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

February 2025

Executive Summary

In this paper it is demonstrated that performing accelerated migration tests on thin FCM films at elevated temperatures in combination with liquid food simulants can increase the permeability of the film. This physical change to the test specimen enables the penetration of the liquid food simulant through the substrate towards the outside of the food packaging. This results **in direct contact between the food simulant and the non-food contact side, and in the direct extraction of printing ink components by the food simulant.** As contact between the foodstuff and the outside of the printed packaging does not occur under the worst-case foreseeable conditions of use, **the results of accelerated migration tests cannot therefore lead to a conclusion of non-compliance of the non-DFC pFCM whenever such simulant penetration is detected.**

Summary

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 migration tests in accordance with the Plastic Regulation (EU) N° 10/2011. Since testing in real foods can pose analytical challenges, the Plastic Regulation allows for the use of simplified test media which imitate food. These so-called “food simulants” can be either solids (MPPO; Tenax®) or liquids (most often ethanol-in-water solutions).

As some FCMs may be in contact with the foodstuff for weeks or even months (as is often the case for food packaging), the Plastic Regulation allows the assessment of compliance by testing the FCM for 10 days if the test temperature is increased. The scientific basis for reducing the test time while increasing the test temperature is derived from the Arrhenius Equation (see Appendix 1); the rate at which substances diffuse from the food packaging towards the food simulant increases with the temperature. However, the Plastic Regulation acknowledges that when migration tests are executed with contact conditions that cause physical changes to the FCM, the migration test should instead be carried out under the worst foreseeable conditions of use in which these changes do not occur.

This paper demonstrates that **such physical changes may occur when FCM films are tested at elevated temperatures in combination with ethanolic food simulants**, thereby invalidating the test results. More specifically, it is shown that the test conditions can lead to an increase in the permeability of the test specimen, resulting in penetration of the food simulant towards the non-food contact side, and ultimately in direct extraction of the outside surface of the packaging (see Figure 2). This is especially detrimental to the validity of the test whenever the FCM is printed on the non-food contact side, as in this case the food simulant will be in direct contact with the printing ink.

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In a real-life situation, the foodstuff will not migrate through the packaging and extract the printing ink on the outside. Therefore, whenever penetration of the food simulant occurs in an accelerated migration test on a non-DFC pFCM, the test results are not representative of the worst-case foreseeable use of the packaging. In this case it is not possible to conclude non-compliance of the pFCM, and the test has to be repeated under the worst foreseeable conditions of use in which these physical changes do not take place.

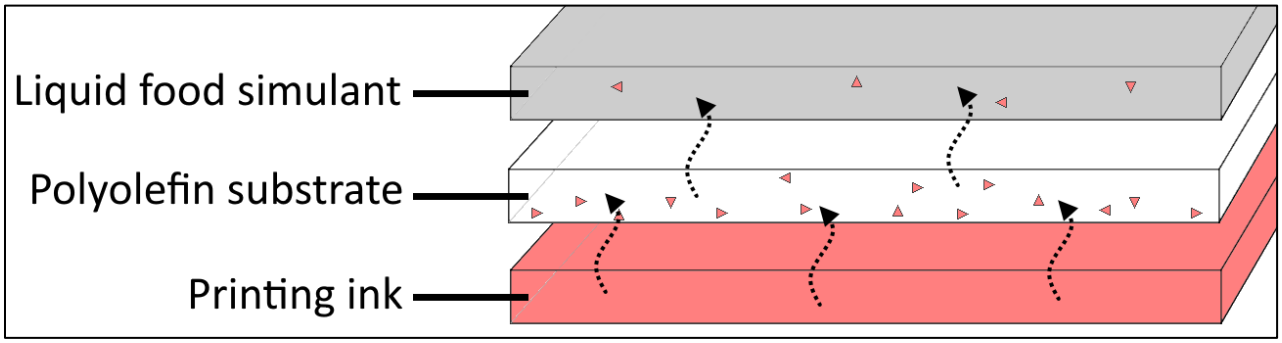

Figure 1: Normal diffusion-based migration.

