The European voice of the adhesive and sealant industry

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FEICA guidance on evaluating the food contact status for adhesives containing mineral oil hydrocarbons

Content

Executive Summary

In 2011, health concerns were raised after studies were published indicating that consumers could be exposed to mineral oil hydrocarbons (MOH) in foodstuffs, with the likely major sources being food packaging and additives, processing aids, and lubricants. The EFSA opinion[1] put the main focus on mineral oil migration from recycled paper, where the mineral oil from the printing inks was a major contributor to the contamination of the packed food. The supporting studies were published by the Official Food Control Authority of the Canton of Zurich, Switzerland.

Despite the fact that several new studies have been published since the EFSA opinion, the situation remains complex and challenging for the whole supply chain. For adhesives, the lack of official analytical methods especially for simulating migration means that the test results may not reflect reality. In addition, adhesives' raw materials multi-constituent substances such as mineral oils, waxes, resins and oligomers are difficult to analyse, especially in complex matrices.

In this guidance document, FEICA aims to provide some clarification on how to risk assess mineral oil hydrocarbons in adhesives and, in case testing is needed, how to perform the test and evaluate the results. A decision tree is also included to allow customers to evaluate an adhesive for its intended application. This guidance aims to support adhesives producers and their downstream users in making sure that the adhesive in its intended application complies with article 3 of the Framework Regulation 1935/2004.

Types of mineral oil hydrocarbons

The term mineral oil is imprecise and encompasses a wide range of mixtures of hydrocarbons.

The EFSA opinion defines mineral oil hydrocarbons (MOH) or mineral oil products as: "*hydrocarbons containing 10 to about 50 carbon atoms, where the crude mineral oils remain by far the predominant source of the MOH considered, but equivalent products can be synthesised from coal, natural gas or biomass.*" This definition includes all the various refinery grades and also substances which are evaluated as non-hazardous or not harmful to humans or the environment. Highly purified mineral oils and paraffins have been used in cosmetic or medical applications for decades and for food and food contact applications as well. They are approved and supported by adequate toxicological data. Thus, mineral oils are part of our daily life and an accurate differentiation in their toxicological evaluation should be the key in every discussion.

The EFSA opinion divides MOH into two main types:

- Mineral oil saturated hydrocarbons (MOSH), which comprise linear and branched alkanes, and alkyl-substituted cyclo-alkanes.
- Mineral oil aromatic hydrocarbons (MOAH), which comprise mainly alkyl-substituted polyaromatic hydrocarbons.

However, even this categorisation is imprecise due to a lack of a precise definition of MOSH and MOAH. The MOSH and MOAH fractions are mainly determined by the results derived through the commonly used analytical method.[2] Because of their complexity, it is not possible to resolve MOH mixtures into individual components for quantification.[3]

[1] http://www.efsa.europa.eu/en/efsajournal/doc/2704.pdf

[2] The most frequently used testing method is based on on-line coupled HPLC-GC-FID (high performance liquid chromatography – gas chromatography – flame ionisation detector), published by the Zurich Cantonal Laboratory (KLZH) and Germany's Federal Institute for Risk Assessment (BfR).

[3] http://www.efsa.europa.eu/en/efsajournal/doc/2704.pdf

Sources of mineral oil hydrocarbons in food

The European Food Safety Authority Panel on Contaminants in the Food Chain (CONTAM Panel) identified the following potential sources of mineral oil hydrocarbons in food (EFSA 2012 "Scientific Opinion on Mineral Hydrocarbons in Food", The EFSA Journal 10(6):2704, pp 140 - 141).:

Food contact materials

- Food packaging materials made from recycled paper and board.
- Off-set printing inks applied to paper and board for food packaging.
- Mineral oils used as additives in the manufacture of plastics for food contact (e.g. internal lubricants in polystyrene, polyolefins).
- Wax paper and board.
- Jute or sisal bags with mineral batching oil.
- Lubricants for can manufacture.
- Wax coating directly applied to food.

In addition, some types of adhesives may contain mineral oil hydrocarbon components.

Contaminants

- Environmental contaminants: lubricating oil from engines without catalyst (mainly diesel), unburned fuel oil, debris from tyres and road bitumen.
- Harvesting machinery: diesel oil, lubricating oil.
- Lubricating oils in pumps, syringe type dosing machinery and other industrial installations used in food processing.
- Cleaning agents, solvents consisting of pure MOH or C10-C14 mixtures.

