Screening for Migrating Substances from Food Contact Laminates

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Introduction and Aims

Laminates are widely-used in the food packaging industry. Migrating substances from PE sealing layers commonly are stabilizers like antioxidants and processing aids (antistatics and slip agents). The migraton of these additives is regulated and there are numerous specific migrations limits (SML's) that have to be observed.[1]

The aim of the project was to identify additives and other migrating substances in the substitute simulants for fatty food contact (iso-octane, 95 % ethanol). The value for the overall migrate (OM) determined by gravimetry shall be elucidated by the migration of non-volatile substances.

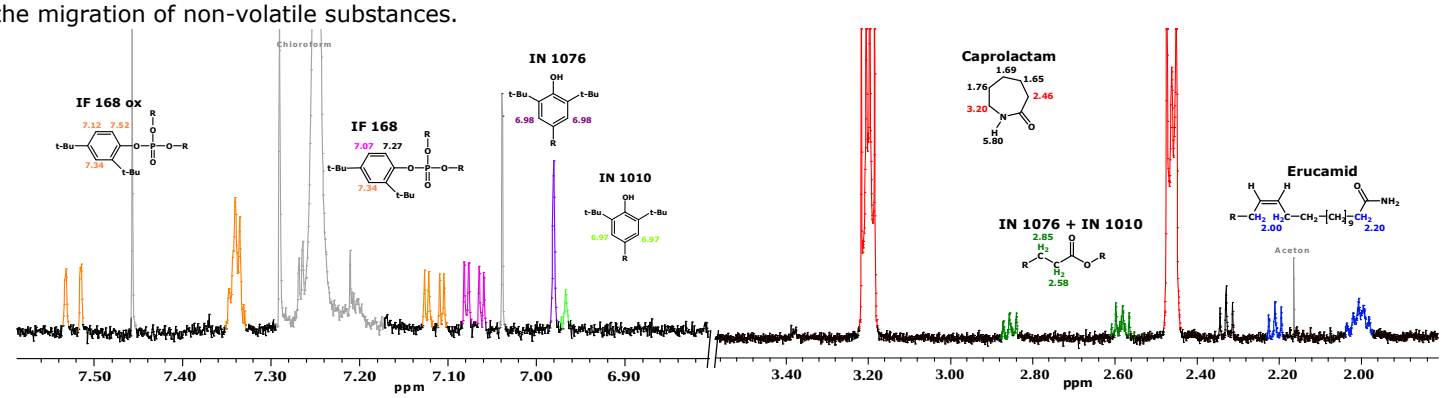

Fig. 1: Extracts of the ^{1}H-NMR spectra of the 95 % ethanolic migrate (4h, 60 °C) prior to saponification

Identification of the migrating additives

The determination was carried out in the substitute simulants 95 % ethanol (4h, 60°C) and iso-octane (2h, 60°C). The screening for additives in the migrates was carried out with ^{1}H-NMR.[2] Defined moieties of additives are allocated due to specific shifts in the spectra (**Fig. 1**).

For identification and quantification, respectively, a formerly introduced RP-HPLC-screening method with UV-, FL-, ELS- and CLN-detection was carried out.[3] The overall migration was determined gravimetrically for both substitute simulants. Thus, for 95% Ethanol the overall migrate was determined with 1.2±0.1 mg/dm² und for iso-octane 5.6±0.1 mg/dm².

The amounts of additives in both migrates are shown in **Tab. 1**. It is obvious that in the 95% Ethanol and iso-octane, respectively, the values of IF 168ox and IN 1076 are similar. Furthermore IN 1010 and erucamide can't explain the large difference in the overall migrates.

Tab. 1: Amounts of the additives in the 95 % EtOH and iso-octane migrate of a laminate[1]

additive	concentration [µg/dm²]	
	95 % ethanol	iso-octane
Irgafos (IF) 168ox	296 ± 5	260 ± 14
Irganox (IN) 1076	178 ± 6	168 ± 5
Irganox (IN) 1010	13 ± 5	56 ± 10
Erucamide	201 ± 14	98 ± 5

[1]The caprolactame shown in **Fig. 1** is a volatile component of the migrate and is not included in the determination of the overall migration because it is not detected with the gravimetric method.

Quantification of migrating PE-oligomers by ^{1}H-NMR

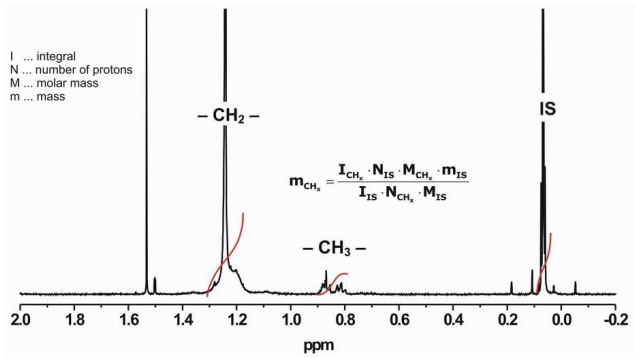

Fig. 2: ^{1}H-NMR spectra for quantification of PE (IS = Decamethylcyclopentasiloxan)

The amount of migrating PE oligomers is determined by NMR.[4] Therefor the additives have to be separated by saponification and extraction. The PE oligomers are quantified using the integrals of the CH_2- und CH_3-shifts (**Fig. 2**). The amount of PE oligomers in the 95 % ethanolic migrate was determined with 410±50 µg/dm² and for the iso-octane migrate with 4640±170 µg/dm².

Clarification of the OM in the substitute simulants

The quantitative determination of the migrating low molecular weight PE oligomers contributes an essential step for the clarification of the overall migrate of plastic laminates. The overall migration of a composite film could be explained approximately complete. **Fig. 3 a** und **b** show the balance of the non-volatile substances in both migrates.

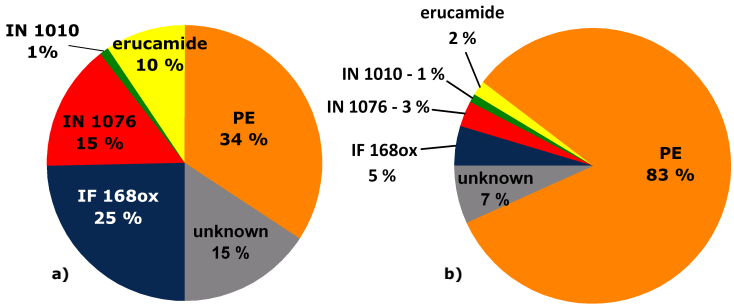

Fig. 3: Balance of the overall migrate [% specific substances of the gravimetric overall migrate] **a)** 95 % ethanol (4 h 60 °C, OM=1.2 mg/dm²) **b)** iso-octane (2 h 60 °C, OM=5.6 mg/dm²)

Literature

[1] European Commission: Synoptic Document, SANCO D3/LR (2005)
[2] Ehret-Henry et al.: Food Addit. Contam., 1998, 9, 303-314
[3] Paul et al.: Poster presented on the 36. Dt. Lebensmittelchemikertag in Nuremberg 2007
[4] Malz and Jancke: J. Pharm. Biomed. Anal. , 2005, 38, 813–823

Acknowledgement

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