MAGNETIC PARTICLE SPRAY MASS SPECTROMETER USING RESTRICTED ACCESS MAGNETIC COPOLYMER FOR THE EXTRACTION OF TETRACYCLINE RESIDUES IN MILK SAMPLES

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INTRODUCTION: Tetracyclines are broad-spectrum antibiotics and are the first choice in veterinary and agricultural medicine for treatment, prophylaxis and weight gain in animals. Rapid quantification of tetracyclines at low concentrations in foods of animal origin is necessary, such as in milk. Magnetic particle spray mass spectrometry (MPS-MS) is a new rapid and sensitive analytical technique, recently developed by our research group, in which analytes are extracted by solid-phase extraction by magnetic dispersion, followed by direct analysis of the extracted molecules by their direct desorption at the inlet of the mass spectrometer, using a solvent and high voltage. OBJECTIVE: To synthesize and characterize a magnetic restricted access copolymer (M-RACP) to be used in the extraction of tetracycline, oxytetracycline and chlortetracycline from milk samples, followed by analysis with MPS-MS. MATERIALS AND METHODS: The synthesis was carried out in four stages: formation of Fe_3O_4; coating with tetraethylorthosilicate and 3-(trimethoxysilyl)propyl methacrylate; polymerization; coating of the polymer with bovine serum albumin (BSA). For characterization, infrared spectroscopy (IR), thermogravimetric analysis (TG), differential scanning calorimetry (DSC), protein exclusion, kinetics and isothermal adsorption tests were performed. The MPS-MS conditions were optimized and the method was duly validated for the analysis of tetracyclines. RESULTS AND CONCLUSION: About the characterization, the IR spectrum showed stretching bands, referring to the chemical bonds established during synthesis. TG and DSC showed thermal events to confirm the synthesis steps. Protein exclusion confirmed restricted access. In the adsorption kinetics and isotherms, the equilibrium reached after 1 min and the maximum adsorption amount was 104,5 mg. g^{-1}. The calibration curves were built from 50 to 450 µg. L^{-1}; with R^2>0,99; LOD from 30.7 to 46.1; LOQ=50 µg. L^{-1}. Precision and accuracy were adequate for low, medium and high points and no molecules were detected in the actual samples. It was the first time that the MPS-MS method was used for food analysis, being fast, low cost, sustainable, with good linearity, precision and accuracy.

KEYWORDS: magnetic particle spray mass spectrometry, intelligent materials; tetracyclines; solid phase extraction.
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