



Assessment of temporal distribution of pesticide residues in vineyard soils of La Rioja (Spain)

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The use and application of pesticides in vineyard is a common practice, which is important to prevent pest and diseases and improve the crop health and production, but on the other hand it could involve a potential risk for humans and the environment. For this reason, it is important to develop and validate a simple and fast multiresidue method to determine the presence of these compounds in soils. La Rioja region (Spain) is one of the most important wine-growing regions in Spain, which also entails that could be an important area of pesticide pollution. The objective of this work is to assess the temporal distribution of the possible pesticide pollution in soils from different areas of La Rioja (Spain).

The pesticides selected in this study included fungicides (metalaxyl, and its metabolite CGA62826, pyrimethanil, tebuconazole, myclobutanil, kresoxim-methyl, triadimenol and flutriafol); herbicides (fluometuron, terbuthylazine and its metabolites desethylterbuthylazine and hydroxyterbuthylazine, lenacil, ethofumesate and acetochlor) and insecticides (methoxyfenozide and pirimicarb). The pesticide residues were evaluated by two analytical techniques, gas chromatography and liquid chromatography (GC-MS and LC-MS). The extraction procedure of pesticides from soils was optimized using two soil samples (blank soils) with different texture and characteristics collected from areas without pesticide application. Recoveries were studied in soil samples fortified with all pesticides at two levels of concentrations (the agronomic dose, 0.1 mg kg⁻¹, and ten times this dose, 1 mg kg⁻¹). Different extraction solvents were tested. The best results were obtained with methanol:acetone (50:50) mixture or methanol:CaCl₂ 0.01 M (50:50) mixture for hydroxyterbuthylazine and CGA62826.

The accuracy (average recovery) and precision (reproducibility and repeatability) of the method were assessed using six replicates and the limits of detection (LODs) and quantification (LOQs) were estimated. Recoveries were above 70% for all pesticides. Good linear relationships of the calibration curves (0.01-1 µg mL⁻¹) were obtained for all the compounds by the two analytical methods with regression coefficients (r²) higher than 0.99 in the range of concentrations studied. Detection limits were < 2 ng g⁻¹ for pesticides studied.

After the development and validation of an analytical method, the study of the temporal variability of the pesticide pollution in different periods of time (September 2011, and March, June and October 2012) were performed. Herbicide residues were found in most of the soils and an increment in concentrations of these compounds and in the number of positive samples were detected in samples collected in March 2012 in relation to those collected in September 2011. This increase in the number of positive samples was of 30 % for terbuthylazine. Fungicide residues were detected in greater concentrations in samples collected in September. Both behaviours may be related with the time of application of both types of compounds. Herbicides are commonly applied in March-April and fungicides are usually applied in summer, when different grapevine diseases like botrytis or mildiu commonly appear. Moreover, new pesticide residues were detected in March 2012, which were not detected in September 2011 in any soil sample. Further analysis of results is in course.