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Henkel Adhesive Technologies

WHITE PAPER

MIGRATION AND MIGRATION TESTING

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Globally relevant content

CONTENT

Simulating the Contact of Packaging Material with Food

A wide range of standardized migration tests has been established in order to determine if food packaging really is food-safe. The contact of packaging material with foodstuff is simulated with different test setups that take into account the intended food contact conditions as well as the nature of the food contact material.

Migration Testing

Regulation (EC) No 1935/2004, the Framework Regulation, applies to all food contact materials and articles. Article 3 of this Regulation states that food contact materials and articles shall be manufactured so that they do not transfer their constituents to food in quantities which could a) endanger human health or b) bring about an unacceptable change in the composition of the food or c) bring about a deterioration in the organoleptic characteristics thereof.

To assess the transfer of constituents from the food contact material onto the packed food, migration tests can be carried out. These tests should represent the real conditions of use as best as possible in order to achieve the most realistic results possible. During migration, the packaging material is brought into contact with a food simulant and incubated for a certain period of time at a certain temperature. A suitable test setup can be arranged based on the following information:

- What kind of foodstuff will be packed?
- What are the time and temperature conditions of contact?
- Are there any special applications like hot filling, retort conditions or microwave treatment?
- What is the ratio of foodstuff to the surface of the food contact material (surface-to-volume ratio)?

Commission Regulation No 10/2011, the Plastics Regulation, is currently the most comprehensive guideline for migration testing. It defines relevant migration conditions like choice of food simulant, testing temperature and testing time.

According to the Plastics Regulation, the packed foodstuff is represented by certain food simulants. Examples are 3 % acetic acid to simulate acidic foodstuff like fruit juice or acidic dressings and 50 % ethanol to simulate dairy products or Modified Polyphenylene Oxide (MPPO, Tenax®) to simulate dry food like rice or frozen foodstuff. Further food simulants are 10 % ethanol, 20 % ethanol and vegetable oil, as well as the substitute simulants 95 % ethanol and isooctane.

As a general rule, migration tests are carried out under the worst forseeable contact conditions. Considering the real application, the longest forseeable contact time and the highest forseeable contact temperature are applied. Long-term storage above 30 days can be simulated in accelerated tests, e.g. storage above 6 months at room temperature or below can be simulated by migration for 10 days at 60 °C.

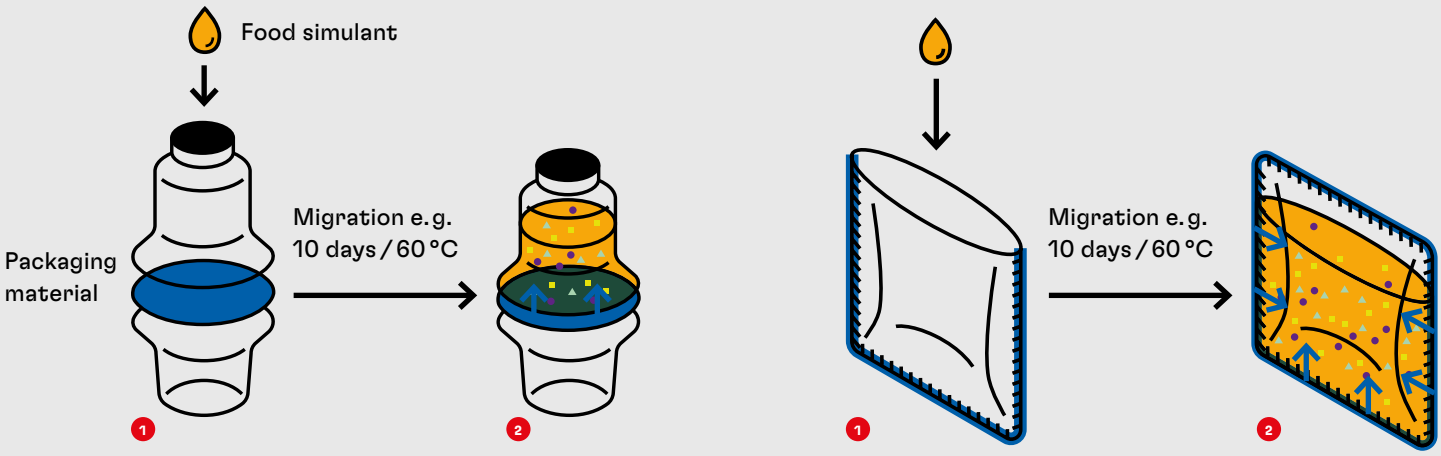
Special conditions like hot filling or retort need to be considered as well. A sterilization process followed by long-term storage at room temperature can be simulated, for example, by migration under retort conditions for a short time followed by migration at elevated temperatures for 10 days.

