Multiresidue method for the determination of 13 pesticides in three environmental matrices: water, sediments and fish muscle

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A B S T R A C T
Pesticide residues in aquatic ecosystems are an environmental concern which requires efficient analytical methods. In this study, we proposed a generic method for the quantification of 13 pesticides (azoxystrobin, clomazone, diflufenican, dimethachlor, carbenazim, iprodion, isoproturon, mesosulfuron-methyl, metazachlor, napropamid, quizalofop and thifensulfuron-methyl) in three environmental matrices. Pesticides from water were extracted using a solid phase extraction system and a single solid–liquid extraction method was optimized for sediment and fish muscle, followed by a unique analysis by liquid chromatography coupled with tandem mass spectrometry (LC–MS/MS). Limits of quantification were below 5 ng L\(^{-1}\) for water (except for fluroxypyr and iprodion) and ranged between 0.1 ng g\(^{-1}\) and 57.7 ng g\(^{-1}\) for sediments and regarding fish, were below 1 ng g\(^{-1}\) for 8 molecules and were determined between 5 and 49 ng g\(^{-1}\) for the 5 other compounds. This method was finally used as a new routine practice for environmental research.

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1. Introduction

The widespread use of pesticides for agricultural activities represents thousands of molecules with a large range of physicochemical properties. It results in the contamination of numerous aquatic ecosystems, including water, sediments and biota [1–3]. A wide spectrum of pesticides can be found in environmental matrices. Some of them, often combined in a mixture, can impact fish health [4,5] or accumulate in edible tissue of fish, with a potential risk for humans [6,7]. However, a lot of residues of pesticides are not investigated in the different environmental matrices (water, sediment and fish) and, so, their levels and behavior in the environment are poorly understood.

Indeed, if numerous analytical methods deal with pesticides analysis in water [8], few of them report the simultaneous analysis of traces of pesticides in sediment and biota [9,10]. Moreover, when bioaccumulation of organic contaminants in fish is documented, the studies focus on organohalogens [11–14], but there is still a lack of data concerning more recent pesticides.

The analysis of traces of organic pollutants in such complex matrices should include an effective sample preparation allowing high recoveries of the analytes while minimizing the presence of interferences. This preparation step should be followed by an analytical method that is sufficiently sensitive and selective to quantify compounds in trace amounts.

Gas chromatography (GC) and liquid chromatography (LC) coupled to mass spectrometry (MS) are the most used techniques for monitoring pesticides residues in food and environmental matrices [15,16]. Due to low volatility of many compounds, GC–MS requires a derivatization before analysis [17]. Consequently, LC–MS is the most frequent choice to explore a large range of low volatile and medium-high polar compounds as pesticides [18]. The choice of the extraction method depends on the type of matrix. Finding a simple and easy method to extract compounds with very different physicochemical properties remains an analytical challenge. Pesticides from water are generally extracted by a solid-phase extraction (SPE), offering good recoveries for a large range of compounds [2,19]. For solid matrices, various methods are used. SPE is also applied as purification step but requires a long sample preparation [20]. Consequently quicker methods, such as solid–liquid extraction and the “QuEChERS” (Quick Easy Cheap Effective Rugged and Safe) methods are preferred to extract compounds from fish muscle [21,22]. Considering sediment or soil matrices, accelerated solvent extraction [23–25] and micro-wave assisted extraction [26] systems are usually performed.

In this context, the aim of this work is the development of a multi-residue method for the simultaneous analysis of a list of 13 pesticides, concerning a large range of physicochemical properties. It results in a contamination of numerous aquatic ecosystems, including water, sediments and biota [1–3].

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