The European voice of the
adhesive and **sealant industry**

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Migration testing of adhesives intended for food contact materials

FEICA, the Association of the European Adhesive & Sealant Industry, is a multinational association representing the European adhesive and sealant industry. With the support of its national associations and several direct and affiliated members, FEICA coordinates, represents and advocates the common interests of our industry throughout Europe. In this regard, FEICA aims to establish a constructive dialogue with legislators in order to act as a reliable partner to resolve issues affecting the European adhesive and sealant industry.

This document outlines the specific guidelines for conformity testing of adhesives as components of food contact materials. This guidance is provided by the FEICA Paper & Packaging Working Group for the benefit of users of food contact adhesives such as packaging producers and their downstream users as well as other stakeholders involved in regulatory or legislative matters of food contact.

To learn more about the guidance papers of the other sectors, refer to the information on the FEICA website at www.feica.eu/our-projects/food-contact.

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1 Introduction / Objectives

Food packaging and *food service articles* are everyday examples of *food contact materials and articles*. Food contact, however, occurs in a much wider group of articles, ranging, for example, from tableware, takeout and storage containers, articles used in food preparation to machinery and storage equipment used in industrial food processing.

While food contact materials are not *intended* to become part of food, the possibility of *incidental transfer of chemical substances* from food contact materials onto food ('migration') needs to be given attention. For a number of years, the EU has therefore set out harmonised legislation for *food contact materials and articles*, such as Regulation (EC) No 1935/2004 on materials and articles intended to come into contact with food (the '*Framework Regulation*') and Regulation (EC) No 2023/2006, related to good manufacturing practice (GMP).

Separate *material-specific measures* are foreseen to exist under the framework of Regulation (EC) No 1935/2004. Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food (the '*Plastics Regulation*') affects many articles on which adhesives are used. Other material groups, for example, packaging materials such as paper but also adhesives themselves are not (yet) governed by a specific harmonised legislation. These food contact materials remain subject to the *Framework Regulation* and, where applicable, to relevant EU Member State national measures.

Since the Plastics Regulation (EU) No 10/2011 provides an extensive list of evaluated and authorised substances as well as a correlation of food types and contact scenarios to migration test conditions, it is often used as a regulatory reference when adhesives are assessed. Experience shows that care needs to be taken with this approach to adhesives or adhesive-containing food contact materials, particularly with migration testing as described in the Plastics Regulation and articles, since adhesives are *not* plastics.

This document, therefore, outlines specific guidance for the conformity testing of adhesives as (components of) food contact materials and articles. The need for such specific guidance results from the specific properties and the nature of adhesive applications in food contact materials and articles, which differ from other material groups, including plastics.

Following this guidance will support adhesives users in demonstrating that their products can fulfil the requirements of EU food contact regulations and will help to provide adequate information to their customers for the safe use of their products.

For more details of how to *choose* a suitable adhesive for application in food contact materials, adhesive users may consult separate FEICA Guidance.[1]

[1] FEICA Guidance paper 'Guidance for a food contact status declaration for adhesives'

2 Adhesives in food contact materials and articles

Adhesives are additive components in the manufacture of food contact materials and articles. They generally do not represent the structural element nor the bulk of the final product. Compared to the main food contact materials, such as plastics and paper, adhesives typically make up only a small part of a food contact article, often less than5% by weight

In another differentiation from plastics and paper, for most adhesive applications, a direct food contact is not intended. Adhesives are used to bond two materials together. It is these materials, such as plastic films, paper or other materials, that face the foodstuff.

The main pathways for the transfer of chemical substances from adhesives in indirect food contact scenarios are different from the situation for the main food contact material which is in direct contact. In some adhesive applications, the substrates may form a functional or absolute barrier, [2] separating the adhesive fully from the filling. In other adhesive applications, transfer may occur by migration through the different layers of the food contact article due to unintentional contact at seams and edges and/or by gas phase transfer of volatile compounds.

It should be noted that a limited number of specific direct-contact adhesive applications do exist in the form of heat seals and of pressure sensitive adhesives for direct food labelling, e.g., for fruit stickers. Migration testing of these adhesive applications should employ specific conditions.

Considering the low quantity of adhesives in a food contact article and their typical use in indirect contact, their contribution to overall migration[3] is typically low compared to that of the main materials in the food contact article. Consequently, specific migration testing (i.e., for individual, low migration limit compounds that may originate from adhesives) is generally of greater relevance than determining the contribution to overall migration originating from an adhesive.

