

Dispersive solid-phase extraction with a new Fe₃O₄:Eu,Tb nanocomposite as sorbent for antibiotic determination

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Abstract

A novel Fe₃O₄:Eu,Tb nanocomposite has been synthesized for its use as sorbent in dispersive solid phase extraction, which has been applied to the determination of quinolone and tetracycline antibiotics in meat samples. These nanocomposites have been synthesized [1] in two simple steps, which consist in the formation of the magnetic core by a co-precipitation method and a further treatment with Eu(III) and Tb(III) salts obtaining magnetic nanomaterial with these lanthanide ions in its surface. The magnetic properties of these hybrid nanoparticles ease the performance of dispersive solid phase extraction and the presence of Eu(III) and Tb(III) in their composition allows a relatively selective interaction with antibiotics bearing β -diketonate and carboxylic acid groups, such as quinolones and tetracyclines, which have been chosen as model analytes. Oxolinic acid (OXO), nalidixic acid (NAL), flumequine (FLU), oxytetracycline (OTC), tetracycline (TET) and chlortetracycline (CTC) have been simultaneously determined using ultra high performance liquid chromatography with fluorometric determination in less than 5 min. Chromatograms were simultaneously detected at two excitation/emission pairs of wavelengths, which are 255/360 and 390/512 nm for quinolones and tetracyclines, respectively.

Under the optimum conditions, the dynamic ranges and detection limits for the analytes were 0.5 – 2000 and 0.25 ng mL⁻¹ for OXO; 1.5 – 2000 and 0.7 ng mL⁻¹ for NAL; 2.5 – 2000 and 1.2 ng mL⁻¹ for FLU; 2 – 7500 and 1 ng mL⁻¹ for OTC; 3 – 7500 and 1.5 ng mL⁻¹ for TET; and finally, 10 – 1000 and 3.8 ng mL⁻¹ for CTC. Intra- and inter-day precision data were assessed for retention times and areas at two concentration levels of each antibiotic, yielding values in the range of 0.07 – 0.21% and 2.6 – 9.1% for retention times and areas, respectively, in intra-assay precision experiments, and 0.5 – 1.8% and 4.2 – 14.4% for retention times and areas obtained after inter-assay experiments, respectively. The method was applied to the analysis of different meat samples, such as chicken and pork muscle, which were spiked at 50, 100 and 200 μ g kg⁻¹, giving mean recovery values for each analyte in the range of 79.1 – 91.8%. These results prove the usefulness of the developed method to the control of these antibiotic residues in meat samples.

References

[1] Ma, Z.Y.; Dosev, D.; Nichkova M.; Gee, S.J.; Hammock, B.D.; Kennedy, I.M. *J. Mat. Chem.*, **2009**, 19, 4695.