DETERMINATION OF BETA-BLOCKERS MAGNETIC PARTICLE SPRAY MASS SPECTROMETRY

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INTRODUCTION: Beta-blockers are widely prescribed for cardiovascular diseases, but they are also used as doping agents in sports of precision. Therefore, developing a simple and sensitive method for their detection is of great interest. Magnetic particle spray mass spectrometry (MPS-MS), recently developed by our research group, is an ambient ionization technique that allows direct quantification of analytes extracted via magnetic dispersive solid-phase extraction (d-SPE). In this approach, desorption and ionization occur directly from magnetic particles introduced into the MS. The use of magnetic molecularly imprinted polymers (M-MIP) is advantageous for MPS-MS providing selectivity for the analytes and eliminating matrix interferents. OBJECTIVE: This study proposed, for the first time, to combine MPS-MS and M-MIP, and the determination of atenolol, metoprolol, labetalol, propranolol, nadolol, and pindolol in human plasma. MATERIALS AND METHODS: M-MIP was synthesized, characterized, and used in MPS-MS. A magnetic d-SPE was performed for 20 min. Then, the M-MIP was attached to the tip of a metal probe and inserted into the MS inlet. A solvent was dispensed onto the particles, enabling efficient desorption/ionization of the analytes. Detection was carried out using a triple quadrupole MS. The method was optimized and validated. RESULTS: Characterization confirmed magnetic nanoparticles functionalization and polymerization. The M-MIP showed high selectivity to beta-blockers over non-analog molecules. Elovich and Sips models best fit the adsorption kinetic and isotherm, respectively. Adsorption equilibrium was reached in 10 min, with a qe_{max} of 36.08 mg g^{-1}. Calibration curves were linear from 3 to 80 μg L^{-1} for all analytes. Precision, accuracy, and limit of detection ranged from 3.95 to 21.20%, 17.05 to 18.93%, and 0.64 to 2.41 μg L^{-1}, respectively. The detection limits were 3 μg L^{-1} for all analytes. CONCLUSION: MPS-MS is a fast (54 analyses per h), simple, and sensitive technique, that does not require chromatographic separation and exhibits a minimal ionic suppression. It requires only 100 μL of sample, 75 μL of solvent, and 0.5 mg of sorbent per analysis cycle, making it highly suitable for doping control and routine analyses, besides being fully aligned with green chemistry principles.

Keywords: ambient ionization; molecularly imprinted polymers; dispersive solid phase extraction; magnetic sorbent