Migration analysis of perfluorinated compounds (PFC) in real food samples

Ludwig Gruber[1], Martin Schlummer[1], Jan Ungewiss[1], Gerd Wolz[1], Angela Möller[1], Nicole Weise[1], Hermann Fromme[2]

[1]Fraunhofer Institute for Process Engineering and Packaging (IVV), Giggenhauser Straße 35, 85354 Freising, Germany, email: gruber@ivv.fraunhofer.de, phone: ++49 8161 491 706
[2]Department of Environmental Health, Bavarian Health and Food Safety Authority, Veterinärstraße 2, D-85764 Oberschleißheim, Germany

Introduction

There are indications, that food may play a major role as source for perfluorinated compounds (PFC) for the non-occupationally exposed population. One possible source for PFC in food is the migration from food contact materials. Begley and coworkers [1] demonstrated that the traditional food simulants water and oil are not the both extremes for these tensidic compounds. Furthermore the presence of soy emulsifier (lecithin, 0.05%) can change oil into a potent solvent for migration of PFC. Therefore it is recommendable to perform these migration analyses not in food simulants but in real food samples. The aim of this study was to develop a robust analytical method for different kinds of food samples. The development of the method was based on a publication by Taniyasu et

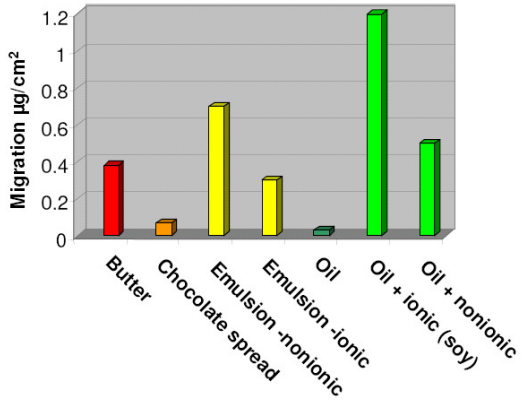

Figure 2: Example for PFOA migration from coated paper [3]

Method

Sample preparation:

The food sample has to be blended and homogenized. A 30 g aliquot of the homogenized sample is filled in a PP tube and fortified with internal standards. The internal standards $^{13}C_4$-PFOA and $^{13}C_4$-PFOS were purchased from Wellington (Ontario, Canada).
After addition of 2 ml of water and 10 minutes of ultrasonic extraction, the samples are centrifuged for 10 minutes at least with 4500 rpm and the supernatant has to be collected in a PP vessel. The sediment has to be twice re-extracted with methanol in an ultrasonic bath.
The combined extracts are diluted with the fivefold amount of water, filtrated through a syringe filter and subjected to solid phase extraction (SPE). Weak anion exchange SPE cartridges (Oasis® Wax, 150 mg, 6 ml, 30 µm) are preconditioned with 2 ml of methanol and water, respectively, and the extracts are passed through the preconditioned cartridges. The cartridges are then washed with methanol/water (1/1; v/v) and eluted with 1% NH_4OH in methanol. SPE eluates were evaporated under a gentle stream of nitrogen and diluted with water to a final volume of 1 ml.

HPLC-ESI-MS/MS:

Identification and quantification of perfluorinated substances was performed on a Surveyor Plus HPLC connected to a Quantum Ultra AM mass spectrometer (both Thermo Fisher). Chromatographic separation was achieved by a Fusion RP phase (20 x 2 mm, 2 µm, Phenomenex). Gradient HPLC was performed with methanol and 5mM ammonia acetate in water (pH 3.5), increasing methanol from 20 to 100% within 10 minutes. Mass spectrometry was performed by electron spray ionization (ESI) in the negative ion mode and subsequent single reaction monitoring.
Even with HPLC-ESI-MS/MS interferences can disturb the quantification of PFOS significantly [3]. Taurodeoxycholate, which coelutes with PFOS isomers, produces significant interference on the most sensitive MS/MS transition of PFOS, 499 to 80. The interference can be avoided by monitoring the more specific transition 499 to 99 [4].

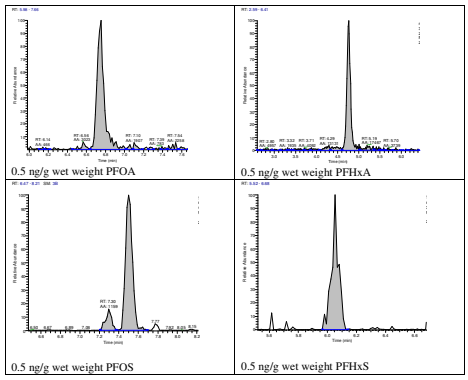

Figure 2: Sensitivity of the method shown by MS/MS fragmentograms of PFOA, PFOS, PFHxA, and PFHxS reflecting 0.5ng/g wet weight in diet samples

Results and Conclusions

The described method proved to be a robust and sensitive means to analyze PFC in food. In fish and mixed food samples, limits of detection were between 0.05 ppb for PFOS and PFOA and 0.1 ppb for the other target compounds. This analytical method enables laboratories to perform migration experiments for perfluorinated compounds (PFC) with real food samples in order to avoid problems for exposure estimates.

References

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