Migration Testing of Plastic Materials

For testing of plastic materials including plastic multilayer-materials and articles held together by adhesives (i. e. flexible packagings), detailed test conditions are laid down in the Plastics Regulation.

For plastic multilayer materials, contact between the packed food and the packaging material is only expected on one side: The food contact side of the packaging material. To simulate one-sided contact during migration testing, the packaging material is placed in migration cells and covered with the food simulant. Depending on the material and the intended conditions of use, it is also possible to seal pouches that can be filled with the food simulant. The migration cells or pouches, respectively, are then incubated for a certain period of time at a certain temperature.

Migration testing of a plastic film in migration cells or pouches
Figure 1



1. Laminate is covered with food simulant in a migration cell
2. Substances migrate from the laminate into the food simulant

1. Pouch is filled with food simulant and then sealed
2. Substrates migrate from the laminate into the food simulant

Migration Testing of Adhesives

Adhesives and coatings, as part of food contact materials, present a large and diverse group of substances. Due to their wide range of applications and complex chemistry, not all types of adhesives and coatings can be tested in the same way. In particular, they cannot be tested in the same way as plastic materials and articles. A comprehensive overview on migration testing of adhesives can be found in the FEICA Guidance “Migration testing of adhesives intended for food contact materials”.

Reactive polyurethane adhesives

Reactive polyurethane adhesives are predominantly used for lamination of plastic films, as well as other materials such as paper and aluminum. If used as a flexible packaging, bonded multilayer materials with at least one plastic layer are subject to the Plastics Regulation. As a result, the contribution of the polyurethane adhesive to migration must meet the overall and specifc migration limits.

In general, laminates where the food contact side is made of plastic can be tested according to the rules laid down in the Plastics Regulation. Since polyurethane adhesives are reactive systems, proper curing conditions have to be ensured prior to testing. To assess the impact on migration of the adhesive alone or of the individual layers of a multilayer material, each layer should be tested seperately.

Hotmelt adhesives

Hotmelt adhesives are usually used on paper and cardboard packaging. Direct food contact is not intended, the main transfer route of hotmelt constituents is via gas phase. In some cases, however, direct food contact cannot be excluded on seams and edges.

Testing procedures according to the Plastics Regulation are mainly based on liquid food simulants. Liquid simulants with high organic content may re-dissolve the low molecular weight fraction of the hotmelt adhesive, which the real food would not do. This leads to extraction rather than migration, which in turn might lead to a huge overestimation of migrating compounds. Testing with simulants with low organic content or Tenax® can be a suitable alternative.

Furthermore, testing at elevated temperatures, e. g. 60 °C, might cause softening of the hotmelt, leading to a physical change of the hotmelt’s properties and thus to overestimation of the real migration.

Testing at lower temperatures, i. e. temperatures not leading to softening of the hotmelt adhesive, as well as carefully choosing the right food simulant, may yield realistic migration results. Detailed testing conditions should be chosen depending on the real application.

Adhesives other than reactive polyurethane adhesives and hotmelt adhesives

Further adhesive and coating types include adhesives based on natural polymers, dispersions and emulsions based on poylmers such as polyvinyl acetate or polyacrylates, as well as heatseal and coldseal coatings.

If direct contact is assessed, adhesive films should be applied on a suitable substrate and dried prior to testing. Some substrates, however, cannot be tested with all kinds of food simulants. Aluminum foils, for example, will be dissolved during contact with 3 % acetic acid. In this case, the adhesive film should be applied on another suitable substrate.

Regardless of the substrate, the adhesive film might be redissolved when in contact with liquid food simulants. Due to the clear physical change of the adhesive film after dissolution, migration results strongly overestimate the real application and can only be considered for screening purposes.