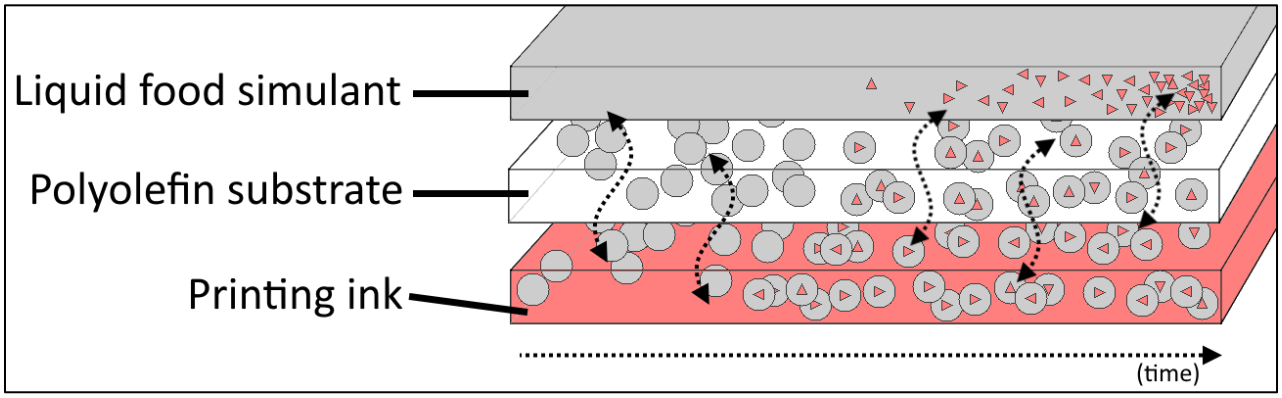

Figure 2: Penetration of simulant leading to extraction of the non-food contact side.

In Figure 1, components of the printed FCM (red triangles) are migrating (diffusing) through the substrate, and towards the food simulant. In Figure 2, the substrate has become permeable to the food simulant (grey circles) due to the selected migration test conditions.

Penetration of the food simulant towards the non-food contact side of the FCM leads to **direct extraction of printing ink components**. The direct contact between the food simulant and the printing ink layer ultimately leads to much higher detected concentrations of printing ink components in the simulant, no longer representative to the worst foreseeable conditions of use.

Results

A 50 µm OPP film was used as the substrate. This is the same substrate as used in the EuPIA Migration Study (published April 2024).

Experimental design

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 (see Figure 3). Because the permanent marker is easily dissolved in alcohol, any penetration of ethanolic simulant will be detectable by dissolution of the mark[1].

The substrate was placed in a migration cell, with the "R"-mark facing down. The unmarked food contact side was exposed to 50 ml of two liquid food simulant (50% ethanol and 95% ethanol). Cells were incubated at the following temperatures and durations:

- Incubation at 60 ± 1°C for up to 10 days. These accelerated test conditions correspond to real storage of the packaged foodstuff at room temperature for 6+ months (Plastic Regulation).
- Incubation at 40 ± 1°C for up to 10 days. These accelerated test conditions correspond to real storage of the packaged foodstuff at room temperature for up to 30 days (Plastic Regulation).
- Incubation at room temperature (20 ± 2°C) for up to 6 months. This condition was added as a control for realistic long-term storage at room temperature.

The integrity of the mark was monitored and photographed throughout the incubation period. The photographs are shown in Figures 4 and 5.

Results

The following observations were made for both simulants (50% and 95% ethanol):

- Incubation at 60°C led to clear signs of dissolution after only 3 hours of incubation. After 3 days, the mark had (nearly) completely dissolved.
- Incubation at 40°C led to clear signs of dissolution after 3 days of incubation. After 10 days the mark had heavily deteriorated.
- Incubation at room temperature did not show any signs of dissolution, even after incubation for 179 days (6 months).