Food additives, processing aids and other uses

- Release agents for bakery ware and sugar products.
- Oils for surface treatment of foods, such as rice, confectionery.
- Mineral oils in feeds, e.g. binders for minor additives added as powder.
- Defoamers.
- Authorised paraffinic waxes (e.g. for chewing gum or coating of certain fruits).
- Pesticide formulations.
- Anti-dusting agents for cereals.

Potential health issues

The MOSH and MOAH paradigm is a generic terminology that is used to describe an analytical fraction and is an overall descriptor of hydrocarbons from variable petroleum, synthetic and in some cases even natural sources.

Because of the diverse nature of MOH, there is a lack of reference standards for human exposure and information relating to actual health effects. Although the European Food Safety Authority identified potential concerns about MOH in food, it acknowledged considerable uncertainties in

assessing any potential risks and concluded that further studies were needed (see Technical Appendix).

According to EFSA 2012 (last update)[4], MOSH and MOAH exhibit the following properties:

- MOSH, mainly the fraction comprising carbon chains of 16 - 35 atoms (C16 - C35), may accumulate in the human body, especially in lymph nodes, spleen and liver. However, EFSA has also stated that this has not been associated with adverse health consequences. (Note that this has been explained further by pathologist K. Fleming at MOCRINIS 2013[5].)
- MOAH with three or more, non- or simple-alkylated, aromatic rings may be mutagenic and carcinogenic, and is therefore considered by the European Food Safety Authority Panel of higher concern than the MOSH fraction.

In general, toxicologists focus on polycyclic aromatic hydrocarbons (PAH) and especially 3-7 ring PAH. Unlike these 3-7 ring polycyclic aromatic compounds, of which some are known to be carcinogenic, highly alkylated 1-2 ring systems are not genotoxic and many are not considered to be a concern for carcinogenicity[6],[7],[8], [9]. Hence, although these species are likely to contribute to the "MOAH" identified in MOH products, they, in and of themselves, do not pose a carcinogenic risk in the MOH products. To conclude, the presence of MOAH in itself is not indicative of a carcinogenic potential. This must be taken into consideration when performing a risk assessment on MOH in adhesives intended for food packaging.[10]

EFSA have assessed a few MOH that are listed and specified on the positive list of the Plastics Regulation EU/10/2011. As they are included among chemicals that could be safely used in plastics production intended for food contact, these MOH are also assumed to be safe in use for the production of adhesives intended for food packaging. See Technical Appendix for the full list of EFSA assessed MOH including their descriptions and specifications.

[4] http://onlinelibrary.wiley.com/doi/10.2903/j.efsa.2012.2704/epdf
[5] https://www.concawe.eu/uploads/Modules/Publications/rpt_14-2-2014-00300-01-e.pdf
[6] Florin I, Rutberg L, Curvall M, Enzell CR (1980) Screening of tobacco smoke constituents for mutagenicity using the Ames' test. *Toxicology* **15:** 219-232
[7] Höke H, Zellerhoff R (1998) Metabolism and toxicity of diisopropylnaphthalene as compared to naphthalene and monoalkyl naphthalenes: a minireview. *Toxicology* **126:** 1-7
[8] Kulka U, Schmid E, Huber R, Bauchinger M (1988) Analysis of the cytogenetic effect in human lymphocytes induced by metabolically activated 1- and 2-methylnaphthalene. *Mutation Research Letters* **208:** 155-158
[9] USEPA (2003) BIOPESTICIDES REGISTRATION ACTION DOCUMENT: 2,6-Diisopropylnaphthalene https://www3.epa.gov/pesticides/chem_search/reg_actions/registration/decision_PC-055803_1-Oct-03.pdf.
[10] Concawe and EWF position on the 4th version of the EU Commission Recommendation on the monitoring of mineral oil hydrocarbons in food and materials and articles intended to come into contact with food: https://www.concawe.eu//uploads/Modules/Publications/concawe-response-eu-com-moh-monitoring-final-draft-(003).pdf

Testing for mineral oil hydrocarbons

Generally, food contact materials such as food packaging have to be evaluated with regard to substance transfer (so-called “migration”) from the packaging material into the packed foodstuff. Adhesives, as a part of the food contact material, may in some cases contribute to migration with their low molecular weight fractions such as resins, waxes or oils.