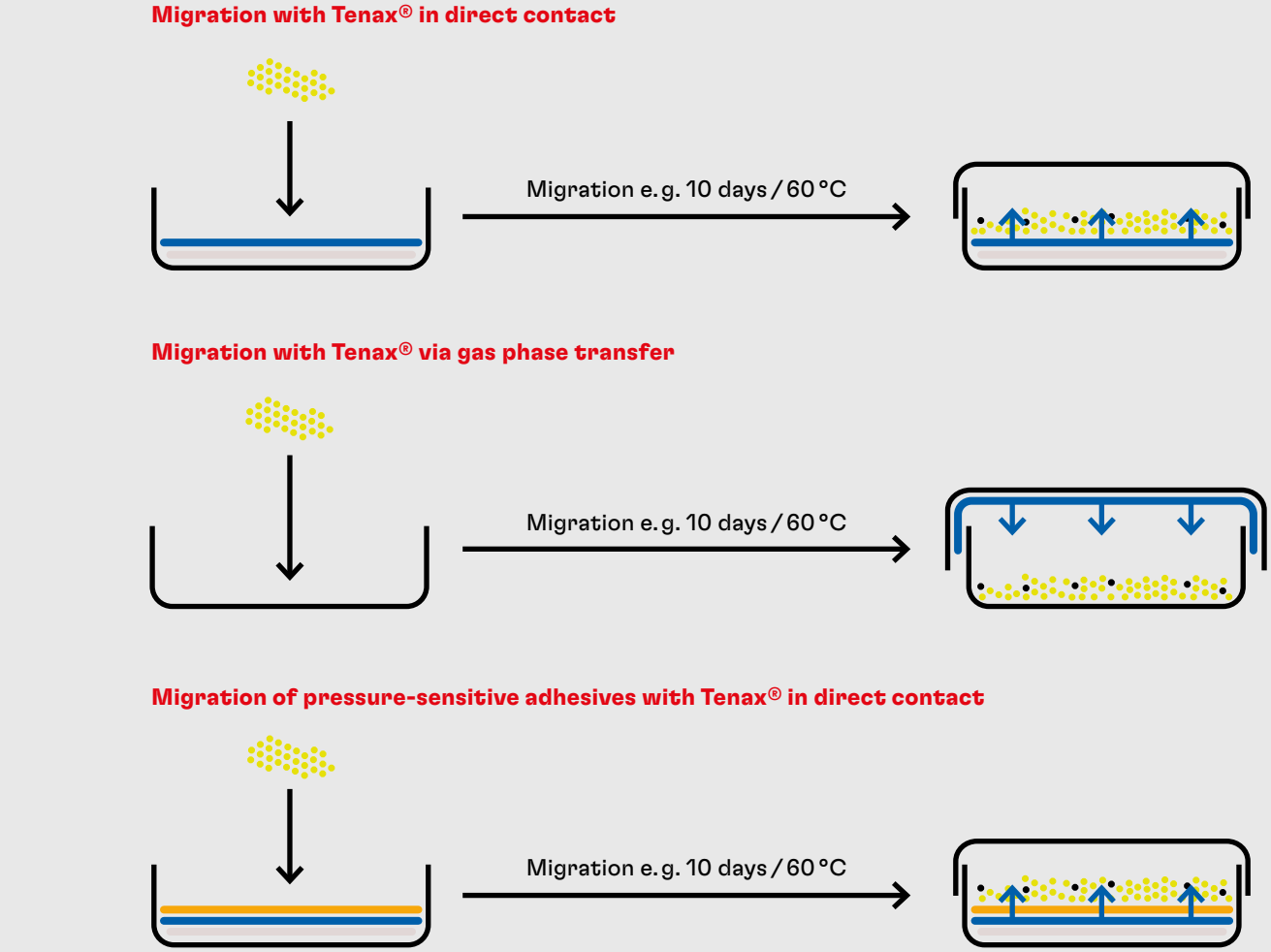
In paper applications, migration of constituents from adhesives mainly takes place via gas or vapor phase. Tenax® can be used as a simulant for testing of such gas phase transfer. In these tests, the adhesive and the simulant are separated by a certain distance – with no direct contact. This makes it possible to assess the transfer of gaseous constituents. For applications where direct contact cannot be ruled out, tests that involve direct contact between the adhesive and simulant (Tenax®) can be conducted.

A special test setup might be needed for testing of pressure-sensitive adhesives in direct contact with Tenax®. Since these adhesives remain very tacky, the dry food simulant Tenax® will stick to the adhesive and cannot be tested properly after migration. In this case, the pressure-sensitive adhesive should be applied on a substrate with no or marginal barrier properties. Tenax® can be applied on the substrate side and can be removed easily after migration.

Testing with Tenax® in direct contact can be performed in migration cells or in other suitable vessels like petri dishes.

In summary, there are no universal migration conditions for testing of adhesives and coatings. In general, the finished article should be tested to include effects of other parts/ layers of the food contact material besides the adhesive. The test setup should be chosen on a case-by-case basis to simulate the real application without changing the physical properties of the adhesive.