These results show that incubation at 60°C can lead to a very quick change in the permeability of OPP films, resulting in penetration of the food simulant, and subsequently in direct contact between the simulant and the non-food contact side. As such behaviour was not observed for the sample that was stored at room temperature for 6 months, it follows that the observed

[1] The permanent marker enables visual detection of simulant penetration. Printing inks may not exhibit such rapid dissolution whenever simulant penetration occurs. Therefore, penetration of simulant in a real migration test will be less noticeable than demonstrated in the experiment with the permanent marker. Although deleterious effects to the printing ink layer, such as loss of adhesion, are sometimes observed to occur, the absence of any visual defects of the printing ink layer does not exclude the possibility that the food simulant has penetrated the substrate (and therefore does not exclude that direct extraction of printing ink components from the non-food contact side has occurred).

increase in permeability effected by the incubation at 60°C constitutes a physical change to the test specimen which does not occur under the worst foreseeable conditions of use of the food packaging[2].

Additional, supporting experiments were performed and are documented under Appendix 2.

Figure 3: Illustration of the experimental setup. Because the “R”-mark is made on the reverse side of the transparent substrate, it appears mirrored. The side facing up is in contact with the simulant and is not marked.

[2] The mark on the substrate incubated for 6 months at room temperature was noted to have faded somewhat over time. This is likely caused by either colourant breakdown due to ambient light, or by actual migration of the colourant through the substrate. However, the “R”-mark never showed any signs of dissolution when incubating at room temperature, indicating that even after 6 months no simulant penetration occurs.

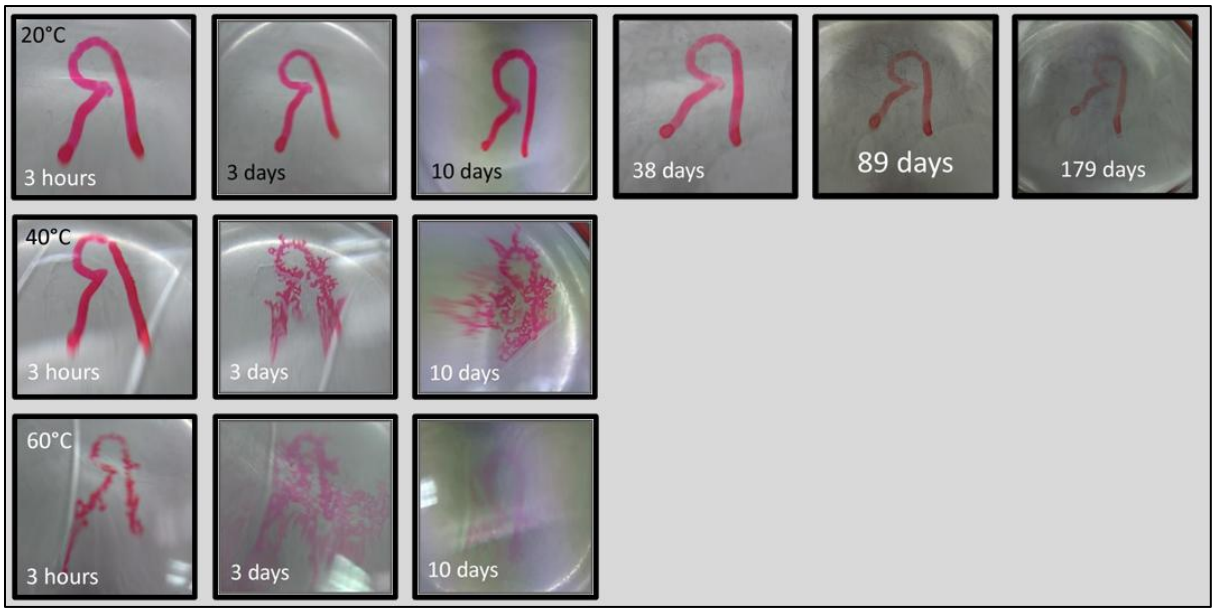

Figure 4: Photographs of the mark, at different timepoints, using 50% ethanol as the food simulant.

