaqueous suspensions is 3 5 rel. % against 10 15 rel. % for aqueous suspensions. Even the glycerol suspensions are more stable; ethylene glycol media are more convenient in operation because homogenizing glycerol suspensions is more complex task due to its very high viscosity.

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