Migration Testing Challenges: Evidence Accelerated Migration Tests can Induce Physical Changes in Thin Polyolefin Food Contact Materials

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Background

Food contact materials (FCMs) may transfer substances from the packaging to the foodstuff. The Framework Regulation (EC) N° 1935/2004 states that this transfer of substances is not allowed to endanger the health of the consumer. Compliance with the Framework Regulation is often assessed by performing accelerated migration tests with food simulants and at elevated temperatures, as specified in the Plastic Regulation (EU) N°10/2011.

To provide a safe margin for the compliance demonstration, the (EU) N°10/2011 test conditions are designed to over-estimate the migration results to the real-life situation. However, dubious migration concentrations have been reported which seem to indicate extractive conditions of the non-direct food contact (DFC) side. This study aims to determine the mechanism in which unrealistically high migration concentrations may be reported when testing polyolefin films printed on the non-food contact side, at elevated temperatures, and in combination with liquid food simulants.

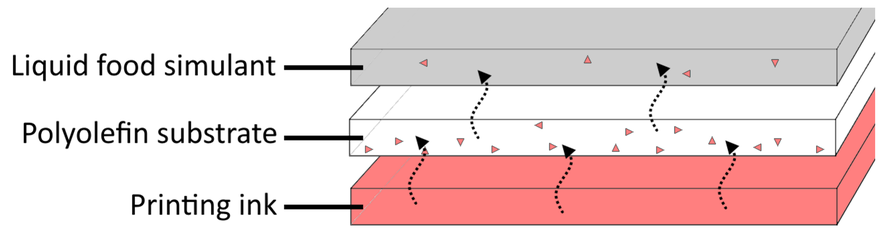

Figure 1: Diffusion-based migration. Grey layer = food simulant; White layer = polyolefin substrate; Red layer = printing ink.

Methods

Permeability Experiment

Alcohol-based permanent markers (Artline 70N) were used to mark the OPP film with the letter “R”, denoting the non-food contact side of this mock FCM. The substrate was placed in a migration cell (R-side facing down).
The unmarked food contact side was exposed to 50 ml ethanolic food simulant (EtOH). The cells were incubated according to the Plastics regulation (EU) N° 10/2011 (40 ± 1°C for up to 10 days; 60 ± 1°C for up to 10 days) and compared with a control for realistic long-term storage (20 ± 2°C for up to 6 months).

Figure 2: Migration cell.

Oxygen Transmission Rate

Samples of the OPP film were further analysed using a MOCON OTR Analyser based on ASTM F2422-08 to assess the barrier properties. Samples of the OPP film were taken as received (at ambient temperature), following exposure to 60 ± 1°C for 24 hours and following exposure to 60 ± 1°C in contact with ethanol for 24 hours.
The samples were placed inside a cartridge forming a semi-barrier between two chambers held at constant temperature, pressure and humidity before purging one side of the chamber with nitrogen and exposing the opposite chamber to the oxygen carrier gas. The volume of oxygen gas that diffused through the samples over the course of 24 hours was measured.

Results

Permeability Experiment

Incubation at 20°C

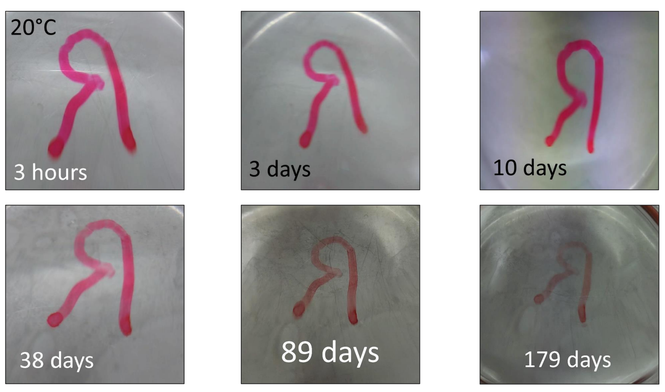

Figure 3: 50% EtOH as the food simulant at different timepoints at 20 ± 2°C.

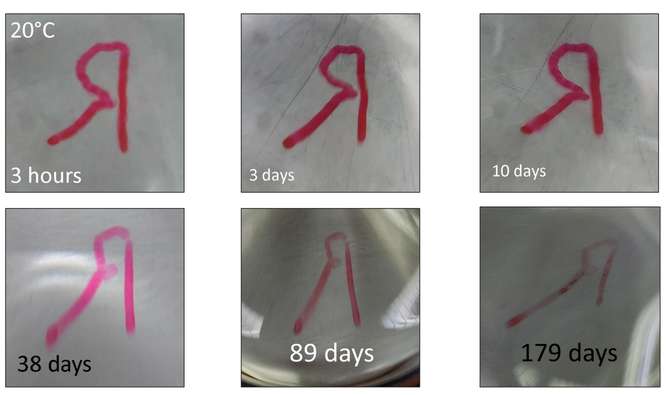

Figure 4: 95% EtOH as the food simulant at different timepoints at 20 ± 2°C.

- No simulant penetration observed

Incubation at 40°C

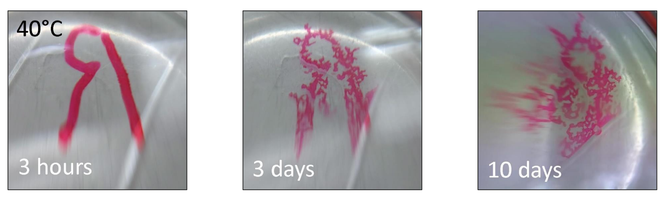

Figure 5: 50% EtOH as the food simulant at different timepoints at 40 ± 1°C.

