Validation of a SPE clean-up method for ochratoxin A determination in red wine

Ana Luísa De Sousa [1]; Armando Venâncio [1]; Luís Abrunhosa [1]

ABSTRACT

The quality of Portuguese wines has improved considerably during the last decades as modern viticulture and enological practices were adopted. To maintain high quality standards it is also important to control any hazard that may jeopardize wine safety. One potential hazard for wines is the occurrence of the mycotoxin, ochratoxin A (OTA). This fungal metabolite occurs and exerts its toxic effects in small quantities, thus sensitive and reliable methods are required for monitoring its occurrence in foods. In order to minimize the interfering effect of the matrix and improve the selectivity and sensitivity of the analytical method, a concentration and cleaning step is often necessary. Solid phase extraction (SPE) is a technique with numerous advantages for that purpose. The objective of this work was to optimize and validate an analytical method for the determination of OTA in red wine using the SPE column Strata-X-A (Phenomenex). A not contaminated local red wine was fortified with OTA at concentrations of 0.05 to 10 µg/L, samples were clean-up using Strata-X-A columns in triplicate, analyzed by HPLC with fluorescence detection, and method recoveries, selectivity, stability, linearity, limit of detection (LOD), and limit of quantification (LOQ) determined. The method showed a linear response within the concentration range of 0.05 to 10 µg/L with a correlation coefficient of 0.9999. Within this concentration range, recoveries varied between 111% and 87%, respectively. The intra-day RSD was below 8%. The LOD and LOQ of the method was 0.005 and 0.015 µg/L, respectively. Portuguese red wines were analyzed using this method. Four wines did not reveal any OTA, and seven add OTA in concentrations that ranged between 0.02 and 0.441 µg/L. In conclusion, 20 years after the first studies reporting the presence of OTA in wines, levels of this mycotoxin in Portuguese wines are still low.

Keywords: Ochratoxin A, wine, solid-phase extraction

1 - CEB - Centre of Biological Engineering, University of Minho, 4710-057 Braga, Portugal