Adhesive (general definition) 'An adhesive is a non-metallic substance capable of joining materials by surface bonding (adhesion), and the bond possessing adequate internal strength (cohesion).'[4] Adhesives set by either evaporating a solvent or cooling, or they cure by chemical reactions that occur between two or more constituents.[5]

[3] Overall migration was established as a measure of inertness of plastic materials in contact with foodstuff and therefore does not translate well to adhesive applications. Overall migration is a measure of the total mass of migrating substances but not their identity; it therefore contains no information of toxicological relevance.
[4] EN 923:1995, Adhesives — Terms and definitions, 2.1.1 adhesive
[5] Hermann Onusseit, Rainer Wefringhaus, Gunther Dreezen, Jürgen Wichelhaus, Joel Schall, Lothar Thiele and Ansgar van Halteren 'Adhesives, 1. General ' in Ullmann's Encyclopaedia of Industrial Chemistry 2010, Wiley-VCH, Weinheim. doi:10.1002/14356007.a01_221.pub3

3 Typical adhesive systems used in food contact applications

This section provides an overview of the types of adhesives that commonly can be found in food contact materials and articles and describes their typical applications.

Reactive polyurethane (PU) adhesives

Polyurethane adhesives are based on reactive chemistry where isocyanates react with polyols and/or water. The high thermal resistance of polyurethanes, including the ability to withstand retort[6] processes, results from chemical crosslinking. This crosslinking is achieved either using a two-component liquid polyurethane adhesive system or through moisture curing of a one-component reactive polyurethane adhesive. In the case of liquid adhesive systems, solvent can be added to one or both components to reduce their viscosity. Reactive PU adhesive systems may also be supplied in water-based form.

To ensure both technical performance as well as food contact safety, adequate curing conditions, according to the recommendations of the adhesive manufacturer, must be observed for reactive PU adhesives.

Reactive polyurethane adhesives are predominantly used for the lamination of different combinations of polymer films, aluminium foil and paper to form flexible packaging. The applications of the finished articles encompass all types of foodstuffs and include conditions of prolonged storage (> 6 months) at all temperatures from frozen and refrigerated storage (below 0 °C or below 4 °C) up to retort processing (up to 145 °C for up to one hour) as well as microwavable and ovenable packaging.

Adhesives based on natural polymers

Adhesives based on natural polymers are derived from either animal or plant material. In the case of animal sources, the polymeric substances are typically proteins, such as casein. In the case of plant sources, they are generally carbohydrates, such as starch or dextrins. Adhesives can also contain mixtures of animal- and plant-based polymers, which can further be combined with synthetic polymers as well.

Since natural polymers are typically hydrophilic, they are generally dissolved in water in the form of so-called colloidal solutions.[7] A special case is natural rubber latex, which is not hydrophilic and forms a dispersion in water. The setting of the adhesive occurs in either case by evaporation of water.

Adhesives based on carbohydrates such as dextrins and starches are mainly used for producing paper- and cardboard-based packaging for dry foodstuff and secondary and tertiary fibre-based packaging. Conditions of use include prolonged storage (> 6 months), generally from frozen and refrigerated storage (below 0 °C or below 4 °C) to room temperature. Direct contact is not intended but cannot be excluded on seams or edges.

Protein-based adhesives are used for glass bottle labelling and as wet laminating adhesives in the production of aluminium foil / paper laminates used for flexible packaging, spiral wound tubes and laminated lids. When protein-based adhesives are used on glass, which forms an absolute barrier, all kinds of foodstuffs may be packed. When these adhesives are used for paper laminates, foodstuffs in contact are generally dry but may contain fat on their surface. Conditions of use include prolonged storage (> 6 months), generally from frozen and refrigerated storage (below 0 °C or below 4 °C) to room temperature.

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[6] High temperature sterilisation by steam or water immersion.

[7] A mixture that has particles ranging between 1 and 1,000 nanometres in diameter yet still able to remain evenly distributed throughout the solution.

Cold seal adhesives are generally based on natural rubber latex and are used to seal the edges of packaging made from a range of flexible substrates such as paper and plastic films. As pressure-activated systems, no heating is required to obtain the seal: two cold seal coated sides need only to be pressed together. The absence of heat during sealing makes cold seals ideal for packaging temperature-sensitive foodstuff such as ice cream, chocolate, or biscuits. Foodstuffs in contact are generally dry but include foodstuffs with fat on their surface. Conditions of storage include prolonged storage (> 6 months) and temperatures from frozen and refrigerated storage (below 0 °C or below 4 °C) up to room temperature. Direct contact is not intended but cannot be excluded on seams or edges.

Water-based adhesives based on synthetic resin dispersions, emulsions and solutions

Water-based adhesives can be produced from a range of synthetic polymers and are available in three main forms: polymer *dispersions* and polymer *emulsions* are systems in which polymer particles are delivered in water as a carrier medium - In water-based *solutions*, the polymers are dissolved in water and therefore not present in the form of particles.

