

Comparison between SPE and MEPS – Application in fatty alcohol analysis

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Introduction

Most time of the laboratory work is spent in sample preparation. For these last years, new processes have been created. The Micro Extraction by Packed Sorbent (MEPS), is commercialised since 2007 by SGE. In order to test its performances, it is compared to the SPE (Solid Phase Extraction). The objective is to show if the SPE conditions can be directly adapted to MEPS. These techniques of preparation were employed to analyze fatty alcohols that can be found in cosmetic products. The dodecanol was determined in raw materials such as sodium dodecyl sulphate pure at 99% (SDS 99), to test the recovery rates of MEPS and SPE.

Materials and methods

Sample preparation

The SPE cartridges were filled with 500 mg of sorbent (C18). Their capacity was about 5% (w/w). The MEPS was carried out thanks to a syringe of 250 μL . A BIN (Barrel Insert and Needle) was screwed onto the syringe with a nut. It contained 4 mg of sorbent (C18) whose the capacity was about 5% (w/w) too.

The both techniques needed several steps to be executed. To start, a conditioning was carried out to eliminate possible interferences on the sorbent. Then, the sample was poured through the sorbent. The next step consisted in rinsing the solid phase with a solvent in which the analyte was not soluble. Afterwards, the analyte was eluted in an appropriate solvent. To finish, in case of MEPS, a very eluting solvent was passed through the sorbent to clean it. For the SPE, this cleaning was not necessary since the cartridges were not reusable.

Chromatography

Samples were analyzed by HPLC (High Performance Liquid Chromatography) after derivatization of dodecanol with 2-furoyle chloride (figure 1). The samples were prepared with biphenyl as an internal standard at a concentration of 2 mg.L⁻¹ for each sample. The device was composed of a P680 pump, an ASI 100 auto sampler, a TCC-100 thermostated compartment for the column and a UVD340U UV detector, by Dionex Company. The used column was a GL Wakosil II 5C18RS (C18 phase) by SGE Company. Its dimensions were 25 cm x 2.1 mm x 5 μm and it was thermostated at 25°C. The injected volume of sample was 20 μL . The mobile phase was a water / acetonitrile mix with a flow rate at 0.2 mL.min⁻¹. It started with 30% of water and 70% of acetonitrile for five minutes. It reached 100% of acetonitrile in 40 minutes and was held for 5 minutes. Then, it came back at initial conditions in 1 minute and it remained constant for 14 minutes. The selected wavelengths for detection were 249.0 nm and 251.0 nm.

Results and discussions

It was shown that dodecanol was eluted in the acetonitrile fraction. Several doped samples were analyzed after extractions on MEPS or on SPE and without extraction. Recovery rates were calculated thanks to the ratios between areas under chromatographic peaks corresponding to the ester from dodecanol and the internal standard.

Figure 1 : Derivatization reaction of dodecanol with 2-furoyle chloride.

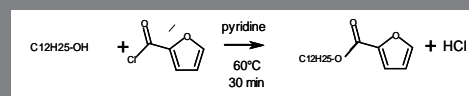
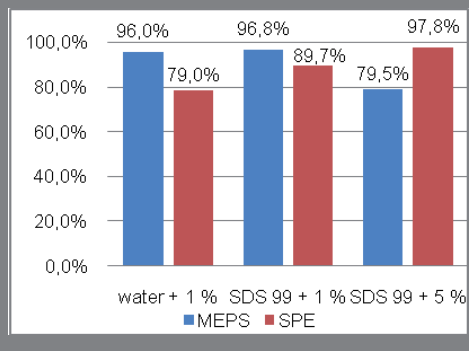


Figure 2 : Average recovery rates obtained with MEPS and SPE for three solutions : water with 1% of dodecanol, SDS 99 with 1% of dodecanol and SDS 99 with 5% of dodecanol.



The bar chart, figure 2, shows that MEPS had better recovery rates than SPE for the solutions containing 1% of dodecanol. However, SPE had the highest recovery rate for the solution which contained 5% of dodecanol.

MEPS uses about 1.6 mL of solvent against 30 mL for SPE. Moreover, four MEPS sequences can be carried out for one extraction by SPE.

Conclusion

This study demonstrates that, using the same solvents in the extraction procedure, MEPS offers better recovery rates than SPE. Nevertheless SPE seems to be more efficient for higher concentrations in dodecanol. Moreover, it is possible to gain time and economize a very high quantity of solvents for extractions with MEPS. Other advantage, MEPS can be used up to 100 extractions with only one micro-cartridge.

