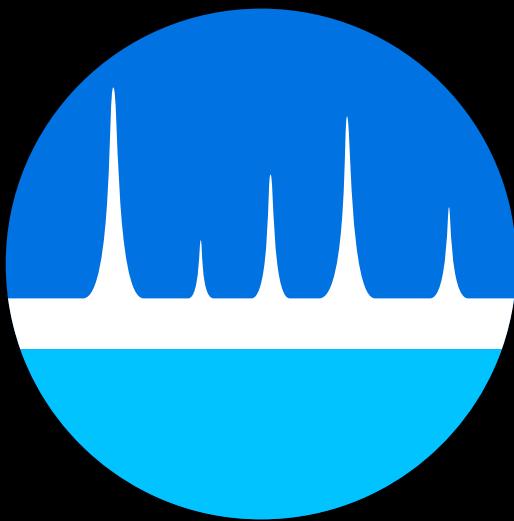


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GRAPHENE-CHITOSAN BIOSORBENT: AN ENVIRONMENTAL MICROEXTRACTION APPROACH FOR THE ASSESSMENT OF PESTICIDES AND ANTIBIOTICS IN FOOD MATRICES

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Advances in agricultural and veterinary practices have introduced pesticides and antibiotics into food commodities, raising concerns about their harmful effects on human health. Consequently, innovative microextraction protocols have been developed to enable accurate trace-level analysis through effective clean-up and preconcentration. In this context, microextraction by packed sorbent (MEPS) has demonstrated great potential for the analysis of challenging matrices. Moreover, with the advent of automation, researchers have overcome the limitations of previous laborious MEPS procedures, expanding its applicability. Additionally, the development of bio-based sorbents aligns with the principles of green analytical chemistry (GAC) by promoting the use of enhanced biodegradable materials as alternatives to conventional sorbent phases. In this study, silica-graphene oxide@chitosan (SiGO@CS) was synthesized via an in-situ reaction and employed for MEPS extraction of atrazine and thiamethoxam from corn and tomato samples, respectively, as well as ceftiofur and sulfonamide from milk samples. Characterization assays confirmed the successful modification of the SiGO@CS. Method optimization identified draw/eject and washing cycles as the most significant parameters for pesticide extraction from corn and tomato, while washing cycles were critical for sulfonamide extraction from milk. The method achieved limits of detection (LOD) and quantification (LOQ) ranging from 0.020 to 0.045 $\mu\text{g L}^{-1}$ and 0.045 to 1.0 $\mu\text{g L}^{-1}$ for pesticides, and from 5 to 15 $\mu\text{g L}^{-1}$ and 15 to 20 $\mu\text{g L}^{-1}$ for antibiotics, respectively, with recoveries between 82% and 109%. The manual MEPS protocol was successfully adapted to an automated lab-made platform, offering insights into the balance between analytical performance and environmental impact. Application to regional samples revealed thiamethoxam and atrazine at levels exceeding the recommended daily intake in one tomato and one corn sample. Overall, the use of the novel SiGO@CS biosorbent demonstrated a green, high-performance strategy, reinforcing the potential of bio-based sorbents for both manual and automated microextraction.

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