Adhesives based on Polyvinyl acetate (PVAc) or ethylene vinyl acetate (EVA) copolymers

Polyvinyl acetate (PVAc) and ethylene vinyl acetate (EVA) copolymer-based adhesives are available as dispersions, emulsions or solutions in water. They are mainly used for producing paper- and cardboard-based packaging for dry foodstuff and for secondary / tertiary fibre-based packaging. PVAc-based and EVA copolymer-based adhesives are also used for wet lamination of paper to foil. Foodstuffs in contact are generally dry. Conditions of use include prolonged storage (> 6 months), generally at or around room temperature. Direct contact is not intended but cannot be excluded on seams or edges.

Certain EVA copolymer dispersions are also used as heat seals (direct food contact) on aluminium foil, and on polyester and various polyethylene and polypropylene films, used for lidding applications. Foodstuffs are generally dry or liquid and may contain fats. Conditions of use include prolonged storage (> 6 months), generally at temperatures from frozen and refrigerated storage (below 0 °C or below 4 °C) up to room temperature. For heat seals used for tray and cup lidding, direct contact with the foodstuff may occur but is typically not intentional.

Adhesives based on acrylic polymers and copolymers, including styrene acrylate terpolymers

Acrylic polymers, copolymers or terpolymers are available as dispersions, emulsions or solutions in water. They are mainly used for paper- or cardboard-based packaging of dry foodstuff or for secondary / tertiary fibre-based packaging, applied on different substrates such as paper, cardboard or polymer films. Conditions of use include prolonged storage (> 6 months), generally at or around room temperature. Direct contact is not intended but cannot be excluded on seams or edges.

Acrylic polymers, copolymers or terpolymers are also used for the lamination of different combinations of polymer films and paper to form flexible packaging. The applications of the finished articles encompass all types of foodstuffs and include conditions of prolonged storage (> 6 months) at all temperatures from frozen and refrigerated storage (below 0 °C or below 4 °C) up to room temperature. When crosslinked with a reactive second component, laminates may be suitable for elevated temperature processing such as hot-fill, boil-in-bag or retort as well as microwavable packaging.

Ethylene acrylic acid (EAA) and ethylene methyl acrylic acid (EMAA) dispersions are used for wet lamination of paper to foil and for lidding applications where the lidding material is aluminium foil, polyester, or polyethylene and polypropylene film. Conditions of use include prolonged storage (> 6 months), generally at temperatures from frozen and refrigerated storage (below 0 °C or below 4 °C) up to room temperature. For heat seals used for tray and cup lidding, direct contact with the foodstuff may occur but is typically not intentional.

Commonly, acrylic dispersions are also used to produce pressure sensitive adhesive applications in the production of self-adhesive labels. Adhesion can be permanent, or the label can be (easily) peelable by hand. Pressure sensitive applications are also used for reclose features in snack, meat and cheese packaging and re-closable lids. Foodstuffs are generally dry but may contain fat on their surface. Conditions of use include prolonged storage (> 6 months), generally at temperatures from refrigerated storage (below 4 °C) up to room temperature. Direct contact is not intended in most labelling applications but may occur in reclose applications.

Acrylic polymer dispersions may also be used in direct food contact in direct labelling of foodstuff, e.g., fruits and vegetables. Conditions of use are generally limited to short storage times (natural shelf-life of produce), generally at temperatures from refrigerated storage (below 4 °C) up to room temperature.

Synthetic resin-based heat seals

Heat sealing refers to the combining of two substrates via heat and pressure. This process is predominantly used for lidding applications, where paper, aluminium or plastic lids are bonded to plastic food containers, for example, in the packaging of dairy products, for plastic trays for convenience food and meat, and for instant noodle cups.

Heat seals are in most cases non-reactive synthetic resin-based products, delivered in solvent, and are applied and dried on one or both substrates that will later be sealed together. Substrates include polyester, polyethylene, polypropylene films and aluminium foil.

Resins used in solvent-based heat seals include polyester, acrylic and vinyl resins, and may also include nitrocellulose. Heat seals can also contain hydrocarbon or rosin or modified rosin-based materials as tackifiers plus natural and synthetic waxes.

Foodstuffs are generally dry or liquid and may contain fats. Conditions of use include prolonged storage (> 6 months), generally at temperatures from refrigerated storage (below 4 °C) up to room temperature.[8] When used as the sealing layer of pouches, heat seals are intentionally in contact with foodstuff. When heat sealing is used for tray and cup lidding, direct contact with the foodstuff may occur but is typically not intentional.

(Non-reactive) Hotmelt adhesives

Non-reactive hotmelts adhesives belong to the group of physically setting adhesives, where the setting takes place without any chemical change to the polymer. Before application, the adhesive is heated to a melt, typically at temperatures above 100 °C. The adhesive is then applied in liquid (molten) form to the substrate. A physical setting takes place during cooling and turns the hotmelt back into a crystalline or amorphous solid.

[8] Special cases exist where heat seals are used on PET film to create pouches suitable for elevated temperature processing such as boil-in-bag, microwavable or ovenable packaging for meals.