Migration testing with Tenax®
Figure 2



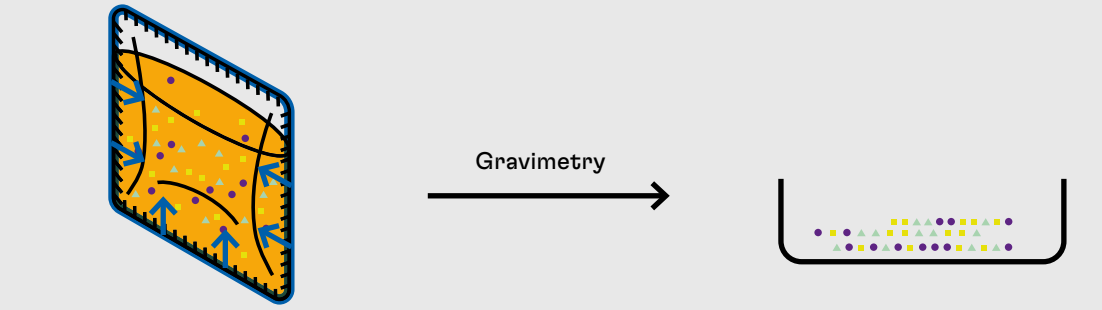
Analysis of Migration Solutions

Following migration, the migration solutions are investigated by suitable analytical methods.

The overall migration indicates the inertness of a food contact material. The maximum allowed migration of a plastic material, **the overall migration limit** (OML), is 10 mg/dm^2 as laid down by the Plastics Regulation. It is checked by evaporation of the food simulant and gravimetrical determination of the residues.

The weight of the residues indicates stability of the food contact material with respect to the filled foodstuff. However, it gives no information about the identity or toxicological impact of the migrating substances.