Because it is often not possible or desirable to test migration on real food, these migration properties usually have to be simulated. This can be done either via migration modelling based on residual concentration of migrant, or via migration testing. For migration testing, food simulants such as "MPPO" (poly(2,6-diphenyl-p-phenylene oxide), particle size 60-80 mesh, pore size 200 nm) can be an appropriate option. After migration, the food simulant has to be analysed for all migrated compounds. In contrast to paper and cardboard investigations, direct extraction of the adhesive is not an option, as the low molecular weight fractions would be dissolved to a much greater extent than the migration which can be expected in the real food contact scenario. In addition, the adhesive never surrounds the food in its entirety and is used only in dots and stripes and in general is not in contact with the food at all (migration via the gaseous phase, predominantly into dry foodstuff needs to be considered). Only migration tests are capable of simulating the real substance transfer, and all further analytical determinations should be based on the food simulant from the migration test.

With regard to the analytical determination of MOH, the most frequently used testing method is based on on-line coupled HPLC-GC-FID (high performance liquid chromatography – gas chromatography – flame ionisation detector), published by the Zurich Cantonal Laboratory (KLZH) and Germany’s Federal Institute for Risk Assessment (BfR). The procedure was developed and optimised for analysis of mineral oil hydrocarbons in foodstuff as well as in recycled paper and cardboard.

In this test setup, the liquid chromatographic separation via HPLC defines two fractions of substances, based on a difference in polarity. Substances in the non-polar fraction will be assigned as MOSH, whereas the substances in the polar fraction will be assigned as MOAH. However, it has to be noted that the subsequent GC-FID determination of these pre-separated fractions is neither capable of resolving the complex substance mixtures into individual compounds, nor is the flame ionisation detector capable of assigning chemical structures unequivocally.[11]

When evaluating the MOH transfer from an adhesive into foodstuff, this HPLC-GC-FID procedure should be applied on the poly(2,6-diphenyl-p-phenylene oxide), particle size 60-80 mesh, pore size 200 nm food simulant obtained from a suitable migration test setup. It should not be applied on the extraction solution of the pure adhesive. Applying the HPLC-GC-FID procedure on adhesive extraction solutions may result in extremely high and unrealistic MOSH and MOAH values, because a simple HPLC-GC-FID analysis does not differentiate between substances from mineral oils and substances from non-mineral oil sources, such as tackifier resins and oligomers from polyolefins (POSH). When analysing for the presence of mineral oils, the low molecular weight fraction of tackifier resins in adhesives could produce ‘false positives’ (Lommatzsch, Biedermann, Grob, & Simat, 2016). [12]

[11] (EFSA Journal 2012;10(6):2704, chapter 5.2)

[12] HARRPA statement about MOSH & MOAH in Food Contact Materials – December 2016

Risk assessment of non-listed raw materials, including mineral oils

As mentioned above, some MOH are assessed by EFSA and adhesives manufacturers can refer to the Plastics Regulation (EU) No. 10/2011 for guidance on mineral oil hydrocarbons, as explained in the Technical Appendix.

In the absence of EU harmonised specific measures for non-plastics, the adhesive manufacturer may also use non-listed substances. In this case it needs to perform an in house risk assessment.

A useful tool to assist adhesives manufacturers in their risk assessment is the FCA guideline on non-listed substances:[13]

It should always be acknowledged that, depending on the nature of the packaging material and its production process, adhesives will not be the main source of mineral oil hydrocarbons in the packaging. As a result, full compliance of (EC) No. 1935/2004 can only be addressed by the manufacturer of the final packaging material because only it has an oversight of all the components of the packaging. To assist in this process, adhesive companies are obliged to provide packaging manufacturers with information about the adhesives they supply.

Conclusions from the EFSA Opinion

In June 2012 the European Food Safety Authority (EFSA) published its "Scientific Opinion on Mineral Hydrocarbons in Food", which was updated in August 2013 (The EFSA Journal 2012; 10(6):2704). This concluded the following:

- There are various sources of mineral oil contamination in food.
- With analytical methods it is not possible to separate MOH into individual components.

In the study, EFSA recommended the following approaches:

- Reference standards and materials should be established to be able to develop validated analytical methods.
- The analytical methods and monitoring systems should be improved to enable better assessment of the risks posed by mineral oil hydrocarbons and to distinguish between MOAH and MOSH and their subclasses.
- Sources of mineral oil contamination at various stages of the production should be identified.
- Further toxicological studies on the possible hazards posed by the various mineral oil fractions are needed.
- An investigation into how to transfer the findings of animal studies regarding MOH to humans should be made.

FEICA's recommendations for the adhesive industry

FEICA represents Europe's adhesive manufacturers. These manufacturers are committed to continuous improvement in the health and safety aspects of their products. To this end, FEICA's technical experts and specialists from the adhesive manufacturers have developed a set of recommendations to help the industry to evaluate and where necessary reduce or eliminate mineral oil hydrocarbon contact with foodstuffs.