Polyolefins as wells as ethylene vinyl acetate copolymers (EVA) are the main polymers that are employed in (non-reactive) hotmelt adhesives. Traditionally, hotmelts are based on high levels of waxes and low levels of polyolefin copolymers with low softening point, such as EVAs, and tackifiers. Many newer types of hotmelts mainly consist of ethylene vinyl acetate (EVA) or polyolefin (PE, PP) copolymers and have a higher softening point. They contain low levels of waxes and tackifiers. In addition, acrylic or styrene (co)polymers (e.g., SBS, SIS) may be added.

Hotmelts are applied on paper and cardboard packaging for dry foodstuff (including bags and sacks) and for secondary / tertiary fibre-based packaging. Conditions of use include prolonged storage (> 6 months), generally at temperatures from frozen and refrigerated storage (below 0°C or below 4°C) up to room temperature. Direct contact is not intended but cannot be excluded on seams or edges.

4 General considerations for migration testing adhesive applications

Due to the wide range of applications and their varied chemistry, no single unified testing condition can be defined that would apply equally for all adhesive applications. Additionally, in many cases, the commonly used migration testing defined for plastics by Regulation (EU) No 10/2011 cannot be used on adhesive applications. Instead, an adhesive-specific testing approach is typically required.

This section provides general considerations for the migration testing of adhesives whereas the following section will provide detailed guidance for the testing of specific adhesive types and applications. At the end of this section, a decision tree provides an overview of the key considerations when adhesives are tested.

No compliance testing of adhesives on their own

In terms of the requirements of Regulation (EU) No 1935/2004, it is the finished food contact material or article that is to be evaluated for safety, consideration being given to its actual conditions of use. Testing a single component of a food contact article, such as an adhesive, on its own can therefore be considered only as a screening tool,[9] not as something to demonstrate compliance or non-compliance. It is therefore recommended to perform compliance testing of adhesives applications in the finished article wherever possible.

Sufficient and representative application and adequate curing of reactive adhesives

Adhesives should never be tested for migration in their delivery form but in applied form. The sample to be analysed should be representative of the actual food contact material or article, including the amount and form of adhesive as well as the conditions of application (e.g., drying).

For reactive adhesives, it must further be confirmed that the curing conditions of the adhesive(s) contained in migration testing samples comply with the recommendations of the adhesive manufacturer. Testing not fully cured adhesive applications will provide erroneous results.

Consideration of the substrate's migration profile

When the recommended testing approach is carried out on an adhesive that is applied to a substrate (the main packaging material), the substrate (e.g., paper or plastic) itself needs to be investigated thoroughly. Migration from the substrate must be evaluated separately in order to differentiate the substrate's contribution to migration from that of the adhesive (generation of a 'blank' without adhesive).

Adhesives used in or on plastic food contact materials

The scope of the Plastics Regulation (EU) No 10/2011 covers food contact materials and articles both when made solely of plastic and when plastic is combined with other materials. It therefore applies also to plastic materials and articles that are bonded with adhesives.

[9] In specific cases where a worst-case calculation of an adhesive application's contribution to migration does not demonstrate safety or cannot be carried out due to missing compositional information, direct testing of adhesives may be considered in order to obtain worst-case migration information. Special care, however, needs to be taken that test conditions are suitable for a direct contact with the adhesive and do not lead to exaggerated results due to dissolution or decomposition of the adhesive. Attention to this aspect needs to be paid especially in cases where the actual adhesive application occurs behind a functional or absolute barrier.

Care, however, needs to be taken when applying migration testing as described in the Plastics Regulation to adhesives or adhesive-containing food contact materials since adhesives are *not* plastics.

As the Plastics Regulation is binding for the plastic layers in multi-layer and/or multi-material articles but not for the adhesive layer(s), migration testing of adhesive applications may deviate from the conditions mandated for plastics. Although the migration testing conditions provided in the Plastics Regulation can in certain cases successfully be applied to the whole packaging (adhesive included), in other cases, it is advisable to adjust the testing conditions based on the remarks in this guidance.

> *Regulation (EU) No 10/2011, Article 2(1):*
> This Regulation shall apply to materials and articles which are placed on the EU market and fall under the following categories:
>
> *(a) materials and articles and parts thereof consisting exclusively of plastics;*
>
> *(b) plastic multi-layer materials and articles held together by adhesives or by other means;*
>
> *(c) materials and articles referred to in points (a) or (b) that are printed and/or covered by a coating;*
>
> *(d) plastic layers or plastic coatings, forming gaskets in caps and closures, that together with those caps and closures compose a set of two or more layers of different types of materials;*
>
> *(e) plastic layers in multi-material multi-layer materials and articles.*

Adhesives used in or on paper-based food contact materials

No EU-wide material-specific regulation for paper and board exists at the time of writing. The Council of Europe has issued Resolution AP(2002)1 titled 'Paper and board materials and articles intended to come into contact with foodstuffs' and a Technical Guide under Resolution CM/Res(2020)9 titled 'Paper and Board Used in Food Contact Materials and Articles'. The latter suggests options for the migration testing of materials and articles based on paper and board. These conditions include the use of two food simulants also used by the Plastics Regulation, namely, Tenax® (Poly[2,6-diphenyl-p-phenylenoxid]) and 3% acetic acid.

