

整体柱专栏

甲基丙烯酸酯毛细管整体柱分离微囊藻毒素的研究

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摘要 以甲基丙烯酸丁酯为单体,乙二醇二甲基丙烯酸酯为交联剂,在致孔剂存在的条件下原位聚合制备了甲基丙烯酸丁酯毛细管整体柱(150 μm i.d.)。实验中优化了用此整体柱分离3种微囊藻毒素(MC-LR,-YR和-RR)的色谱条件(流动相种类、缓冲溶液浓度、pH、流动相流速),建立了微囊藻毒素的整体柱毛细管液相色谱分离方法,该方法可以在9 min之内实现3种微囊藻毒素的基线分离。将该方法应用于实际水样中微囊藻毒素的分析,成功实现了培养水样和巢湖水样中微囊藻毒素的快速分离,两种样品中均检测到MC-LR。结果表明,所制备的甲基丙烯酸酯毛细管整体柱具有良好的重现性、渗透性,在微囊藻毒素的常规检测中具有很好的应用前景。

关键词 [聚合物整体柱](#) [毛细管高效液相色谱](#) [微囊藻毒素](#)

分类号

Study on Separation of Microcystins Using Polymethacrylate-Based Capillary Monolithic Column

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Abstract

Polymethacrylate-based monolithic column was prepared in 150 μm i.d. capillary column in situ using butylmethacrylate and ethylene dimethacrylate. The application of methacrylate monolithic column in capillary high performance liquid chromatography ($\mu\text{-HPLC}$) for separation of microcystins (MCs) with ultraviolet (UV) detection has been studied. The properties of the monolithic column could be easily tailored by altering the preparation conditions. For the good reproducibility in preparation of monolithic column, this method could be used for the analysis of samples in practical. The effects of composition of mobile phase, pH value, concentration of the buffer, and the flow rate of the mobile phase on the separation of microcystins were investigated. The optimum separation for microcystins, MC-LR, MC-YR, and MC-RR, was achieved with a gradient elution of mobile phase A (0.01 mol/L phosphate buffer, pH 2.5) and mobile phosphate B (acetonitrile). The standard microcystins could be baseline-separated within 9 min. The limits of detection (LODs) (S/N=3) for three standard microcystins were in the range of 0.80-1.03 mg/L. The intra-day and inter-day precisions of the method were obtained with the values of relative standard deviation less than 1.0% and 2.0%, respectively. This method was successfully used to analyze bloom samples and laboratory-cultured samples of cyanobacteria after performing solid phase extraction (SPE) using C18 cartridges for preconcentration. The whole procedure provided low LODs for MCs, e.g. the LOD for MC-LR was found to be 420 ng/L. This family of microcystin is analyzed by $\mu\text{-HPLC}$ using methacrylate monolithic column for the first time. It is shown that this method is promising in the routine analysis of microcystins in water samples in practical.

Key words [polymer monolithic column](#) [capillary high performance liquid chromatography \(\$\mu\text{-HPLC}\$ \)](#) [microcystins](#)

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