Gravimetrical determination of overall migration
Figure 3

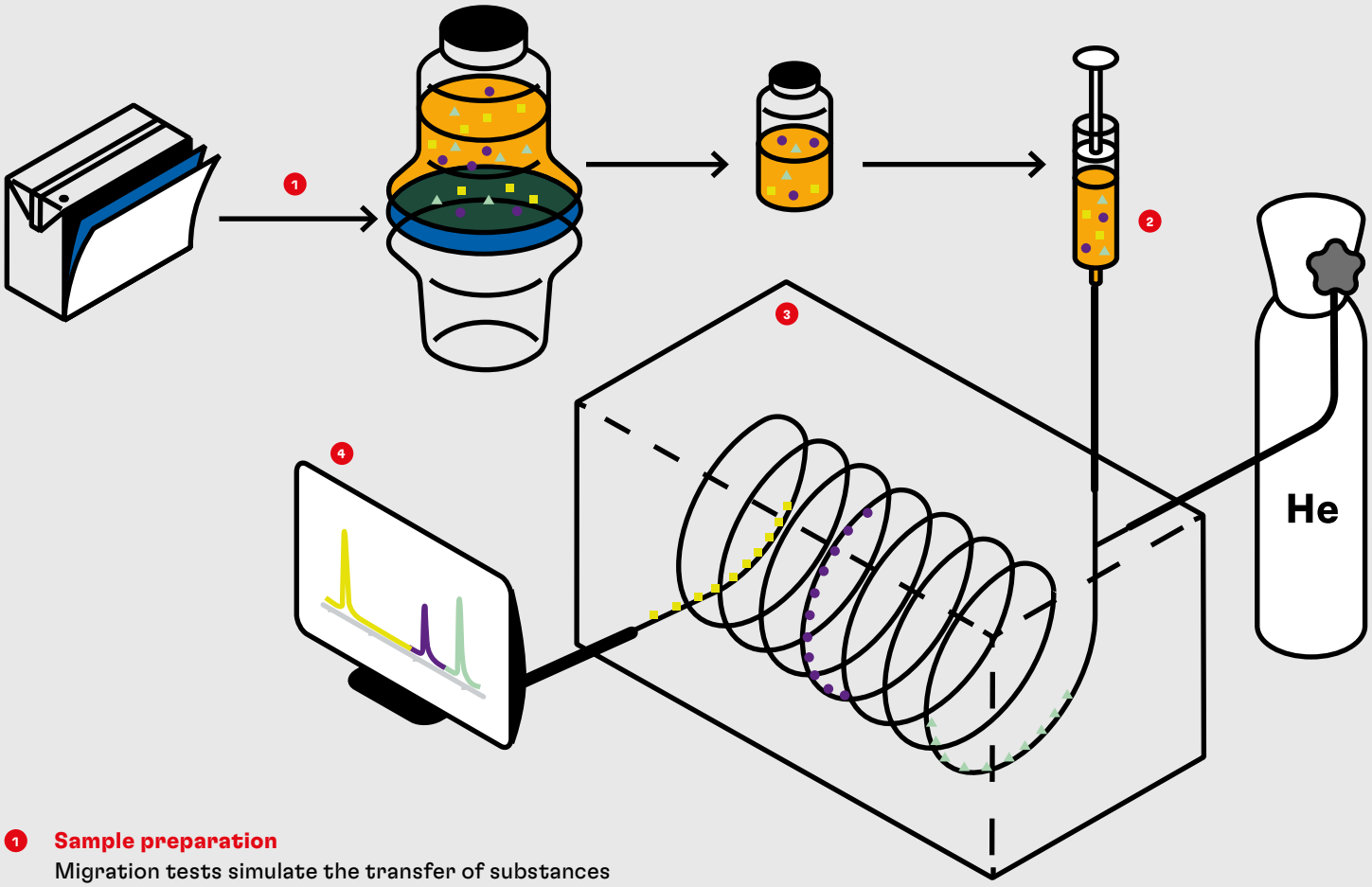


1 Substances migrate from the laminate into the food simulant

2 Evaporation of solvent, weigh-out of solid residues

Specific migration limits (SML) are given for individual substances or groups of substances (SML(T)). The specific migration is checked by specific analytical techniques that offer information about the identity of the compounds, as well as their concentration in the migration solution. Chromatographic methods like gas chromatography or liquid chromatography can be used for this purpose. An aliquot of the migration solution is transferred onto a chromatographic column where the individual compounds are separated depending on their chemical and physical properties. Comparison with reference substances allows for identification and quantification via retention time and peak area.

Determination of specific migration via chromatographic methods
Figure 4



1 **Sample preparation**
Migration tests simulate the transfer of substances from packaging materials into packaged food.

2 **Injection**
A very small sample amount, just 1 microliter in most cases, is injected into the GC and vaporized in the injector.

3 **Separation**
Separation of the compound into its individual components takes place in the capillary column. The amount of time an analyte spends in the column depends on its boiling point and the polarity of the compound.

4 **Evaluation**
After separation, the components are detected and displayed in a chromatogram (intensity versus time).

Evaluation

Migration results are usually given in mg of migrating substance per dm^2 of packaging material. The overall migration limit is 10 mg/dm^2. It can be directly compared to the migration results and applies to plastic materials or multilayer materials with at least one plastic layer that is held together by laminating adhesives.

Specific migration limits are indicated as mg of substance per kg of food. In order to convert migration results from mg/dm^2 to mg/kg of food, the real ratio of packaging surface to packed foodstuff has to be considered (surface-to-volume ratio). For sheets and films that are not yet in contact with food, a standard ratio of 6 dm^2/kg is assumed (EU standard cube).

Knowing the kind of foodstuff, intended time and temperature conditions, as well as the real surface-to-volume ratio, migration testing can be performed to assess migration of constituents under real conditions of use.

Key take aways

- Henkel's analytical department has established a wide range of standardized migration tests in order to determine if food packaging is food safe.
- Migration test setup is based on the following information: Kind of foodstuff, time and temperature conditions of contact, special applications (where required) and surface-to-volume ratio.
- For testing purposes, the packaging material is brought into contact with the food simulant and incubated for a certain period of time at a certain temperature.
- Adhesives cannot be tested in the same way as plastic materials. Applying conditions of the Plastics Regulation may lead to complete dissolution or other physical changes to the adhesive.
- To assess migration of adhesives, the test setup should be chosen on a case-by-case basis to represent the real application as best as possible.
- Following migration, the overall migration limit (OML) is checked by evaporation of the food simulant and gravimetrical determination of the residues. The specific migration limits (SML) can be tested via specific methods like chromatography.

References

Regulation (EC) No 1935/2004 of the European Parliament and of the Council of 27 October 2004 on materials and articles intended to come into contact with food and repealing Directives 80/590/EEC and 89/109/EEC (L 338, p.4)

Commission Regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food (L 12 p. 1)

FEICA Guidance Paper "Migration testing of adhesives intended for food contact materials", 27 May 2016, publication ref.: GUP-EX-F03-010

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Dr. André Weiss has a professional background in food chemistry and food law. Since 2015, he has worked in Henkel's Corporate Analytics team. As project manager for food safety, he is responsible for migration testing and the evaluation of food contact materials. His work places a special focus on adhesives and coatings.

Thank you.

Contact

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