In paper and cardboard packaging for dry foods, migration of adhesive ingredients into foodstuff is largely confined to gas phase (vapour phase) transmission. This migration pathway is adequately simulated using Tenax® as a food simulant.[10]

Experience shows that adhesive applications on paper / cardboard can typically not be tested for migration board with simulants such as 3% acetic acid (as well as ethanolic solutions) as liquid simulants may extract or even dissolve adhesive applications which are only separated from the liquid simulant by a permeable paper layer. As this may lead to unrealistically high migration results

[10] In addition, headspace analysis of the adhesive may be applied as a first screening tool for determination of volatile components and their risk assessment.

or analytical artifacts (see further below), testing with the dry simulant Tenax® should be favoured for adhesive applications on board and paper.

Typically, Tenax® is not applied directly onto the food contact surface but placed within a certain distance to simulate vapour phase transition. When direct contact of an adhesive with the foodstuff cannot be excluded in the actual application, migration may, however, be performed in direct contact to obtain worst-case results.[11]

Migration time and temperature should be chosen according to the expected conditions of use.

Accelerated tests at elevated temperature

Certain sets of migration conditions as defined by the Plastics Regulation (EU) No 10/2011 apply elevated temperatures that do not reflect the real conditions of use but constitute an accelerated test.[12] For example, conditions of 60 °C for 10 days are often utilsed as an accelerated test for prolonged storage (> 6 months) conditions at room temperature.

Accelerated tests are a very useful tool to reduce testing time and provide migration results in a timely manner. Anytime accelerated test conditions lead to a change of the physical properties of the food contact material compared to the properties it exhibits in the actual food contact scenario, however, results should be evaluated with caution.

For certain types of adhesive applications, a change of physical properties will take place at temperatures of 60 °C. The observed migration at 60 °C will in such cases be substantially different from the real long-term migration at room temperature or even at 40 °C. In these cases, the migration results obtained may not be valid, and may be typically higher than the worst-case scenario at real contact conditions.

In cases where there is doubt concerning the validity of accelerated migration test results, the test should be repeated using the actual contact temperature and contact time expected from the intended application.

Weakening of the packaging structure through high temperatures and simulants

The testing of flexible packaging under hot-fill, boil-in-bag, or retort conditions, as well as under accelerated testing conditions, may lead to a decrease of bond strength with liquid food simulants. If the food contact article's structure is weakened substantially or even destroyed by the simulant, test results should be evaluated with caution.

In general, whenever a high temperature migration test with a simulant leads to damage or destruction of a food contact article that is fit for use in its intended application, testing may need to be repeated under the actual conditions of use and/or with the actual foodstuff rather than with simulants to ensure valid results. Adhesive users are encouraged to reach out to the adhesive manufacturer for advice on the correct application and testing of the adhesive for high-temperature applications.

[11] See section 5 for specific consideration for pressure sensitive adhesive applications.
[12] This approach, which is based on the Arrhenius' law, is explained in Annex V, Chapter 2 of Regulation (EU) No 10/2011.

False-positive results due to high temperatures occurring in analytical techniques

A migration test generally leads to a liquid sample which contains the migrated substances, and which is subjected to chemical analytical techniques to identify and quantify the observed migration.

The technique of gas chromatography coupled with mass spectrometry (GC-MS) has developed into the most common way of obtaining an overview of the migration profile of food contact materials and is today the most used migration screening methodology.

When analysed via gas chromatography (GC), the migration sample is heated up to more than 200 °C, which may lead to the creation and detection of thermal decomposition products. Such results are analytical artefacts as the decomposition products are generated artificially in the analytical device rather than being themselves present in the original sample.

The understanding of thermal decomposition plays a pivotal role in the evaluation of migration test results for adhesives that are obtained via GC-MS screenings. Findings of possible breakdown products should be verified by changing the temperatures within the analytical system. Any change of the detected substance amounts with changing temperature may be an indication of thermal decomposition falsifying the result.

Due to the wide variety of substances used in food contact materials and adhesives, it is not possible to define a single critical temperature that, when exceeded, will result in thermal decomposition. As a rule, thermal impact close to and above 100 °C should be evaluated carefully.[13]

Extraction of polymers and oligomers and related false-positive monomer results from extracted constituents

In extension of the considerations of the effect of high temperature analytical techniques, special attention must be paid to testing conditions that may give rise to extraction of polymers or oligomers from adhesive applications.

