

DETERMINATION OF SOIL ELEMENTAL COMPOSITION USING PORTABLE XRF ANALYSER

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Abstract: Ex-situ elemental analysis of soil and silica-sand growing medium using portable XRF analyser Niton XL3t GOLD+ is presented. Object of study was *Haplic Cambisol* (Vatín, Czech Republic) and silica-sand growing medium. Both of them are supposed to be used for further pot experiments with selenium application. Application two forms: sodium selenium and selenium particles; and two concentrations: 0.2 mg and 2 mg). Therefore elemental analysis and trace elements determination was necessary. We came to the conclusion, that portable XRF analyser represents quick and convenient methods for determination of elemental composition in soils and growing medium.

Key Words: elements, soil, growing medium

INTRODUCTION

Elements contain under natural conditions may in soil differ widely and might be characterized by variety of parent material, chemical processes, differing in the extraction agent, concentration, time of extraction and others. Mainly parent material, its mineralogical composition, intensity of pedogenetic and weathering processes influencing elemental composition of soil (Kabata-Pendias, Pendias 2001). The result of given processes is soil colloidal complex formation. According to Tessier et al. (1979) different elements and their speciation in soil could be defined as exchangeable, bound to the carbonates, bound to the manganese and iron oxides, bound to soil organic matter, and residual. Modern analytical procedures and methods have been developed and multielemental determination (e.g. AAS, ICP-AES, ICP OES) is today used (Soltanpour 1991, Marchand et al. 2011). The aim of our work was to characterize soil and growing medium (substrate) using the latest performance of Niton XL3t GOLD+ portable XRF analyser. X-ray fluorescence (XRF) is a non-destructive analytical technique used to determine the elemental composition of materials. XRF analyzers determine the chemistry of a sample by measuring the fluorescent (or secondary) x-ray emitted from a sample when it is excited by a primary x-ray source. Each of the elements present in a sample produces a set of characteristic fluorescent x-rays that is unique for that specific element, which is why XRF spectroscopy is an excellent technology for qualitative and quantitative analysis of material composition.

MATERIAL AND METHODS

Object of study was an organo-mineral soil (*Haplic Cambisol*, Vatín, Czech Republic) and silica-sand growing medium (substrate). It was necessary to characterize both of them before using them in our pot experiments in phytotron, and before selenium application. Samples were air dried, and sieved through 2.0 mm screen. Ex-situ elemental analysis was carried out using a portable XRF analyser Niton XL3t GOLD+ (Figure 1). The analyser features a 50 kV and 200 μ a x-ray tube with an Ag anode and large area Silicon drift detector (SDD). Four filters provide an optimized excitation from potassium ($Z = 19$) to uranium ($Z = 92$). The XL3t analyser does not require any specific calibration, calibration factors were set to the factory values. All analysis were performed in the Mining mode, which is based on the Fundamental Parameter (FP) algorithm. Samples were always fully cupped to ensure “infinitely thick samples” condition. Every sample was analysed three times for

30s per main, high and low filters and 120s per light filter. Measured values were then averaged (www.tttenviro.com/manual-XL3-series). Acidity and conductivity were determined according to Zbíral (1997). Total carbon content was determined according to Nelson and Sommers (1982). Fractional composition of humus was made according to Kononová and Bělčíková method (1963). Evaluation of trace elements content was done according to the declaration 13/1994 Sb., law 334/92 Sb. (Němeček et al. 2010).

Figure 1 Analyser Thermo Scintigic Nitron XL3 Gold



RESULTS AND DISCUSSION

Acidity of silicone-sand growing medium was optimal and varied from 5.5–6.5. Acidity of Haplic Cambisol was acid. Conductivity of growing medium was higher to compare with mineral soil, but the limit for salinity (4 mS/cm^2) was not overstep. Content of humic substances, humic acids, and fulvic acids was comparable in both samples. Much more total organic carbon was found in silica-sand growing medium – see Table 1. We determined the following elements in silica-sand growing medium – *Cd, Pd, Nb, Zr, Sr, Rb, Fe, Ti, Ca, Si, K, Al, P, Cl, and S*. Under detection limits were – *As, Co, Cr, Cu, Se, Mo, Ni, V, and Zn*. As you can see from Table 2 limit (0.4 ppm) for Cd was overstep in silica-sand growing medium (6.602 ppm). In organo-mineral soil (*Haplic Cambisol*) we determined the following elements – *As, Ba, Cr, Nb, Zr, Sr, Rb, Pb, Zn, Fe, Mn, Ti, V, Ca, Si, K, Al, P, Cl, S, and Mg*. Under detection limits were – *Cd, Co, Cu, Mo, Ni, and Se*. We can conclude that wide range of elements were presented in *Haplic Cambisol* and no limits for risk elements content were overstep. Silica-sand growing medium contained less elements (not as broad) to compare with organo-mineral soil. Concerning both organic and inorganic soil constituents, X-ray absorption spektroskopy (XAS) methods have been successfully applied for element speciation and the identification of reaction mechanisms (Totsche et al. 2010). Micro XAS methods combined with X-ray fluorescence allow for a mapping and speciation of, metals as Cu, Pb and Zn (Manceau et al. 2004, Vantelon et al. 2005, Strawn et al. 2008).

