Nanostructured-solvent for the extraction of ochratoxin A in Andalusian wines

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Ochratoxin A (OTA) is a mycotoxin produced by several species of fungi, including Aspergillus and Penicillium. Depending on both environmental and manufacture conditions, OTA occurs at different concentration levels in various foodstuffs and beverages (e.g. cereals, coffee, wine, cocoa, spices, etc). According to a 2002 report on the assessment of dietary intake of OTA by European people, wine resulted in one of the main dietary source (10-20%)[17]. Because of the long half-life of OTA in the organism, its non-reversible disturbances in kidneys, its possible carcinogenic effect and its immunosuppressive and neurotoxic properties, there is a growing need to monitor OTA in food samples, including wine, for which the EU has set a maximum level at 2.0 μg/l.

Analytical methods currently available for OTA determination in wines consist of a concentration/clean-up step followed by reverse-phase LC separation and fluorescence detection or in some cases mass spectrometry with electrospray ionization. The most frequently used concentration/clean-up techniques are solid-phase extraction (SPE) and liquid-liquid extraction (LLE). For LLE techniques, the most important disadvantages are the high organic solvent consumption and the poor recoveries obtained. With regard to SPE, inmunosorbents or inmunoaffinity columns (IAC), which offers high selectivity, are the sorbents more extensively used. In fact, IAC is recommended by the Office International de la Vigne et du Vin (OIV) and the Association of Official Analytical Chemists (AOAC). However inmunosorbents are not recyclable, have a limited storage time (commonly 12 months) and, in some cases, show cross-reactivity with Ochratoxin C.

This research deals with the use of coacervates, colloid-rich liquids, for the extraction/concentration of OTA from white, rosé and red wines, prior to LC/fluorescence quantitation. Water-induced coacervates made up of reverse micelles of decanoic acid in tetrahydrofuran (THF) were used for this application. Extraction of OTA was carried out at pH 2 and it was based on both hydrophobic and hydrogen bonds analyte:extractant interactions. Parametres affecting extaction efficiency and concentration factors were studied. Concentration of decanoic acid and THF were the most influential parametres, being 0.5% of acid and 5% of THF the selected concentrations. The procedure was very robust, so that the extractions were not significantly influenced by the pH and the nature or concentration of the matrix components. Recoveries of the target compounds ranged between 80% and 94% and the concentration factors varied from 128 to 150 for sample volumes of 19 mL. The precision of the method expressed as relative standard deviation was about 5% and the detection limit was 4.5 ngL^{-1} in white and rosé wines and 15 ngL^{-1} in red wines, values which are far below the maximun level established for OTA by EU directives[18]. This method offers a simple, cheap and rapid alternative for the usually tedious and time-consuming wine pre-treatment. The approach developed was applied to the determination of OTA in different wine samples from south of Spain.

[17] Scientific Cooperation (SCOOP) Task Report 3.2.7, Assessment of dietary intake of Ochratoxin A by the population of EU Member States, 2002, http://europa.eu.int/comm/food/fs/scoop/index en.html.
[18] Commission Regulation (EC) no 123/2005 of 26 January 2005 amending regulation (EC) no 466/2001 as regards ochratoxin A, Off. J. Eur. Union L25 (2005) 3.