Depending on the technique, oligomers and polymers extracted by the simulant or solvent may be prone to thermal breakdown during analysis, which may lead to (additional) false-positive detection of breakdown products and incorrect residual monomer quantification results. Polymers that are prone to producing analytical artefacts through thermal decomposition include:

- Polyurethanes: false-positive detection of isocyanates, polyols possible
- Polyesters: false-positive detection of polyols possible
- Polyacrylates: false-positive detection of acrylate monomers possible
- Styrene-containing resins: false-positive detection of styrene monomers possible

This issue is particularly prevalent when residual content measurements as described in the Plastics Regulation are performed. In these measurements, strong solvents such as dichloromethane are used to achieve total extraction of the food contact material. In case an adhesive application is covered by a plastic layer, the food contact material is typically shredded so as to maximise the solvent's contact surface along the edges of the flakes. Under these severe conditions, not only the residual monomers which are the subject of the test but also oligomers and polymers may be dissolved from the sample.

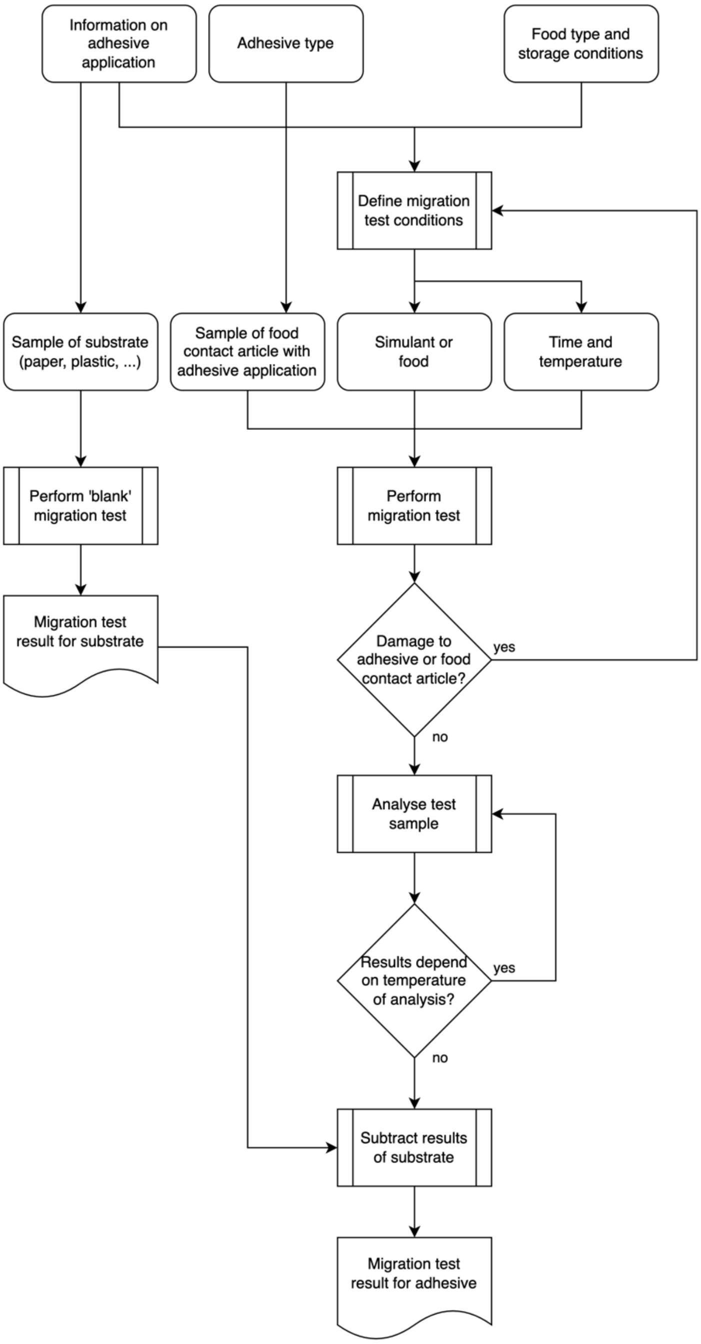
Organic solvents and those food simulants described in Regulation (EU) No 10/2011 that contain ethanol or oil may also extract oligomeric or polymeric parts from an adhesive application in a

[13] There are injection techniques in place that offer injection at relatively low temperatures (e.g., on-column injection). Liquid chromatography (e.g., HPLC = high pressure liquid chromatography) generally involves no thermal impact and should be considered as first choice for the quantification of known substances.

migration testing setup, unless the adhesive is behind a plastic or aluminium foil layer. Actual foodstuff will typically possess a lower potential for extraction of oligomers and in particular polymers from adhesive applications.[14]

Summary: Successful migration testing of adhesive applications

The flow chart in Figure 1 shows how migration testing of adhesive applications can be defined and performed while including the considerations stated above.



