FOOD ANALYSIS

Method optimization for simultaneous analysis of aflatoxin M1, avermectins and organophosphate pesticides in bovine milk by UPLC®-MSMS

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The milk and dairy products are the main dietary sources of calcium, and its quality is not only related to the nutritional attributes, but, above all, its harmlessness. The aim of this work was optimize an analytical methodology of extraction, detection and quantification to monitor simultaneously aflatoxin M1, avermectins and organophosphate pesticides in bovine milk. To optimize the extraction procedure the recovery was evaluated for two QuEChERS [1] methods: original and acetate buffer with three different types of dispersive solid phase extraction: $MgSO_4$ and PSA; $MgSO_4$ and C18; $MgSO_4$, PSA and C18. For each method fortifications were made at three levels: 0.5, 1, 0 and 1.5 times the MRL of each analyte, totalizing 18 experiments. The extracts were injected in system Acquity UPLC® Quattro Premier XE® operated with ESI source in the multiple reaction monitoring (MRM) in positive mode, scanning two fragmentation reactions per analyte. The chromatographic separation was performed on Waters Acquity UPLC BEH®C18 column with gradient elution: mobile phase A (aqueous 5mM ammonium formate + 0.1 % formic acid) and mobile phase B (acetonitrile: mobile phase A, 95:5): 0-1min. (10% B) 5 to 9.5 min. (100% B), 10min. (10% B) at a flow rate of 0.2 mL/min., injection volume: 10µL. External standardization was used to determine the concentration of following analytes in milk: abamectin, acephate, aflatoxin M1, azinphos ethyl, azinphos methyl, chlorpyrifos methyl, diazinon, doramectin, eprinomectin, ivermectin, methamidophos, methidathion, mevinphos, moxidectin, pirimiphos-ethyl and pirimiphos-methyl. The best result was obtained with the original method using d-SPE over $MgSO_4$, PSA and C18 with recoveries between 80 and 110% for all analytes. These values are within the recommended concentrations above 10µg/kg [2], showing that the analytical method is suitable for the determination of these analytes in milk.

[1] LEHOTAY, S.J.; MASTOVSKA, K.; LIGHTFIELD, A.R. *J. AOAC Int*. **2005,** 88, 615–629.
[2] BRASIL. Ministério da Agricultura Pecuária e Abastecimento. **Manual de garantia da qualidade analítica.** Ministério da Agricultura Pecuária e Abastecimento. Secretaria de Defesa Agropecuária. Brasília : MAPA/ACS, 2011. 227 p.