



Stacking As Sample On-Line Pre-concentration Technique in Microemulsion Electrokinetic Chromatography

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Microemulsion electrokinetic chromatography (MEEKC) is an electro-driven separation technique based on capillary electrophoresis (CE) which utilizes microemulsions (ME) as separation background electrolytes (BGE). O/W ME are typically employed for MEEKC separations, which contain oil droplets (e.g., n-octane), suspended in aqueous buffer. Surfactants, usually sodium dodecyl sulphate (SDS), are present in excess of their critical micelle concentration (CMC), to facilitate droplet formation by lowering the surface tension. A short-chain alcohol (e.g., 1-butanol) is also added as co-surfactant, which further lowers surface tension and stabilizes the microemulsion system. These ME are used as pseudo stationary phase in MEEKC. MEEKC separates solutes based on hydrophobicity and differences in electrophoretic mobility, and it offers highly efficient separation of charged and neutral solutes covering a wide range of water solubility. The separation is a combination of electrophoretic mechanism and chromatographic partitioning with the ME droplets. The partitioning occurs between the oil droplets and the aqueous phase. Uncharged, highly hydrophilic solutes reside in the aqueous phase of the ME and migrate quickly with the EOF towards the detector. Charged species are also electrophoretically separated based on their mass, charge, and interaction with the oil droplet. Positively-charged solutes may form an ion-pair with the surface of a negatively-charged droplet while negatively-charged solutes in general will be repelled by the droplet. The more the analyte is incorporated into the micelle, the slower the analyte will migrate. The analytes are detected in an increasing order of the distribution co-efficients at the cathodic end [1,2].

Compared with micelles in micellar electrokinetic chromatography (MEKC), MEEKC provides better selectivity. This could be due to easier penetration of solute molecules into the surface of the ME droplet along with improved mass transfer between the ME droplet and the aqueous phase, compared with the more rigid micelles in MEKC. Also, ME have more adjustable parameters such as type and concentration of oil and co-surfactant and larger solubilization capacity for highly hydrophobic analytes. Therefore MEEKC would provide better selectivity, higher separation efficiency, and could be more suitable to separate water-soluble and fat-soluble compounds simultaneously [1,2].

In spite the fact that CE has found vast applications over the past few years, it suffers from poor concentration sensitivity due to the short optical path length and a small sample volume injection. Currently, several on-line pre-concentration techniques are often employed in CE to enhance the sensitivity. On-capillary focusing techniques have been developed to pre-concentrate the analyte within the capillary before separation and detection. This can be done by injecting a large volume of sample solution and focusing the analyte into a narrow band inside the capillary.

Sample stacking is among the most widely used pre-concentration methodologies and it occurs as ions cross a boundary that separates regions of the high electric field sample zone and the low electric field background solution (BGS) zone. The most common of which is reversed electrode polarity stacking mode (REPSM). In this mode, a large plug of sample is injected hydro dynamically into the capillary

followed by application of negative voltage which allows removal of the sample matrix out of the capillary as well as stacking of the analyte species at the boundary between the low conductivity sample zone and the high conductivity BGE. This is followed by switching the polarity back to the positive mode to carry out the actual separation of stacked analytes. This technique has been widely applied to improve detection sensitivity in MEEKC determination of many drugs of different polarities [3].

References

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