

Evaluation of two methods to determine glyphosate and AMPA in soils of Argentina

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Argentine agricultural production is fundamentally based on a technological package combining no-tillage and the dependence of glyphosate applications to control weeds in transgenic crops (soybean, maize and cotton). Therefore, glyphosate is the most employed herbicide in the country, where 180 to 200 million liters are applied every year. Due to its widespread use, it is important to assess its impact on the environment and, therefore, reliable analytical methods are mandatory. Glyphosate molecule exhibits unique physical and chemical characteristics which difficult its quantification, especially in soils with high organic matter content, such as the central eastern Argentine soils, where strong interferences are normally observed. The objective of this work was to compare two methods for extraction and quantification of glyphosate and AMPA in samples of 8 representative soils of Argentina. The first analytical method (method 1) was based on the use of phosphate buffer as extracting solution and dichloromethane to minimize matrix organic content. In the second method (method 2), potassium hydroxide was used to extract the analytes followed by a clean-up step using solid phase extraction (SPE) to minimize strong interferences. Sensitivity, recoveries, matrix effects and robustness were evaluated. Both methodologies involved a derivatization with 9-fluorenyl-methyl-chloroformate (FMOC) in borate buffer and detection based on ultra-high-pressure liquid chromatography coupled to tandem mass spectrometry (UHPLC-MS/MS). Recoveries obtained from soil samples spiked at 0.1 and 1 mg kg⁻¹ and were satisfactory in both methods (70% – 120%). However, there was a remarkable difference regarding the matrix effect, being the SPE clean-up step (method 2) insufficient to remove the interferences. Whereas the dilution and the clean-up with dichloromethane (method 1) were more effective minimizing the ionic suppression. Moreover, method 1 had fewer steps in the protocol of sample processing than method 2. This can be highly valuable in the routine lab work due to the reduction of potential undesired errors such as the loss of analyte or sample contamination. In addition, the substitution of SPE by another alternative involved a considerable reduction of analytical costs in method 1. We conclude that method 1 seemed to be simpler and cheaper than method 2, as well as reliable to quantify glyphosate in Argentinean soils. We hope that this experience can be useful to simplify the protocols of glyphosate quantification and contribute to the understanding of the fate of this herbicide in the environment.