Figure 6: 95% EtOH as the food simulant at different timepoints at 40 ± 1°C.

- Simulant penetration observed after 3 days with 50% EtOH
- Simulant penetration observed after 3h with 95% EtOH

Incubation at 60°C

Figure 7: 50% ETOH as the food simulant at different timepoints at 60 ± 1°C.

Figure 8: 95% EtOH as the food simulant at different timepoints at 60 ± 1°C.

- Simulant penetration observed after 3h with 50% EtOH
- Simulant penetration observed after 3h with 95% EtOH

Print Sample at 60°C

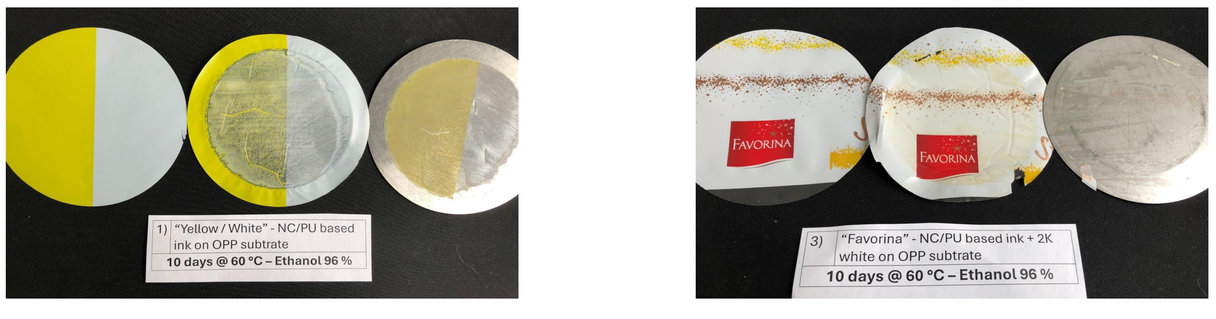

Figure 10: 96% EtOH with different coatings on OPP substrate at 10 days at 60 ± 1°C.

- Physical changes of the test specimens were observed to occur; "washing off" of the ink layer due to a reduction in ink adhesion and creasing due to swelling of the substrate.

Oxygen Transmission Rate

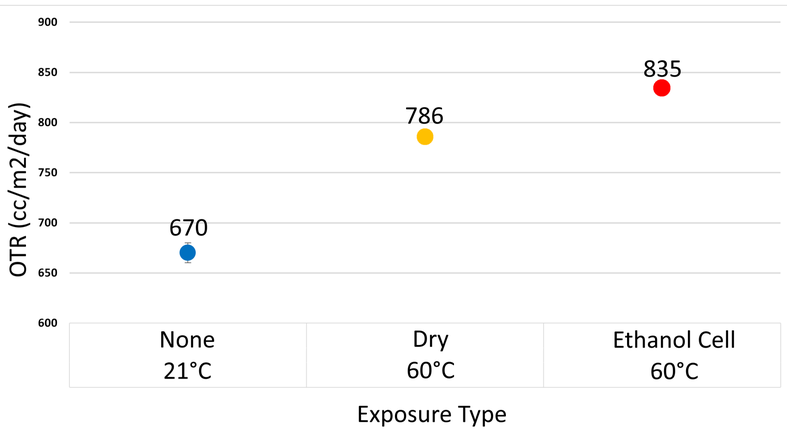

Figure 9: Oxygen transmission rate in cc/m²/day for different exposure types.

Conclusion

These experiments prove that exposing FCM films to ethanolic food simulant at elevated temperatures can increase the permeability of the film, quickly leading to penetration of simulant and ultimately in direct contact between the simulant and the non-food contact side of the FCM.
As such simulant penetration does not occur when incubating at room temperature for 6 months the observed permeability change constitutes an unacceptable physical change to the test specimen which does not occur under the worst foreseeable conditions of use of the pFCM.
As direct contact between the simulant and the printed side of a non-DFC pFCM will lead to direct extraction of the printed side rather than migration simulation, it is concluded that whenever simulant penetration occurs the results of the migration test cannot be used to conclude non-compliance of the FCM.

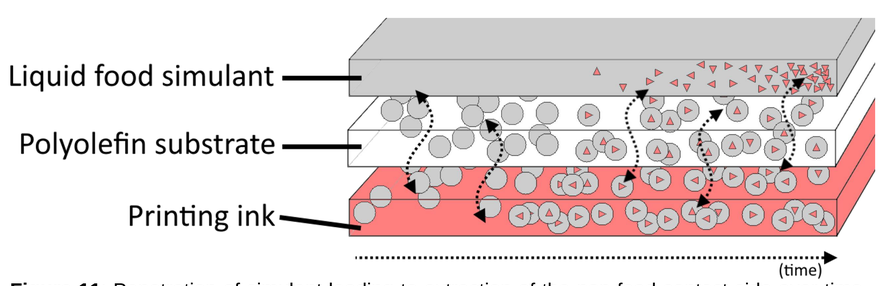

Figure 11: Penetration of simulant leading to extraction of the non-food contact side over time. Grey layer = food simulant; White layer = polyolefin substrate; Red layer = printing ink.