

A novel approach of WD-XRF for the analysis of fluorine content in soil with suggested correction factors for Fe/Mn interference

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In our previous study, the wavelength dispersive X-ray fluorescence spectrometry (WD-XRF) was assessed to be feasible for the analysis of fluorine content in soil [1]. However, the limit of detection (LOD) value of 1-to-1 (w/w) pre-treatment method was still too high (812 mg-F/kg-soild) compared with other methods. Therefore, the existing pre-treatment method was modified to reduce the LOD value.

The soil was mixed with a polyvinyl alcohol at 9-to-1 weight ratio to 4 g of total specimen and then pressurized 20 tons over 20 seconds. The LOD value of 9-to-1 (w/w) pre-treatment method was reduced by about 38 times from existing method to 21.3 mg-F/kg-soild of modified method. This LOD value was comparable to that of other methods for determination of soil fluorine concentrations (Table 1).

Analytical instrument	Pre-treatment method	LOD (mg/kg)	Ref.
Selective ion electrode	Alkali fusion	3	[2]
Selective ion electrode	Pyrohydrolysis	20	[3]
WD-XRF	1-to-1 pellet	812	[1]
WD-XRF	9-to-1 pellet	21.3	This study

Table 1: Comparison of several methods.

In WD-WRF analysis, fluorine intensity measured at Bragg angle of 43.174° was affected by other elements having similar Bragg angle, such as Fe and Mn. Therefore, the intensity was corrected by calibration of F intensity with different Fe/Mn contents in soil sample. The sample specimen containing 0, 10, 20, and 50% of Fe₂O₃ and 0, 2.5, 5, and 10% of MnO₂ were prepared to calculate the correction factor (CF).

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[1] An *et al* (2012) *Spectrochim. Acta B* **69**, 38-43. [2] McQuaker, Gurney (1977) *Anal. Chem.* **49**, 53-56. [3] Sredovic, Rajakovic (2010) *J. Hazard. Mater.* **177**, 445-451.