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Determination of trace elements and macronutrients in agricultural soils using energy dispersive X-ray fluorescence as a rapid and precise analytical technique

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Abstract

X-ray fluorescence spectrometry (XRF) is a rapid and inexpensive method for soil analysis. Although, not as precise and accurate as mineral acid digestion-Inductively Coupled Plasma spectrometry for soil testing at present, the XRF method has the potential to be optimised. The objective of this study was to reduce moisture effects, particle size effects and spectra interferences on trace element and macronutrient analyses by improving soil sample preparation and XRF calibration, using bench top Energy-dispersive X-ray Fluorescence Spectrometry (EDXRF). The soil particle size effects study involved samples prepared as pressed powders, pressed pellets, and pressed pellets with a wax binder. After which, the recoveries of Al, Cr, Ni, Mn, Pb, Ca, Fe, K, Mg, P, S and Zn were evaluated as a measure of accuracy and precision. The XRF was calibrated with the fundamental parameters (FP) and matching library (FPML) methods to reduce spectral interferences and validated with certified reference materials. In addition, both XRF methods (FP and FPML) were compared with *aqua regia* digestion (acid digestion) -Inductively Coupled Plasma-Optic Emission Spectrometry (ICP-OES) using concordance correlation coefficient (CCC), whereby a value of 1 indicated good agreement between methods. There were significant differences ($p < 0.05$) between the sample preparation methods and the pressed pellet with wax binder had the best accuracy and precision for all the elements. In addition, for the calibration study, the FPML gave better recoveries of Ni, Ca, Mg, S, P, Cr compared to the FP, however, for Fe, Zn, K and Mn the FP had better recoveries than the FPML. Furthermore, there was good agreement ($CCC > 0.80$) between both XRF methods and ICP-OES for all elements except Al, P, Cu, K, S and Cr ($CCC < 0.60$). *Aqua regia* digestion underestimated the total concentration of Al and K, thus, the XRF had better accuracy for predicting these elements. However, for Cr, S, Cu and P, the XRF overestimated the concentration of these elements in soil. This study has shown that the XRF is as accurate as the ICP-spectrometry for most elements when properly calibrated with the advantage of having a shorter sample preparation. Thus, environmental analysts can monitor soils frequently using the XRF without losing analytical results especially with Al and K and also incurring substantial costs in analyses as with traditional methods.

Keywords: soil; elemental analysis; X-Ray Fluorescence; spectrometry; calibration