

Fully automated on-line pressurized hot water extraction coupled with liquid-chromatographic-tandem mass spectrometric determination of pharmaceuticals in soils

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A new self-assembled apparatus for the extraction of solid samples has been implemented in order to develop a recirculating pressurized hot water extraction (R-PHWE) directly coupled to liquid chromatography-tandem mass spectrometry, using appropriately modified standard chromatographic equipment. The new apparatus was employed for the analysis of 34 target pharmaceutical compounds (PhCs) characterized by heterogeneous physicochemical properties (e.g., $-0.05 \leq \log D \leq 4.31$) in loam, silt-loam and silty-clay-loam soils. The use of a multivariate strategy involving the design of experiments [1] (DoE) allowed the identification of the best extraction conditions for the target analytes, monitoring temperature, pressure and number of extraction cycles. The optimization procedure successfully demonstrated that the number of cycles, in combination with high temperature, has a favourable impact on the recovery of target analytes. Regardless of the textural differences of the investigated soils, the application of the DoE set point (120°C, 7 cycles, and 110 bar) produced good recoveries (60% for most target analytes in all media) and accuracy (25% in almost all cases). The greenness of the proposed method was assessed in comparison with previously published methods for the analysis of PhCs in soils. The value obtained for the proposed method (0.78) was the highest due to a high level of automation and integration between the extraction process and the subsequent chromatographic analysis.

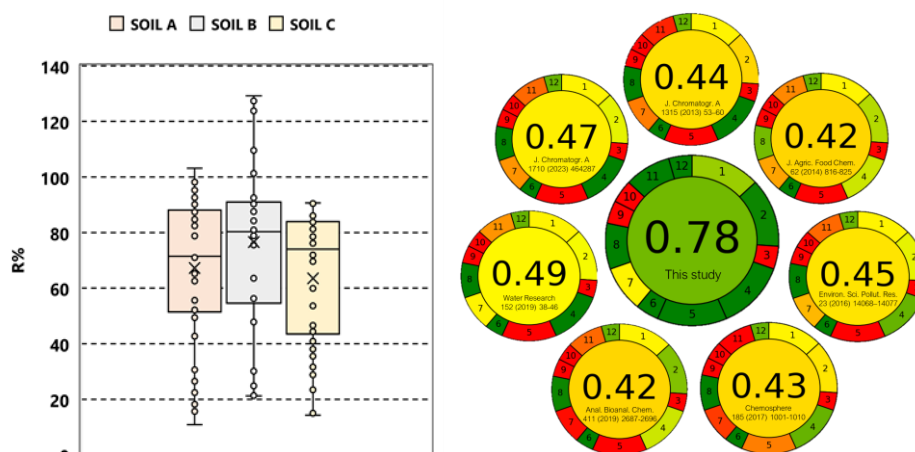


Figure 1: (A) Box plot of recoveries for the three soils under the optimized conditions. (B) Evaluation of greenness of this method compared with other seven studies.

Keywords: On-line extraction, solid samples, LC-MS/MS

References

1. Marzullo L., Ochkur O., et al., *Journal of Chromatography A*, 1677 (2022), pag. 463329. DOI: 10.1016/j.chroma.2022.463329