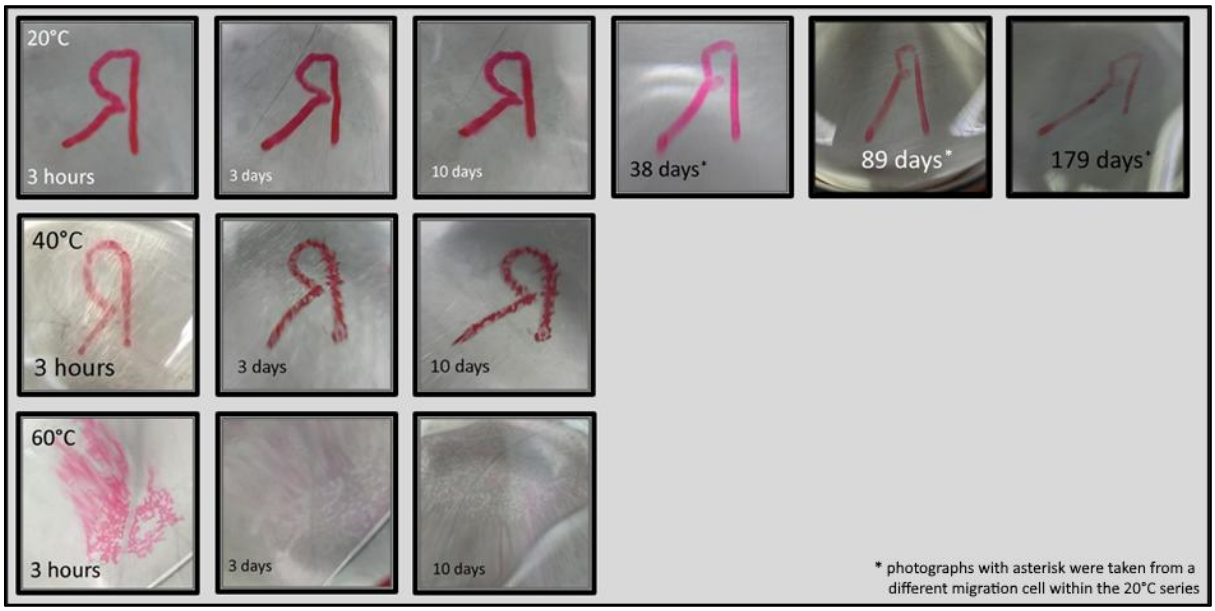

Figure 5: Photographs of the mark, at different timepoints, using 95% ethanol as the food simulant.

Appendix 1

Formula based on the Arrhenius equation, as referenced in regulation EU10/2011.

$$t_2 = t_1 * e^{9627K\left(\frac{1}{T_2}-\frac{1}{T_1}\right)}$$

Where;

- t_1 is the contact time
- t_2 is the testing time
- T_1 is the contact temperature in Kelvin.
- T_2 is the testing temperature in Kelvin.

Appendix 2 Supplemental data

The 50µm OPP substrate was marked with an “R” on the non-food contact side, placed face-down in a migration cell, and the food contact side was exposed to 50 ml of 95% ethanol. A 1:60 time-lapse recording was made during the cell’s 5-hour incubation at 60°C. The temperature of the ethanol was monitored throughout the incubation period, using a temperature probe in a migration cell containing 50 ml of 95% ethanol. The following observations were made (Figure 6):

- After 30 minutes (51°C), the “R”-mark was observed to fade. This may be the first indication that the food simulant is penetrating the substrate.
- After 80 minutes (60°C), the “R”-mark started to diffuse. At this time it is irrefutable that the food simulant is in contact with the “R”-mark.

From these observations, we conclude that the food simulant first contacts the mark on the reverse side of the substrate after only 30’ to 80’. Using the formula in Appendix 1, one can calculate that incubation for 80’ at 60°C would correspond to a storage at 20°C for 2.9 days. Comparing to the actual incubation at 20°C (Figure 5), no dissolution of the mark was observed, even after 179 days. **This indicates that the test conditions at 60°C effect changes to the substrate which do not occur under the realistic conditions.**

The recording of the full time-lapse can be accessed by scanning the QR code in Figure 6, or via the link https://www.youtube.com/watch?v=rE178mcHTs0. The time-lapse recording is sped up 60x; one second of video corresponds to one minute of incubation.