[13] FCA guideline on "Risk Assessment of non-listed substances (NLS) and non-intentionally added substances (NIAS) under the requirements of Article 3 of the Framework Regulation (EC) 1935/2004", http://fca.cefic.org/images/Documents/FCA.pdf

As a first step, adhesive manufacturers have to make a risk assessment of the intended use of their adhesives. They should review their adhesive application and follow the decision tree as recommended in the FEICA guidance for a food contact status declaration for adhesives.

They then have to answer the following questions:

- What is the intended application?
- Is there a sufficient barrier between the adhesive and the foodstuff?
- Is there a risk of migration in the specific application?

The adhesive manufacturer needs to check whether the mineral oil hydrocarbons used in the formulations and which pose a risk for migration are listed in Regulation (EU) No. 10/2011 (FCM Nos 93, 94, 95). If this is the case, the restrictions given by the Plastics Regulation shall be applied. If this is not the case, the adhesive manufacturer should follow the steps below to ensure that their adhesives are fit for the intended use.

Water-based adhesives with expected food contact

Water-based adhesives for some specific applications may contain MOH originating from the defoamer (typically maximum concentrations are not higher than 0.5%).

Typical applications: Construction and closing of paper and paper board packaging, labelling, paper lamination.

Recommendation:

- For the very unlikely case that the intended application of the water-based adhesive would pose a risk for migration into the food, check whether the mineral oil based defoamer is compliant with the relevant food contact legislation or request compositional information and/or toxicological data from the supplier of the defoamer used in the adhesive (e.g. carbon number distribution, content of PAHs , result of Ames test, etc.) and perform a risk assessment).
  If the mineral oil hydrocarbon components cannot be sufficiently evaluated or/and a risk of migration into the food cannot be minimised, the adhesive manufacturer has to reduce the content of the mineral oil defoamer as much as possible. Food contact status: The adhesives manufacturer has to list mineral oil defoamer as a substance with restriction (10ppb), in the specific migration limit (SML) table with a maximum expected concentration and / or provides an own evaluation in order to enable risk assessment by the downstream user.

Hotmelts with expected food contact

Hotmelts such as some ethylene vinyl acetate and polyolefin-based hotmelts for case and carton sealing contain mineral oil hydrocarbons. Sometimes, paraffinic waxes or hydrocarbon resins are used in the formulation, which can be found in the MOH fractions, leading sometimes to a misinterpretation of the results (see chapter: Testing for mineral oil hydrocarbons).

Typical applications: Case and carton sealing, lamination.

Recommendation:

- If hydrocarbon resins are used, please make sure that they are evaluated, either by FCM 97 or by other supporting documents from the supplier (tox data, compliance information).
- If the mineral oil hydrocarbon components are not in compliance with Plastics Regulation (EU) No. 10/2011
    - Either consider replacing them by components complying with FCM Nos 93, 94.
    - Or request compositional information and / or toxicological data from the supplier of the mineral oil hydrocarbon component (e.g. carbon number distribution, content of PAHs, result of Ames test, etc.) and perform a risk assessment for the adhesive in the intended application. If necessary, reduce the content of the mineral oil hydrocarbon components.
      Food contact status: If the risk of migration of mineral oil hydrocarbons into the food cannot be excluded, a functional barrier has to be recommended.

Pressure-sensitive adhesives (hotmelt) with expected food contact

Most hotmelt pressure-sensitive adhesives contain mineral oils. Normally, block polymers are used, which may be accompanied by mineral oil (10% - 30% in the formulation). In PSA hotmelts, the replacement of oil is difficult, but it is possible to use more refined oil with less naphthenic / aromatic components.

Typical applications: Labelling, tapes, packaging tapes, resealable packs.

Recommendation:

- If hydrocarbon resins are used, please make sure that they are evaluated, either by FCM 97 or by other supporting documents from the supplier (tox data, compliance information).
- If the mineral oil hydrocarbon components are not in compliance with Plastics Regulation (EU) No. 10/2011
    - Either consider replacing them by components complying with FCM Nos 93, 94, 95.
    - Or request compositional information and / or toxicological data from the supplier of the mineral oil hydrocarbon component (e.g. carbon number distribution, content of PAHs, result of Ames test, etc.) and perform a risk assessment for the adhesive in the intended application.

