Microextraction of sulphonamide antibiotic residues in meat using supramolecular solvents

E.M. Costi, M.D. Sicilia and S. Rubio
Departamento de Química Analítica
Universidad de Córdoba
Edificio Anexo Marie Curie
coloe22@hotmail.com *y www.uco.es/investiga/grupos/FQM-186*

Quantifying sulphonamide antibiotic (SA) residues in meet is a difficult task owing to the complexity of the samples, the polarity of the analytes and the low concentrations at which they should be determined (below 0.1 mg Kg^{-1}, the maximum residue limit permitted for SAs in Europe and the USA). Reported procedures for the extraction of SA residues from edible animal tissues are lengthy, involve numerous reextraction and cleanup steps, consume large amounts of toxic solvents and/or need the use of equipments no usually available in laboratories. Beside, recoveries obtained frequently are low and/or dependent on the food matrix composition. The quantification of SAs in the extracts is frequently performed by liquid chromatography (LC) with fluorescence (FL) detection after analyte derivatization. Reported LC/mass spectrometry-based methods also provide the sensitivity required for the determination of SA residues in meet but matrix-matched external calibration is required in order to avoid interferences due to matrix components.

This research deals with the evaluation of a supramolecular solvent made up of reverse micelles of decanoic acid (DeA) dispersed in a continuous water:tetrahydrofuran (THF) phase for the extraction of eight SAs (sulfadiazine, sulfamerazine, sulfamethoxypyridazine, sulfachloropyridazine, sulfadoxine, sulfamethoxazole, sulfadimethoxine and sulfaquinoxaline) from meat samples prior to their determination by LC/FLU. SAs were extracted from 200 mg-samples using 1 mL of supramolecular solvent and directly determined in the extract after derivatization with fluorescamine. SAs were effectively extract from meat using the optimized supramolecular solvent-based microextraction (SUSME) approach independent on the analyte polarity and the composition of sample matrices. The capability of the DeA reverse micelle-based solvent to extract highly polar analytes [octanol-water partition coefficients (log K_{ow}) of SAs ranged between -0.09 and 1.68] can be explained on the basis of the different mechanisms that it provides for analyte solubilisation, i.e. hydrophobic interactions and hydrogen bonds formation, and the high number of solubilisation sites that in it exists. Quantitation limits for the determination of SAs using the proposed SUSME/LC/FLU method were in the interval 0.02-0.06 mg Kg^{-1} and the precision, expressed as relative standard deviation ([SAs]= 0.1 mg Kg^{-1}, n =11) ranged between 2.7 and 3.9%. Recoveries obtained by applying this approach to the analysis of pork, beef, chicken, turkey and lamb samples fortified at the μg Kg^{-1} were in the interval 98-109%.