

A green and simple sample preparation method to determine pesticides in rice using a combination of SPME and rotating disk sorption devices

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In this study, a novel environmentally-friendly approach was developed to overcome certain limitations in the analysis of solid food samples. An experimental setup based on solid-phase microextraction apparatus coupled to a rotating disk device is proposed for the determination of polar and slightly-polar pesticides including carbofuran, molinate, atrazine, simazine and tebuconazole in rice samples. In this innovative procedure, a rice sample is inserted into the cavity of the rotating disk device which is then immersed in an aqueous solution followed by high-speed agitation. In this case, water is employed as a green solvent for leaching the analytes from the sample matrix. Simultaneously, a solid-phase microextraction fiber comprised of divinylbenzene/carboxen/polydimethylsiloxane (2 cm length and 50/30 μm film thickness) was immersed in the aqueous solution to extract the slightly polar analytes from the aqueous matrix with subsequent thermal desorption in the injector of a gas chromatograph-mass spectrometer instrument. The optimal extraction conditions were obtained using an extraction temperature of 80 $^{\circ}\text{C}$ for 40 min, with 125 mg of rice inserted in the rotating disk. Using the previously optimized extraction conditions, the analytical performance was satisfactory with correlation coefficients higher than 0.9881 for all analytes, limits of detection ranging from 0.46 to 5.9 ng g^{-1} , limits of quantification from 1.5 to 19.7 ng g^{-1} , relative recoveries from 76 to 109%, intra-day precision ($n = 3$) from 1.3 to 19%, and inter-day precision ($n = 9$) from 3.5 to 6.5%. The proposed method represents a promising alternative for the analysis of complex solid food samples using SPME, since

the SPME fiber damage is substantially decreased when the contact with the solid matrix is avoided.