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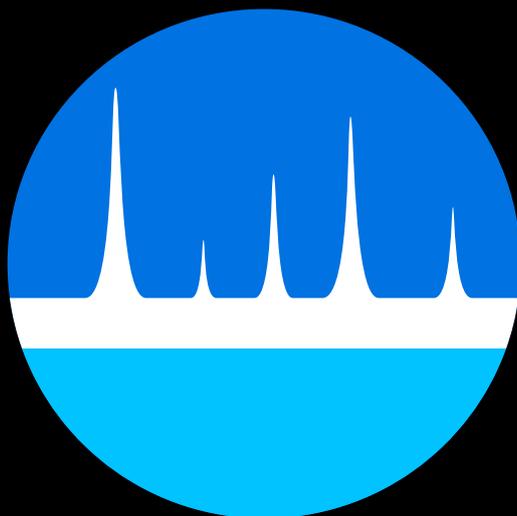
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LIVRO DE RESUMOS
BOOK OF ABSTRACTS

Development and application of a microextraction by packed sorbent method with ionic liquid-grafted silica–graphene oxide for multiclass pesticides in Spanish wines

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Monitoring pesticide residues in wine is essential to ensure food safety, as these compounds and their metabolites can persist in the final product and pose health risks. In this study, a hybrid sorbent was developed by anchoring graphene oxide onto amino-silica particles (GO@Sil) and functionalizing them with ionic liquids (ILs) via direct anion exchange. Among the tested materials, GO@Sil-[VHIm]$^+$PF$_6^-$ showed the best extraction performance due to its multiple interaction mechanisms with the analytes. This sorbent was integrated into a microextraction by packed sorbent (MEPS) system for the determination of six multiclass pesticides (carbendazim, thiodicarb, carbofuran, carbaryl, atrazine, and terbuthylazine) in wines, followed by HPLC-MS/MS analysis. Extraction conditions were optimized using univariate and multivariate approaches, establishing the following protocol: 3 mg of sorbent packed into a 1 mL polypropylene syringe, six extraction cycles of 1 mL wine (draw/eject), one washing cycle with ultrapure water to remove polar interferents, six desorption cycles with 200 µL of acetonitrile:methanol (50:50, v/v), and sorbent regeneration with 500 µL of acetonitrile to prevent carryover. The validated method provided excellent linearity ($r^2 \geq 0.9958$), satisfactory precision (RSD < 15%), and recoveries between 49% and 112%. Limits of detection ranged from 0.030 to 0.130 ng mL^{-1}, with negligible matrix effects in white, red, and rosé wines. The sorbent could be reused for at least six extractions, and batch-to-batch reproducibility met acceptance criteria (all RSDs < 20%), confirming the reliability of the synthesis. Additionally, the method demonstrated notable green advantages, including device reusability and low solvent consumption (0.7 mL per analysis). Sustainability assessments with AGREEprep and BAGI yielded favorable scores (0.52 and 57.5, respectively). Overall, this study introduces a promising and environmentally friendly analytical approach for multiclass pesticide monitoring in wines, with strong potential for routine application.