

Comparative Study of Aqueous and Non-aqueous Capillary Electrophoresis-MS in the Separation of Phenolic Compounds of Virgin Olive Oil

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The analysis of the phenolic fraction of virgin olive oil has been carried out traditionally by using HPLC or GC; however, capillary electrophoresis (CE) generally provides high efficiencies and fast analyses, and is therefore considered a potent alternative method. Most CE methods use an aqueous electrolyte separation medium, although there is also a considerable interest in non-aqueous CE (NACE). In the case of olive oil, the importance of NACE is obvious, as this kind of CE seems to be more compatible with the apolar or hydrophobic olive oil matrix and could facilitate its direct injection.

The aim of this work was to compare the results obtained by a method involving SPE and CE (with aqueous buffers) coupled to ESI-IT MS and those obtained by a NACE-ESI-IT MS method. Both of them offered to the analyst the chance to study phenolic compounds of different families (such as phenolic alcohols, lignans, complex phenols, flavonoids...). from virgin olive oil by injecting methanolic extracts with efficient and fast CE separations. We also studied the direct injection of the olive oil using the NACE method.

All the SPE, CZE and ESI-MS parameters were optimized in order to maximize the number of phenolic compounds detected and the sensitivity of their determination. Electrophoretic separation was carried out, in the aqueous method, using a CE buffer system consisting of 60 mM NH₄OAc at pH 9.5 with 5% of 2-propanol, a sheath liquid containing 2 propanol/water 60:40 v/v and 0.1% v/v triethylamine. A non-aqueous CE method may be developed, using as start values the buffer composition and pH conditions optimized for aqueous separations, although it has to be considered the electrolytes including organic solvents may have very different chemical and physical properties as compared to aqueous electrolytes. We studied in depth the effect of the nature of the electrolyte, the effect of ACN-MeOH mixtures percentages, and other experimental and instrumental CE variables.