      Food contact status: If the risk of migration of mineral oil hydrocarbons into the food cannot be excluded, a functional barrier has to be recommended.

Pressure-sensitive adhesives (water-based) with expected food contact

Water-based pressure-sensitive adhesives may contain MOH from the defoamer (see water-based adhesives above)

Typical applications: Coldseals, self-adhesive labels

Recommendation:

- For the very unlikely case that the intended application of the water-based adhesive would pose a risk for migration into the food, the mineral oil based defoamer should be replaced by a defaomer which is compliant with the relevant food contact legislation.
- If this is not possible, request compositional information and / or toxicological data from the supplier of the defoamer used in the adhesive (e.g. carbon number distribution, content of PAHs, result of Ames test, etc.) and perform a risk assessment for the adhesives in the final application).
- If the mineral oil hydrocarbon components cannot be sufficiently evaluated or/and a risk of migration into the food cannot be minimised, the adhesive manufacturer has to reduce the content of the mineral oil defoamer as much as possible.
- Food contact status: The adhesives manufacturer has to list mineral oil defoamer as a substance with restriction (10ppb) in the specific migration limit (SML) table with a maximum expected concentration and / or provides an own evaluation in order to enable risk assessment by the downstream user.

Conclusion

Some MOAHs, especially the three or more, non- or single-alkylated, aromatic rings may be mutagenic and carcinogenic, and are therefore considered by the European Food Safety Authority Panel of higher concern than the MOSH fraction. Some MOH on the other hand have been assessed by EFSA and are listed and specified on the positive list of the Plastics Regulation EU/10/2011. Unfortunately, this differentiation is often omitted in the public discussion.

Mineral oil hydrocarbons are composed of thousands of different constituents, with variations in toxicological characteristics. The analytical test methods currently available for MOSH and MOAH fraction determination are not able to separate the fractions into individual constituents. These methods were developed for the analysis of paper and board or for the extraction of foodstuff, but there is no method specifically adapted for adhesives.

Applying the current analytical methods for MOSH / MOAH separation will always result in misleading values for adhesives. Only migration tests with poly (2,6-diphenyl-p-phenylene oxide), particle size 60-80 mesh, pore size 200 nm can give a realistic picture of substance transfer into the food. The following MOSH/MOAH analyses should be based on the food simulant rather than on the extraction of the adhesive. Sample preparation and interpretation of the test results for adhesives can be difficult. Cooperation of the adhesives supplier, the customer and test laboratory is important.

FEICA is working in collaboration with specialists from the adhesive manufacturers and the various players in the packaging supply chain. FEICA encourages all adhesive manufacturers to carry out a proper risk assessment of all ingredients of an adhesive formulation. Besides the listed mineral oil hydrocarbons (FCM 93, 94, 95,), unlisted compounds can also be used in the adhesive formulations if the risk assessment doesn't show any reasons for concern. Effective communication in the supply chain is essential to ensure that the adhesive manufacturers provide packaging manufacturers with sufficient information about the adhesives to allow them to carry out their own risk assessments, so that the packaging producer will be able to demonstrate compliance with article 3 of the Framework Regulation for the final packaging. A decision tree in the annex of this guidance document will assist downstream users in their evaluation of the adhesive for the intended application.

Technical Appendix

Mineral oil hydrocarbon compounds evaluated by EFSA

The European Food Safety Authority opinion, published on 6 June 2012, specifies the types of mineral oil hydrocarbons approved for food contact, as detailed in this excerpt:

"Regulation (EC) No. 1935/2004 lays down the general provisions and principles for food contact materials and articles. There are no specific measures regarding mineral oil hydrocarbons, except for the provisions on their use as additives in plastic materials and articles intended to come into food contact laid down by Regulation (EU) No. 10/2011. The following mineral oil hydrocarbons are covered by the positive list of additives:

a. FCM substance No. 95: White mineral oils, paraffinic, derived from petroleum-based hydrocarbon feedstock. No specific migration limit (SML) is defined (i.e. its use is restricted only by the overall migration limit of 60 mg/kg food or 10 mg/dm^2 food contact surface). The product must comply with the following specifications:
   - hydrocarbons with carbon number less than 25, not more than 5% (w/w);
   - viscosity not less than 8.5 mm^2/s at 100°C;
   - average molecular weight not less than 480 Da.

b. FCM substance No. 94: Waxes, refined, derived from petroleum-based or synthetic hydrocarbon feedstock. No SML is specified (i.e. its use is restricted only by the overall migration limit). The product must comply with the following specifications:
   - hydrocarbons with carbon number less than 25, not more than 5% (w/w);
   - viscosity not less than 11 mm^2/s at 100°C;
   - average molecular weight not less than 500 Da.

c. FCM substance No. 93: Waxes, paraffinic, refined, derived from petroleum-based or synthetic hydrocarbon feedstock. An SML of 0.05 mg/kg food is specified. In addition, these oils are not to be used for articles in contact with fatty foods. The product must comply with the following specifications:
   - hydrocarbons with carbon number less than 25, not more than 40% w/w;
   - viscosity at 100°C min 2.5 mm^2/s;
   - average molecular weight not less than 350 Da."

