Characterization of stationary phases functionalized by photochemistry

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Introduction

Monoliths are used as a new stationary phase which enables the miniaturization of systems and give good performances. The advantage of that kind of stationary phase is that their synthesis can be done in-situ, thus, the synthesis is prepared in fused silica capillary of 75 µm inner diameter.
Figure 1 represents a scanning electron microscopy image of a silica monolithic column characterized by interconnected macropores.
The classical silanisation process wide lead to the so called ODS column (OctaDecyl Silane), is very long and laborious. To find a solution to this time consuming process, the Laboratory of Analytical Sciences (LSA) focused on a new photochemical process.
Figure 2 illustrates the original photochemical approach investigated.
The aim of this work was to characterize the new monolithic silica columns. This was done by measuring some chromatographic selectivities (the Tanaka diagram). These characterizations allow the determination of the influence of the spacer, the morphology (monomeric or polymeric) of the obtained layers and the impact of residual surface silanols on the retention of basic compounds for example.

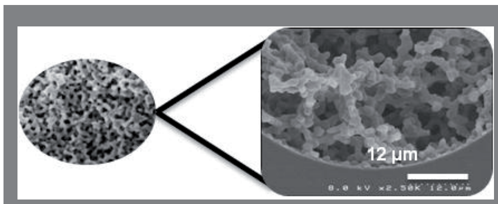

Figure 1 : diagram of a capillary with a zoom on the skeleton of monolith.

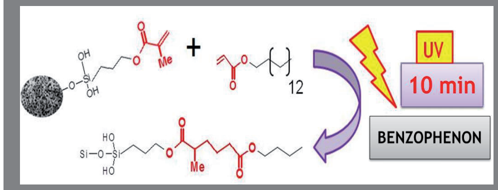

Figure 2: photographting of acrylate C12 carbon chain on a methacrylate actionable under UV. In red, capillary spacer is represented. BME: benzoic methyl ether

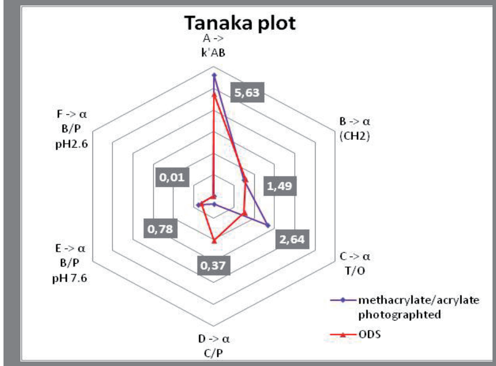

Figure 3: Tanaka plot: A, surface coverage, B, methylene selectivity, C, steric selectivity, D, amount of silanols and E and F silanols activity at pH > 7 and pH < 3. In red the classical column, in violet the photographted one

Methodology

All manipulations were done with a capillary electrophoresis system under 12 bars. The UV detector set on 214 and 254 nm enabled the detection of all the compounds. The Tanaka test is composed of 6 parameters: the surface coverage, the methylene selectivity, the steric selectivity, the amount of silanols and the silanols activity at pH > 7 and pH < 3. For these different parameters, several solute were used: amyl benzene, hexyl benzene for the first and the second tests; ortho-terphenyl, triphenylene for the third; caffeine, phenol and benzylamine for the last three analyses. Thiourea was used to determine the column dead time.

Results and discussion

All the results were combined on a radar plot in figure 3. These results were compared with an ODS column which serves as the reference column. The results are quite similar for tests A and B which means that the hydrophobicity does not vary much between these two columns. The steric selectivity is large for this column which demonstrates the polymeric nature of the layer. Thus, this kind of column can clearly separate compounds by their shape not just their hydrophobicity.

Conclusion

The investigated photografting process is a simple, rapid and versatile process to elaborate a miniaturized column.

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