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特别策划

亲水作用毛细管整体柱的制备及其用于奶制品中三聚氰胺的加压毛细管电色谱分析

李新燕1, 王彦1, 谷雪1, 陈妍2, 阎超1*

1. 上海交通大学药学院, 上海 200240; 2. 江苏泰州市产品质量监督检验所, 江苏 泰州 225300

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摘要 以甲基丙烯酸丁酯(BMA)和3-［N,N-二甲基-［2-(2-甲基丙-2-烯酰氧基)乙基］铵］丙烷-1-磺酸内盐(SPE)为单体,制备了新型的亲水作用毛细管整体柱,并通过三聚氰胺在此柱上的保留行为证明其具有亲水性。以加压毛细管电色谱(pCEC)技术为平台,优化了整体柱基于亲水作用分离分析奶制品中三聚氰胺的色谱条件。当流动相中乙腈与10 mmol/L磷酸盐缓冲液的体积比为80:20, pH为3.0,电压为3 kV,检测波长为215 nm时,三聚氰胺能获得很好的分离。方法学考察结果表明,合成的亲水整体柱具有良好的重现性和渗透性,建立的pCEC分析方法的检出限为0.05 mg/L。该方法简单方便,回收率较高,而且流动相中无需添加离子对试剂,适合于奶制品中三聚氰胺的定量测定。

关键词 亲水作用毛细管整体柱 制备 加压毛细管电色谱 三聚氰胺 奶制品

Preparation of hydrophilic interaction monolithic column and its application in the analysis of melamine in dairy products using pressurized capillary electrochromatography

LI Xinyan1, WANG Yan1, GU Xue1, CHEN Yan2, YAN Chao1*

1. School of Pharmacy, Shanghai Jiao Tong University, Shanghai 200240, China; 2. Product Quality Supervising and Inspecting Institute of Taizhou, Taizhou 225300, China

Abstract

The hydrophilic monolithic column was prepared in 100 μm i.d. capillary in situ using ethylene dimethacrylate as crosslinker, butyl methacrylate and 3-［dimethyl-［2-(2-methylprop-2-enoyloxy)ethyl］azaniumyl］propane-1-sulfonate as monomers. In the study, the hydrophilic interaction behavior of the monolithic column was demonstrated. The application of the column in pressurized capillary electrochromatography (pCEC) for the separation of melamine with ultraviolet detection has been studied. The effects of the composition of mobile phase, pH value, the concentration of the buffer, the flow rate of pump and voltage were investigated. The optimum separation for melamine was achieved with the mobile phase of 10 mmol/L phosphate buffer (pH 3.0) and acetonitrile (20:80, v/v), voltage at 3 kV and UV detection at 215 nm. The limits of detection (LODs) (S/N=3) for melamine standard was 0.05 mg/L. The method is simple, accurate and works well without using the ion-pairing agent. The powdered milk and liquid milk were analyzed quantitatively by pCEC using the hydrophilic interaction monolithic column. It is shown that this method is promising in the routine analysis of melamine in dairy products.

Key words hydrophilic interaction capillary monolithic column preparation pressurized capillary electrochromatography (pCEC) melamine dairy products

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通讯作者 阎超 chaoyan@unimicrotech.com