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Innovative On-line Pressurized Hot Water Extraction with Solvent Recirculation Coupled with Liquid Chromatographic-Tandem Mass Spectrometric Analysis of Pharmaceuticals in Soil

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In this study, a new self-assembled apparatus for the extraction of solid samples was built, using suitably modified standard chromatographic equipment, in order to develop a recirculated pressurized hot water extraction directly coupled to liquid chromatography-tandem mass spectrometry. In order to investigate the potential of this new extraction apparatus, the proposed system was applied to the analysis of 34 target pharmaceuticals compounds characterized by heterogeneous physicochemical properties (e.g., $-0.05 \le \log D \le 4.31$) in three soils with different textural characteristics (i.e., loam, silt-loam and silty-clay-loam). A multivariate strategy based on the design of experiments (DoE) was used to identify the best extraction conditions for the target analytes by studying temperature, pressure and number of extraction cycles. The optimization procedure successfully demonstrated that the number of cycles, in combination with the high temperature, had a favourable impact on the recovery of target analytes. The application of DoE set point to the three reference soils provided average semi-quantitative recoveries ($\ge 60\%$ for most target analytes in all soils) and good precision (< 25% in almost all cases), regardless of the textural differences among samples.

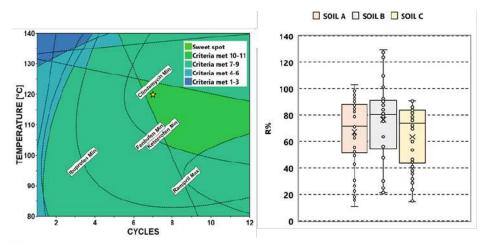


Figure 1: Sweet spot plot for the 12 significant CMAs; the star point refers to the best compromise in terms of R% values and extraction time (120 °C, 110 bar, 7 cycles), (B) box plot of recoveries for the three soils under the optimized conditions.

References:

[1] L. Marzullo, O. Ochkur, S. Orlandini, L. Renai, R. Gotti, O. Koshovyi, S. Furlanetto, M. Del Bubba, Journal of Chromatography A 2022, 1677, 463329.