[14] See, for example, the FEICA-commissioned 2021 study 'Extraction, migration simulation and storage test regarding oligomeric hydrocarbons from hotmelt adhesives used in cardboard packaging' by Laboratory Lommatzsch & Säger.

Figure 1: Recommended flow of defining and executing migration testing of adhesive applications.

Diagnosing migration test results for adhesive applications

The flow chart in Figure 2 shows how migration testing results for adhesive applications can be diagnosed for possible issues that may cause erroneous results.

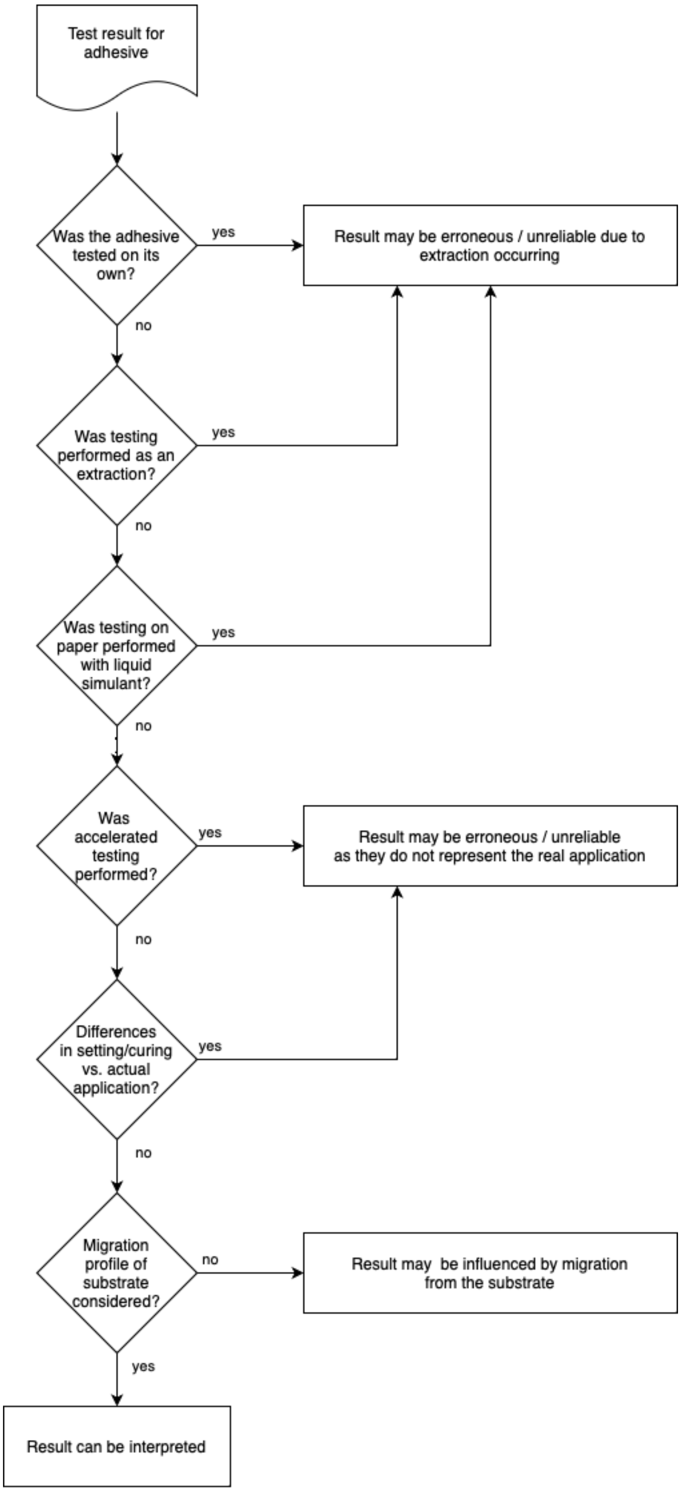


Figure 2: Flow of diagnosing a migration test result for adhesive applications.

5 Material and application-specific considerations when migration testing adhesive applications

This section provides specific considerations for the migration testing of certain types of adhesives. All considerations provided in the preceding section still do apply and should be taken into account in addition to the specific points made in this section.

Reactive polyurethane (PU) adhesives

Polyurethane adhesives are typically used to laminate the different layers of multilayer flexible materials which are used in flexible packaging for foodstuff. These types of packaging are typically subject to and tested according to Regulation (EU) No 10/2011. The following material-specific properties of adhesives have to be considered when laminates for the adhesive contribution to migration are tested.