Table 1 Basic chemical properties of Haplic Cambisol soil and silica-sand growing medium

| Sample | pH/H ₂ O | pH/KCl | Conductivity (mS · cm ⁻¹) | Sum of HS (g · kg ⁻¹) | Sum of HA (g · kg ⁻¹) | Sum of FA (g · kg ⁻¹) | TOC (%) |
|----------------------------------|---------------------|--------|--|---|---|---|------------|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| Silica-sand growing medium | 5.71 | 5.40 | 1.15 | 3.0 | 1.5 | 1.5 | 11.40 |
| Haplic Cambisol | 5.95 | 4.56 | 0.05 | 4.6 | 1.3 | 3.3 | 1.45 |

(1) Soil type, (2) active soil reaction, (3) exchangeable soil reaction, (4) conductivity, (5) sum of humic substances, (6) sum of humic acid, (7) sum of fulvo acid, (8) total organic carbon

Table 2 Trace elements content in silica-sand growing medium and soil sample (Haplic Cambisol)

| | Silica-sand growing medium (ppm) | Error (ppm) | Soil sample - Haplic Cambisol (ppm) | Error (ppm) |
|-----------|----------------------------------|-------------|-------------------------------------|-------------|
| <i>Ba</i> | <LOD | 38.401 | 474.114 | 32.758 |
| <i>Sb</i> | <LOD | 11.114 | <LOD | 11.459 |
| <i>Sn</i> | <LOD | 11.751 | <LOD | 13.280 |
| <i>Cd</i> | 6.602 | 3.816 | <LOD | 6.353 |
| <i>Pd</i> | 3.326 | 2.055 | <LOD | 2.818 |
| <i>Ag</i> | <LOD | 3.286 | <LOD | 2.664 |
| <i>Mo</i> | <LOD | 1.817 | <LOD | 1.855 |
| <i>Nb</i> | 2.687 | 1.181 | 12.827 | 1.572 |
| <i>Zr</i> | 75.626 | 2.270 | 263.109 | 4.827 |
| <i>Sr</i> | 18.222 | 1.169 | 71.290 | 2.404 |
| <i>Rb</i> | 7.882 | 1 | 62.288 | 1.849 |
| <i>Bi</i> | <LOD | 2.618 | <LOD | 6.791 |
| <i>As</i> | <LOD | 3.910 | 12.455 | 5.011 |
| <i>Se</i> | <LOD | 1.519 | <LOD | 2.213 |
| <i>Au</i> | <LOD | 5.733 | <LOD | 8.899 |
| <i>Pb</i> | <LOD | 5.264 | 33.695 | 5.267 |
| <i>Zn</i> | <LOD | 8.414 | 82.486 | 9.630 |
| <i>Cu</i> | <LOD | 13.828 | <LOD | 24.863 |
| <i>Ni</i> | <LOD | 27.679 | <LOD | 37.291 |
| <i>Co</i> | <LOD | 42.041 | <LOD | 118.525 |
| <i>Fe</i> | 3761.366 | 95.046 | 33185.707 | 312.459 |
| <i>Mn</i> | <LOD | 68.245 | 800.928 | 85.569 |
| <i>Cr</i> | <LOD | 26.791 | 144.543 | 24.092 |
| <i>V</i> | <LOD | 30.524 | 162.925 | 40.689 |
| <i>Ti</i> | 617.529 | 32.688 | 5592.479 | 100.257 |
| <i>Ca</i> | 35977.340 | 496.468 | 6023.226 | 327.098 |
| <i>K</i> | 4507.706 | 141.006 | 20381.639 | 356.847 |
| <i>Al</i> | 3562.406 | 186.529 | 45529.781 | 614.541 |
| <i>P</i> | 2496.389 | 87.350 | 522.886 | 110.704 |
| <i>Si</i> | 70739.375 | 491.360 | 223577.297 | 1131.114 |
| <i>Cl</i> | 2406.312 | 26.009 | 559.424 | 17.119 |
| <i>S</i> | 4552.898 | 56.340 | 3019.527 | 46.591 |
| <i>Mg</i> | <LOD | 2158.640 | 6321.030 | 1303.112 |

CONCLUSION

Ex-situ elemental analysis using portable XRF analyser Niton XL3t GOLD+ represents quick and convenient methods for determination of elemental composition in soils and growing medium. In future this study will continue and will be aimed at evaluation of correlation between trace elements content, mineral composition and soil organic matter quality.

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