

Separation of Felted Explosives by Pressurized Microemulsion Electrokinetic Chromatography

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Abstract: A new MEEKC method assisted with pressure-driven mobile phase was presented for the separation of felted explosives. Microemulsion solution was composed of 80 mmol/L heptane – 120 mmol/L SDS (sodium dodecyl sulphate) – 900 mmol/L butanol – 10 mmol/L borate at pH 9.4 and a pressure-driven flow of 0.020 mL/min under 1.3 MPa was employed to manipulate the separation. Explosives HMX (1, 3, 5, 7-tetranitro-1, 3, 5, 7-tetrazacyclooctane) and TATB (triamino-trinitrobenzene), which were felted on fluorine rubber F2311 (polytrifluorochlorethylene and polyvinylidene fluoride 1/1 co-polymerization) and F2314 (polytrifluorochlorethylene and polyvinylidene fluoride 4/1 co-polymerization) were well separated with very good peak shapes.

Keywords: Felted explosives, microemulsion electrokinetic chromatography (MEEKC) separation pressurized MEEKC.

Several modes of capillary electrophoresis have been used in the separation of explosives, such as capillary zone electrophoresis (CZE), micellar electrokinetic chromatography (MEKC), capillary electrochromatography (CEC), microchip electrophoresis, *etc.* It was reported that microemulsion electrokinetic chromatography (MEEKC) was used in separation of proteins¹, vitamins², preservatives³, pharmaceuticals and excipients⁴, *etc.* C. G. Bailey and C. Yan⁵ separated 14 nitroaromatic and nitramine explosive compounds, using a capillary electrochromatography system with 1.5 μm nonporous octyldecylsilica (ODS) packed column. However, the pressurized MEEKC and its applications in the separation of explosives have not been reported up to now. In this paper we developed a new MEEKC method assisted with pressure-driven mobile phase (0.020 mL/min under 1.3 MPa) to separate felted explosives. It was indicated from our experiments that the four components of felted explosives were well separated in 26 minutes by the presented new method.

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Experimental

Sodium dodecyl sulphate (SDS), analytical grade, was purchased from Life Technologies Inc. (MD, USA) and borate was purchased from Beijing Chemical Factory (Beijing, China). Other chemicals, including heptane, butanol, N,N-dimethylformamide (DMF), sodium hydroxide (NaOH) and acetone, were all of analytical grade and purchased from Beijing Chemical Reagents Company (Beijing, China). Pure water prepared by Milli-Q system (Millipore, Bedford, MA, USA) was used for microemulsion solution.

HMX and TATB were dissolved by DMF, F2311 and F2314 were dissolved in acetone at a concentration of 100 µg/mL each. Sample solutions and mobile phase were all filtrated through 0.20 µm membrane filters and degassed by ultrasonication for approximately 15 min *prior to use*.

Instrumentation and conditions

Separation was performed with a Unimicro TriSepTM-2010GV pressure-loading capillary electrochromatography system equipped with a manual syringe pump, a UV/Vis detector, and a computer workstation for data acquirement and processing (Unimicro Technologies Inc., Pleasanton, CA, USA). An uncoated fused-silica capillary, 50 µm i.d. × 375 µm o.d. (Yongnian Optical Fiber Factory, Hebei, P. R. China), with total length of 52 cm and effective length 40 cm, was used for MEEKC separation. The other conditions: constant flow at 0.020 mL/min under the pressure of 1.3 MPa, applied voltage 20.0 kV, UV detection at 210 nm. Sample solution of 20 nL was injected.

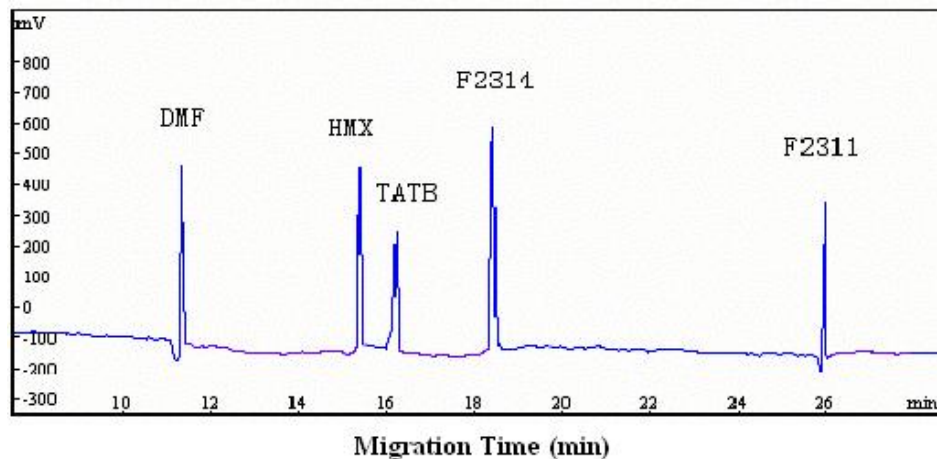
The microemulsion solution was composed of 80 mmol/L heptane – 120 mmol/L SDS (sodium dodecyl sulphate) – 900 mmol/L butanol – 10 mmol/L borate⁶ at pH 9.4.

Before use, new capillary were flushed with 1 mol/L NaOH, 0.5 mol/L NaOH, and pure water for one hour, respectively. Before the first run and between consecutive runs, capillary was conditioned by washing with mobile phase for 30 min and 10 min.

Results and Discussion

Figure 1 shows a MEEKC separation of felted explosives using a 52 cm (40 cm effective) × 50 µm i.d. capillary at 20.0 kV running voltage and 1.3 MPa pressure. It can be seen that perfectly symmetric peaks with high resolution were obtained. **Table 1** gives the chromatographic properties of MEEKC capillary column in this system.

Compared to conventional MEEKC, pressurized MEEKC demonstrated shorter analytical time because the pressure-driven flow is in the same direction as electroosmotic flow, showing that introduction of pressure-driven flow to the MEEKC could be an additional parameter to manipulate the separation. After optimization of the separation conditions, much better results for explosives separation should be achieved, which is in progress in our lab.

Figure 1 Electropherogram of separation of felted explosives by pressurized MEEKC.

Conditions: fused-silica capillary 52 cm (40 cm effective) \times 50 μm i.d. Voltage, 20.0kV (positive). Pressure, 1.3 MPa at a constant flow of 0.020 mL/min. Direct UV detection at 210 nm. Microemulsion system: 80 mmol/L heptane – 120 mmol/L SDS – 900 mmol/L butanol – 10 mmol/L borate (pH 9.4). DMF served as the indicator for electroosmotic flow (EOF).

Table 1 The chromatographic properties of MEEKC capillary column

Peak	Retention factor k'	Column efficiency (effective plates / m)	Relative retention value ()	Resolution (R)
HMX ¹	0.353	61239		
TATB ²	0.425	119833	1-2 (1.203)	R ₁₋₂ (11.46)
F ₂₃₁₄ ³	0.617	171856	2-3 (1.452)	R ₂₋₃ (30.04)
F ₂₃₁₁ ⁴	1.281	588084	3-4 (2.076)	R ₃₋₄ (89.31)

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