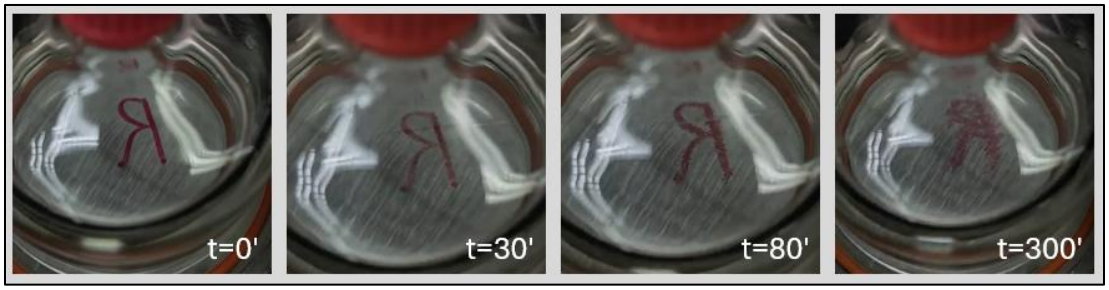

Figure 6: Stills of the time-lapse recording of the migration cell at 60°C, using 95% ethanol as the simulant. The QR code links to the full time-lapse video.

The incubation was stopped after 5 hours, after which the food simulant was discarded. Upon removal of the substrate from the migration cell, it was observed that most of the “R”-mark had transferred from the substrate to the steel carrier disk (see Figure 7). This observation demonstrates that the colourant stays on the non-food contact side while it is the simulant which is mobile.

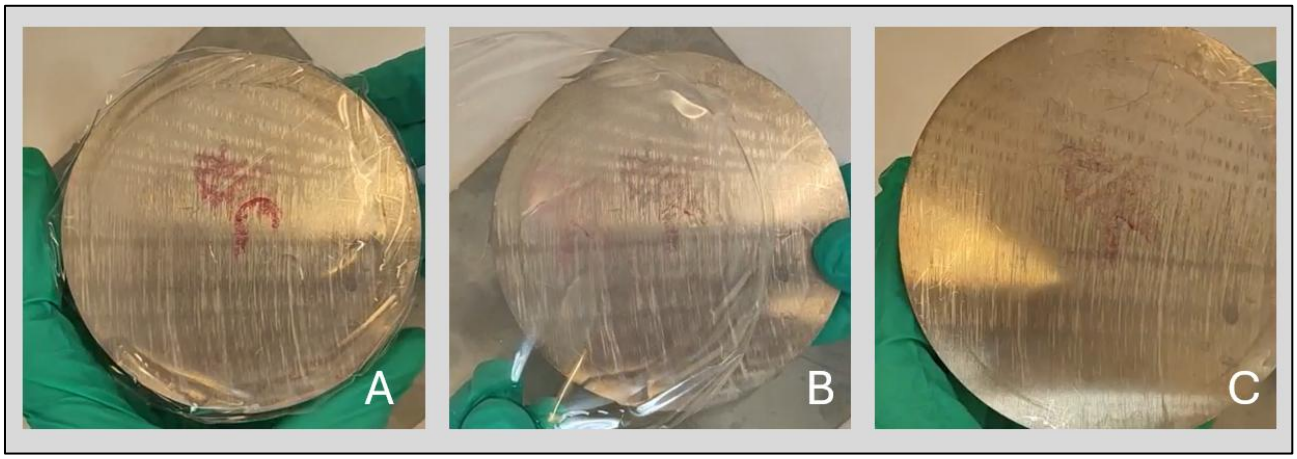

Figure 7: Removal of the substrate from the migration cell after 5 hours of incubation at 60°C with 95% ethanol as the food simulant. A: substrate and steel disc after opening the cell. B: the substrate is removed. Although some colourant is still present on the substrate, most has been transferred to the steel disc due to dissolution of the mark. C: substrate completely removed, showing most of the mark having been transferred to the steel disc.