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Separation of Six Pyridoncarboylxic Acid Derivatives by Micellar and Microemulsion Electrokinetic Chromatography

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Abstract: Micellar and microemulsion electrokinetic chromatography (MEKC & MEEKC) separation of six closely structural pyridoncarboylxic acid derivatives were studied and compared. Both anionic surfactant sodium dodecyl sulfate (SDS) and cationic surfactant hexadecyl-trimethyl ammonium bromide (CTAB) were used to form micellar and microemulsion as pseudostation phases, respectively. The effects of the separation conditions on retention time and selectivity were studied. Good resolutions were obtained in selected systems, indicating that there is markably different selectivity between SDS and CTAB systems.

Keywords: Micellar, microemulsion, electrokinetic chromatography.

Micellar electrokinetic chromatography (MEKC) and microemulsion electrokinetic chromatography (MEEKC) are two kinds of electrokinetic capillary chromatography (EKC), which are characterized of high solubilization capacity and separation efficiency. In our previous work, some polar organic compounds and hydrophobic neutral compounds were separated successfully by EKC[1-3]. In this paper, these methods were used for separating six pyridoncarboylxic acid derivatives with similar structures. The separation in both anionic surfactant SDS and cationic surfactant CTAB MEKC (MEEKC) systems were investigated, respectively, and compared systematically. The effects of composition of micellar solution and microemulsion on separation were studied.

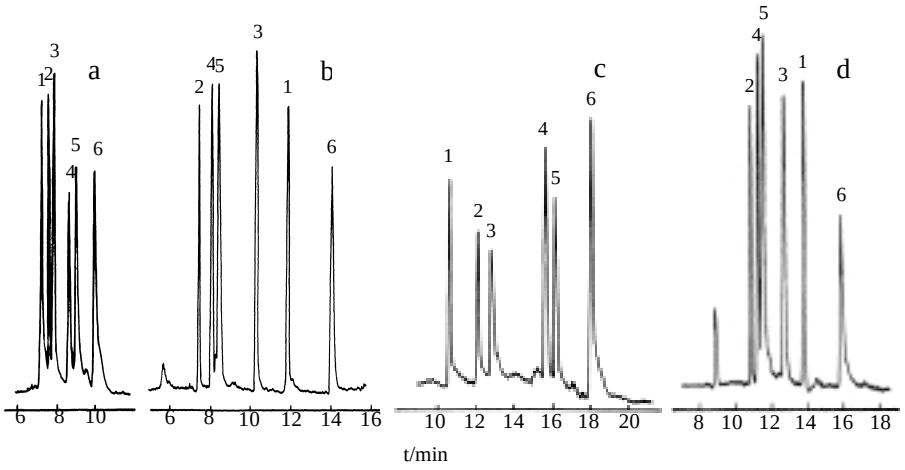
The separations were performed on Waters Quanta 4000 capillary electrophoresis system with UV detector monitoring at 214(254)nm. A 50 μm I.D.x47 cm (39 cm to detector) uncoated fused-silica capillary was used. Six pyridoncarboylxic acid derivatives, norfloxacin, fleroxacin, pefloxacin, lomefloxacin, ciprofloxacin, and ofloxacin were mixed to form test sample.

Figure 1 showed the separations of the test sample in both MEKC and MEEKC systems. Almost baseline resolutions were obtained in both SDS- and CTAB-MEKC systems and the better one was got in CTAB system. The elution order of solutes is different in both systems, which means their selectivity is different. In SDS system n-butanol must be added to micellar solution as organic modifier to get good resolution. Otherwise, in CTAB system organic modifier is unnecessary. These differences are mainly due to their different electrostatic interaction between micellar phase and solutes. Under selected conditions, all solutes were negative charged. Their interaction with

anionic SDS micellar was hindered by electrostatic repulsion. In SDS system n-butanol is helpful to solubilize the solutes into micellar so the separation was improved. However, electrostatic attraction was useful to intensify their interaction with CTAB micellar. Therefore, a better resolution was achieved. Similar results were obtained in MEEKC systems. Because of the higher solubilization of SDS-microemulsion compared with SDS micellar, a better resolution was obtained in SDS-MEEKC system. Also CTAB-MEEKC system showed a higher separation efficiency than which of MEKC.

The concentrations of surfactants and n-butanol exert a strong effct on separation. In all systems the retention time of solutes increased with the increasing concentration of surfactant. In both SDS-MEKC and MEEKC systems with the increasing of n-butanol the retention time of solutes got shorter. Otherwise, in CTAB-MEEKC system the retention time of all the compounds increased with the increasing concentration of n-butanol. This difference may be explained by the different solubilization capacity between SDS and CTAB systems.

Figure 1 Separation of pyridoncarboylxic acid derivatives by MEKC and MEEKC



Operating conditions: applied voltage, 15 kV(-8 kV); temperature, 30^{0}C; UV detection, (a,c) 214 nm, (b,d) 254 nm; micellar solution, (a) 50 mmol/L SDS-7.75%(v/v)- 10 mmol/L $Na_2B_4O_7$-30 mmol/L KH_2PO_4, pH 7.2, (b) 120 mmol/L CTAB-10 mmol/L $Na_2B_4O_7$-30 mmol/L KH_2PO_4, pH 7.2; microemulsion, (c) 80 mmol/L n-heptane-120 mmol/L SDS-10%(V/V) n-butanol-10 mmol/L $Na_2B_4O_7$-30 mmol/L KH_2PO_4, pH 7.2, (d) 80 mmol/L n-heptane-140 mmol/L CTAB-8%(V/V) n-butanol-10 mmol/L $Na_2B_4O_7$-30 mmol/L KH_2PO_4, pH 7.2. Peaks: 1. fleroxacin 2. lomefloxacin 3. ofloxacin 4. ciprofloxacin 5. norfloxacin 6. pefloxacin

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