

利用毛細管電泳線上濃縮法分析防腐劑與酚酸

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摘 要

毛細管電泳是近年來相當受到重視的分離技術，但毛細管的光徑約為 50~100 μm ，利用紫外光吸收法的偵測，無法有效降低偵測極限，而樣品線上濃縮技術是解決問題的方法之一，此方法不必改變儀器設備，即可增加靈敏度 100~10000 倍，是一種既簡單又方便的技術。其原理是增加毛細管中分析物的進樣量，透過不同的方式，將樣品區間聚集濃縮，以增加分析物的濃度。本研究利用陰離子界面活性劑(SDS)線上濃縮分析防腐劑的最佳分離條件：緩衝溶液為 pH 2.6、25 mM citric acid/disodium hydrogen phosphate，含有 30% 甲醇的有機修飾劑與 150 mM SDS 於緩衝溶液中，此時外加電壓為 -25 kV，樣品以 0.5 psi 注射 240 s，分析物配製於 pH 2.6、75 mM citric acid/disodium hydrogen phosphate 的緩衝溶液中(不含甲醇與 SDS)。與 MEKC 方法比較，利用 sweeping 分離防腐劑可以將偵測極限降低 100 倍，並有良好的線性與再現性。

以上述方法進行更進一步的研究，將掃略式線上濃縮結合大體積樣品堆積分析酚酸，其最佳分離條件如下：緩衝溶液為 pH 2.6、15 mM citric acid/disodium hydrogen phosphate，含有 SDS 180 Mm 於緩衝溶液中，此時外加電壓為 -30 kV，樣品以 5 psi 注射 7 min。掃略式線上濃縮結合大體積樣品堆積與陰離子選擇性全注入結合掃掠式線上濃縮進行比較，雖然陰離子選擇性全注入結合掃掠式線上濃縮其樣品在純水的濃縮效率在 10000 倍以上，但因為利用電壓注入法，故極易受樣品基質的干擾，而掃略式線上濃縮結合大體積樣品堆積因為利用氣壓注入法，較不受樣品基質的干擾，且濃縮效率亦可達到 1000 倍以上，為一種既靈敏又可運用於真實樣品中的偵測方法。

Analysis of Preservatives and Phenolic Acids by On-Line Concentration in Capillary Electrophoresis

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Abstract

Capillary electrophoresis is an important separation technique in these years. However, the inner radius of the capillary is around 50~100 μ m, it is not effective to reduce detection limit with direct ultraviolet detection. It is a good method to reduce detection limit by on-line concentration. The way doesn't need to add any instruments; moreover, it provides much higher sensitivity improvements. On-line concentration of preservatives by sweeping was investigated in this study. The optimized pH 2.6 buffer consisted of 25 mM citric acid/disodium hydrogen phosphate with 150 mM SDS and 30 % methanol. Samples were pressure injected at 0.5 psi for 240 s. This method afforded 100-fold improvement in peak heights for preservatives with adequate linearity and reproducibility.

Another on-line concentration method, large-volume sample stacking-sweeping-micellar electrokinetic chromatography (LVSS-sweeping-MEKC), was employed to analyze phenolic acids. The optimized pH 2.6 buffer consisted of 15 mM citric acid/disodium hydrogen phosphate, 180 mM SDS. Samples were pressure injected at 5 psi for 7 min. Compared with LVSS-sweeping-MEKC, anion-selective exhaustive injection-sweeping-micellar electrokinetic chromatography (ASEI-sweeping-MEKC) afforded 10000-fold improvement in peak heights for phenolic acids. Although LVSS-sweeping-MEKC afforded 1000-fold improvement in peak heights which is lower than ASEI-sweeping-MEKC, the advantage is that it wasn't affected by sample solution easily. It is not only high sensitivity but also good techniques.