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论文

识别三唑类农药的片段印迹聚合物的合成及在固相萃取中的应用

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摘要:

采用片段印迹技术, 合成了一系列对7种三唑类农药(三唑酮、烯唑醇、多效唑、烯效唑、戊唑醇、三唑醇和双苯三唑醇)具有识别能力的聚合物. 振荡平衡吸附实验表明, 以邻硝基苯酚为模板的聚合物(M1)对上述7种农药具有最佳的选择性吸附性能. 根据分析物结构对片段印迹聚合物吸附能力的影响, 提出了片段印迹聚合物的识别机理: 三唑类化合物的分子片段末端苯环进入片段印迹聚合物的孔穴中, 同时其羟基与聚合物孔穴外的功能单体4-乙烯基吡啶上的氮原子形成氢键, 二者的协同作用实现对目标分子的选择性识别, 其中分析物末端苯环和聚合物孔穴的匹配是影响片段聚合物识别能力的主要因素. 将基质固相分散(MSPD)与以M1为吸附剂的分子印迹固相萃取(MISPE)联用, 用于土壤样品的前处理. 在3种添加水平下, 各分析物的回收率均为75%~102%, 相对标准偏差为3%~9%($n=5$), 方法检出限(信噪比等于3)0.9~15 $\mu\text{g}/\text{kg}$. 表明该分析方法结合了MSPD的快速提取和MISPE的高选择性的特点.

关键词: 片段印迹; 三唑类农药; 识别机理; 分子印迹固相萃取; 基质固相分散

Preparation of a Fragment Imprinted Polymer for Recognition of Triazole Pesticides and Its Application to Solid-phase Extraction

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Abstract:

A series of fragment imprinted polymers for selective recognition of seven triazole pesticides (triadimefon, diniconazole, paclobutrazol, uniconazole, tebuconazole, triadimenol and bitertanol) were prepared through fragment imprinting technique, utilizing various benzene derivatives as the alternative-template. Results of batch binding experiments show that the fragment imprinted polymer (M1) with 2-nitrophenol as the template exhibit the highest recognition ability for the triazoles. In light of the effects of the structures of the triazoles on the adsorption capacity of the fragment imprinted polymer, the recognition mechanism is proposed as follows: the terminal phenyl group of the triazoles enters a cavity in the fragment imprinted polymer, while the triazoles' hydroxyl standing outside the cavity forms hydrogen bond between with the nitrogen atom of the functional monomer 4-vinylpyridine standing outside the cavity; the former is the key factor affecting the recognition of the fragment imprinted polymer. Finally, the polymer(M1) was used as the sorbent of solid-phase extraction. For the molecularly imprinted solid-phase extraction(MISPE) procedure, the loading, washing and elution conditions were optimized. The optimized MISPE procedure was applied to the clean-up of the matrix solid-phase dispersion(MSPD) extracts from soil samples for the determination of the above-mentioned triazole pesticides. The clean-up of MISPE was proved. Recoveries of MSPD-MISPE extracts from soil samples spiked at three levels were 75%—102%, with good precision(RSD=3%—9%, $n=5$). The lowest limits of detection(the ratio of signal to noise=3) ranged from 0.9—15 $\mu\text{g}/\text{kg}$. This study highlights the potential of the novel method combining the simplicity of MSPD with the high selectivity of MISPE for extraction of trace compounds from complex matrices.

Keywords: Fragment imprinting; Triazole pesticide; Recognition mechanism; Molecularly imprinted solid-phase extraction; Matrix solid-phase dispersion

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