Reaction with food simulants when determining residual isocyanate or primary aromatic amines

When residual isocyanate (monomer) content in a PU application is determined, it has to be ensured that the employed food simulant does not react with isocyanates. For example, ethanol will react with isocyanates to form carbamates. As a consequence, a residual isocyanate content cannot be determined (as such) with ethanol-containing simulants.

Determination of residual isocyanates should therefore be performed using inert solvents such as dichloromethane to prevent side reactions of residual isocyanate monomers. High purity grades of solvents must be employed since less pure grades may still lead to side reactions due to contained impurities or residual moisture. If an inert solvent cannot be used, e.g., specific food simulants are mandatory or significant residual impurities cannot be avoided in the solvent, the applied analytical method has to be capable of detecting and determining the amount of the reaction side-products. For example, determination of residual isocyanates in ethanol-containing simulants can be achieved by quantifying the carbamates they form with ethanol.

As the formation of carbamates from reaction with ethanol is favoured over the hydrolysis by water, determination of primary aromatic amines (PAA) from hydrolysis of isocyanates is disturbed in the presence of ethanol in food simulants. Determination of primary aromatic amines should therefore preferably be performed with 3% acetic acid as the food simulant. For food contact articles used at ambient conditions, it is generally accepted that 70 ˚C for 2 hours ensure hydrolysis of aromatic isocyanates.[15,16]

It should be considered that where laminates are subjected to elevated temperatures in their actual use, e.g., boil-in-bag or retort processing, there is a possibility of thermal degradation of the fully cured adhesive (polymer), releasing isocyanates and hence PAA.[17] For such applications, the actual conditions of use should form the basis of the testing conditions.

[15] BMBF Project FKZ 0330347, Fraunhofer/FABES, 2002 – 2005

[16] Measurement of PAA does not ensure that limits for residual isocyanates are met. However, it may be possible to demonstrate a correlation between PAA and isocyanate monomer content. In such cases, PAA testing may be used as a practical method of demonstrating an adequate cure of the adhesive. It should be noted that aliphatic diisocyanates do not generate PAA; hence only a direct content determination is possible.

[17] This case of formation of PAA is not due to an insufficient cure of the adhesive and residual isocyanate monomer. Isocyanates and PAA are thermally released from a fully cured adhesive which, prior to the thermal impact, did not contain any detectable residual isocyanate monomer.

Hotmelt adhesives

Liquid simulants containing ethanol may re-dissolve large parts of hotmelt applications (e.g., tackifiers). In addition, elevated temperatures above 40 °C are likely to cause a softening of a hotmelt due to the low molecular weight fraction (e.g., waxes). In general, food simulants and conditions in Regulation (EU) No 10/2011 are not appropriate for this category of adhesive. Testing conditions should be defined on a case-by-case basis, depending on the adhesive and application type.

Hotmelt adhesives may in certain cases contribute to migration results of mineral oil hydrocarbons (MOH). Such migration stems from low molecular weight fractions in their constituents, such as resins, waxes or oils. The most frequently used analytical method was developed and optimised for analysis of mineral oil hydrocarbons in foodstuff as well as in recycled paper and cardboard. There is currently no method available that is specifically adapted to adhesives. FEICA provides a separate guidance on the topic of MOH migration from adhesives.[18]

Heat seal applications for acidic foodstuffs

Heat seals may be applied on aluminium foil, metallised plastic films or metallised paper, and such food contact materials may be used in the packaging of acidic foods, such as dairy products. It must be considered that food simulants defined for plastics and paper which are substantially more acidic than actual food (such as 3% acetic acid) may not be suitable in these cases as they will attack the metallisation or aluminium foil, releasing metal salts, and possibly detaching the heat seal from the surface. Both effects may lead to an exceeding of overall migration limits and/or specific migration limits. In such cases, it is advised to apply, for migration testing purposes only, the heat seal on alternative, that is metal-free, substrates.

It should be noted that besides the possibility of the substrate being attacked, the heat seal itself may also be degraded or dissolved if the simulant is substantially more acidic (or otherwise aggressive) than the actual food. In case of doubt, the actual foodstuff should be used for migration testing.

Pressure-sensitive adhesive applications for direct food contact

Pressure-sensitive adhesives (PSA) maintain surface stickiness after application. Migration testing in direct contact with the dry food simulant Tenax® is not possible as Tenax® will stick to the adhesive and will not be able to be removed after migration.

When direct food contact of PSA needs to be simulated, e.g., in the case of direct labelling of fruits and vegetables, applying the PSA on a substrate with low or no barrier properties, e.g., a thin cellulose paper, can be considered. Migration can then be performed from the paper side, preventing Tenax® from directly contacting the adhesive. In this type of migration setup, the substrate should only help to separate the food simulant from the PSA; it should not influence migration. In case of doubt, the actual application needs to be tested.

[18] FEICA Guidance paper 'Guidance for evaluating the food contact status of adhesives containing mineral oil hydrocarbons'

6 Contact

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