AN IMPROVED PDMS SOLID-PHASE MICROEXTRACTION FIBRE OBTAINED BY A SOL-GEL/SILICA PARTICLE BLEND

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Solid-phase microextraction (SPME) is increasingly used as a successful alternative to the conventional extraction techniques. One of the drawbacks of SPME is the fragility of both the coatings and the substrate (optical fibre) used in most the commercially available devices. Some recent advances in the production of robust (i.e. stable to higher temperatures and organic solvents) coatings were achieved by sol-gel coating of fused silica. The use of fused silica as substrate brings the inconvenience that the fibres may easily break after a less cautious manipulation or device irregularities. Consequently, it was decided to use a titanium wire instead. Titanium spontaneously forms a thin oxide layer on the surface. It was shown that Ti-O-Si bonds can be formed from reactions involving surface Ti-OH groups [1]. The sol-gel deposition on fused silica involves covalent bonding of the film via surface Si-OH condensation with OH-terminated sol-gel aggregates. Concurrently, as already shown in previous studies, one can also obtain strong sol-gel film stability on titanium [2]. In the present study it was attempted to enhance the efficiency of a covalently bonded sol-gel polydimethylsiloxane (PDMS) titanium fibre by incorporating porous silica particles in the film.

The wires (0.250 mm diameter) were pre-treated with NaHO 1 mol/dm^3 in order to increase the number of surface Ti-OH groups, and readily assembled to spare parts of commercial devices. A first deposition was carried out by dip-coating the wire on a PDMS sol-gel bath which was then covered with silica particles before immersing it for a second time in a newly prepared sol-gel solution.

The observation of the coated wires by scanning electron microscopy revealed a homogeneous and complete coverage with silica particle blend. This coating exhibited significantly higher extraction performance as compared to the PDMS fibre. It was concluded that the enhanced extraction efficiency of this

fibre was related with the quantity and the dimension homogeneity of the particles incorporated in the film and consequently with the portion of the PDMS film that is bonded to the titanium substrate.

References

[1] Fadeev, A.; McCarthy, T. *J. Am. Chem. Soc.* 1999, *121*, 12184-12185

[2] Azenha, M.; Nogueira, P.; Fernando-Silva, A., submitted