



DEVELOPMENT AND VALIDATION OF METHOD FOR DETERMINATION OF THE HERBICIDE GLYPHOSATE AND ITS METABOLITE AMPA IN WATER OF AGRICULTURAL REGIONS WITH UPLC-MS/MS

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INTRODUCTION: Brazil uses pesticides on a large scale in agriculture. Among the most widely used is glyphosate. Despite protecting crops from weeds, it is known that they cause harm to human health and the environment. The detection of glyphosate is important for understanding the levels present in nature, which can affect flora and fauna. **OBJECTIVE:** Develop and validate a method to detect and quantify glyphosate and its metabolite AMPA in water samples. **MATERIALS AND METHODS:** For the analysis, two methods were employed: derivatization with pentafluorobenzyl bromide (PFBr) and liquid-liquid extraction using methyl tert-butyl ether (MTBE). Sample preparation involved the use of 500 µl of water, 20 µl of internal standard, derivatization with PFBr and, after four hours incubation, liquid-liquid extraction was performed. The analyses were performed on an Acquity I-Class ultra-performance liquid chromatograph, coupled to a XEVO TQ-S Micro triple quadrupole mass spectrometer (UPLC-MS/MS). The method was validated according to FDA (2022), Eurachem (2014) and SANTE (2021) parameters and precision, accuracy, linearity, limit of quantification, matrix effect and stability tests were performed. After the validation, ten water samples from agricultural properties in the city of Rolante-RS were tested. **RESULTS AND CONCLUSIONS:** all the results in the validation were within recommended levels. The assay was linear between 0.1-25 µg L⁻¹ for glyphosate e 0.5-50 µg L⁻¹ for AMPA, precision ranged from 1.09% to 10.79% (%CV) for glyphosate and 0.77% to 11.59 (%CV) for AMPA. Accuracy ranged from 100.22% to 102.46% and 96.87% to 101.01%, for glyphosate and AMPA, respectively, with the matrix effect compensated by the internal standard. In addition, both analytes demonstrated stability for 12 hours in the autosampler. Glyphosate and AMPA were not quantifiable in any of the tested samples, however, they were collected during a period of low pesticide use. The method was validated and proved to be efficient in detecting and quantifying the herbicide and its metabolite. Nevertheless, additional samples will be collected to apply the method to a larger number of analyses and at different times during glyphosate application.

Keywords: Glyphosate; AMPA; Validation.