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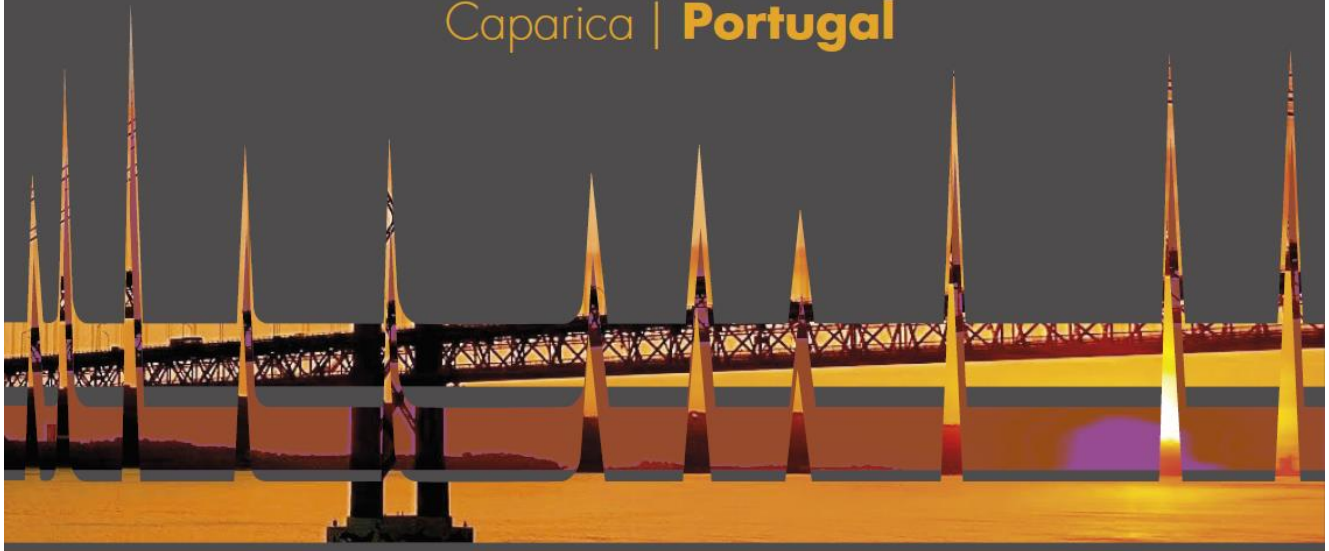

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Optimization and validation of two methods to determine the levels of AFM1 in milk and cheese samples using immunoaffinity columns for extraction and HPLC-FLD for quantification

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Consumption of dairy products has expanded rapidly over the past decade and constitutes an important source of dietary protein. [1] Aflatoxin M1 (AFM1) is a potent carcinogen metabolite that can be present in milk from dairy cows that consume feed contaminated with Aflatoxin B1. Even though it is less toxic than its parent compound, AFM1 is hepatotoxic and carcinogenic, and is stable during milk pasteurization, storage and preparation of various dairy products. [2,3] Due to the toxicity of this molecule, its detection and quantification is extremely important.

The objective of this work was to optimize and validate two methods, according to Commission Regulation (EC) nº 401/2006 of 23 February, to determine the levels of AFM1 in milk and in cheese, using immunoaffinity columns (IAC) for extraction and HPLC with fluorescence detection for quantification.[4]

The method for milk samples was adapted from VICAM – the supplier of the IAC, and for cheese samples was from r-biopharm and VICAM.[5,6] For both methodologies, three levels of spiking in triplicate on two different days were performed. The calibration curve was linear from 0.047 to 4.7 μg L^{-1} and the detection and quantification limits for milk and cheese were 0.001 μg L^{-1} and 0.003 μg L^{-1}, and 0.006 and 0.02 μg kg^{-1}, respectively.

For milk samples, average recoveries determined at spiking levels of 0.020, 0.050 and 0.10 μg L^{-1} were in the range of 62 % – 87 %, with intra-day precision (RSD_r) in the range of 3.4 % – 9.5 %, and inter-day precision (RSD_r) in the range of 5.4 % – 6.2 %.

For cheese samples, average recoveries determined at spiking levels of 0.050, 0.10 and 0.25 μg L^{-1} were in the range of 54 % – 78 %, with intra-day precision (RSD_r) in the range of 2.8 % – 8.7 %, and inter-day precision (RSD_r) in the range of 3.7 % – 6.2 %.

Results of the validation process indicate that, except for the recovery in cheese samples with spiking level of 0.25 μg L^{-1}, both methods are in agree with the provisions of Commission Regulation (EC) nº 401/2006.

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