Quantitative X-ray Fluorescence Analysis of Biomass: Objective Evaluation of a Typical Commercial Multi-Element Method on a WD-XRF Spectrometer

Abstract:
ABSTRACT: With the increasing utilization and trade of biomass, there is a growing need for quick and reliable quantitative chemical analysis of the inorganic elemental composition of biomass. X-ray fluorescence (XRF) spectrometry performed directly on the raw biomass with limited prior sample preparation is an attractive method to this end. However, reliable calibration of XRF spectrometers for universal multi-element analysis can be very hard to implement mainly due to problems with matrix corrections. XRF users thus often rely on commercial precalibrated or standardless methods delivered with their XRF spectrometer. These methods are often sold without any guarantee on performance. Given the actual frequent use of these methods, along with their potential as ready-to-use methods for multi-element analysis of biomass, we investigate here the performance of a typical commercial precalibrated/standardless method recently purchased with a 4 kW wavelength dispersive (WD) XRF spectrometer. The accuracy (trueness and precision) is determined by analyzing the certified inorganic elements in 13 certified reference materials (CRMs) of diverse vegetal/plant origin. The certified elements detected by the XRF are Na, Mg, Al, P, S, Cl, K, Ca, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Rb, Sr, Mo, Ba, and Pb. The relative systematic error (bias, trueness) is typically better than ±20% for elements in the range 25 to 100 ppm, better than ±15% for the range 100 to 1000 ppm, and better than ±10% for concentrations above 1000 ppm. The relative precision (measured as the relative standard error) is better than ±5% (typically ±1–2%) for concentrations >25 ppm. Quantifying elements below 25 ppm is possible in some cases, but it requires a more detailed study for each specific element. Occasionally, larger relative biases of up to ±40% can occur for certain elements in certain samples, so care has to be taken to carefully test the applied method for the particular samples and elements of interest. The XRF method can further be used to estimate the ash yield from biomass combustion with a relative bias (trueness) typically better than ±15%. This involves the determination of silicon (Si) and titanium (Ti) by XRF. The choice of matrix composition in the matrix correction model and the influence of sample moisture and sample grain-size are also addressed. The instrument and method are described in detail allowing for comparisons with other similar XRF spectrometers often already available in industrial analytical laboratories.

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