In addition, one mineral oil hydrocarbon component is approved as a food additive and has an "E" number: Microcrystalline wax (E 905) is approved for use in the surface treatment of confectionery (excluding chocolate), chewing gum, melons, papaya, mango and avocado.

Hydrocarbon Resins

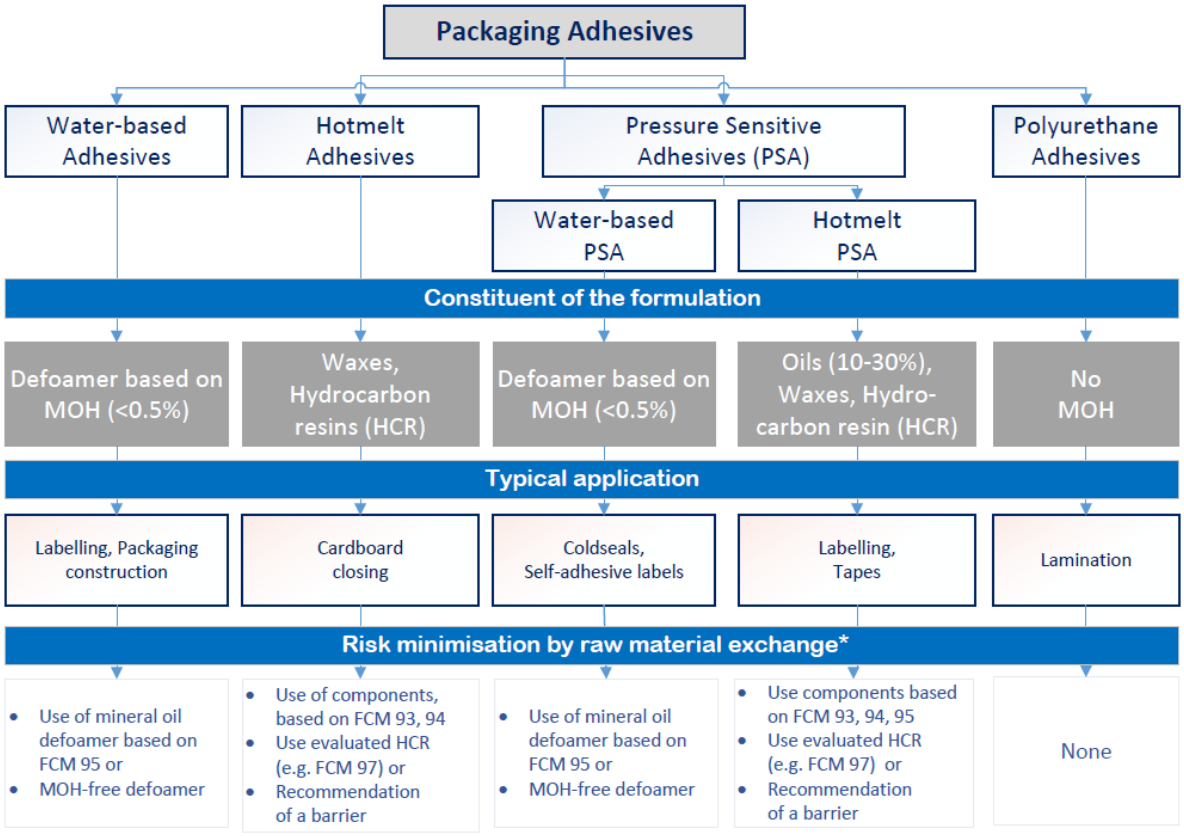
The influence of the hydrocarbon resins in the MOSH and MOAH analytic was described in this Guidance paper. Therefore, we would like to refer also to the group of listed and toxicologically evaluated resins (FCM 97). Further analytical progress and expert knowledge is needed to avoid misinterpretation of the results.

In addition, Regulation (EU) No. 10/2011 lists FCM substance No. 97 as follows:

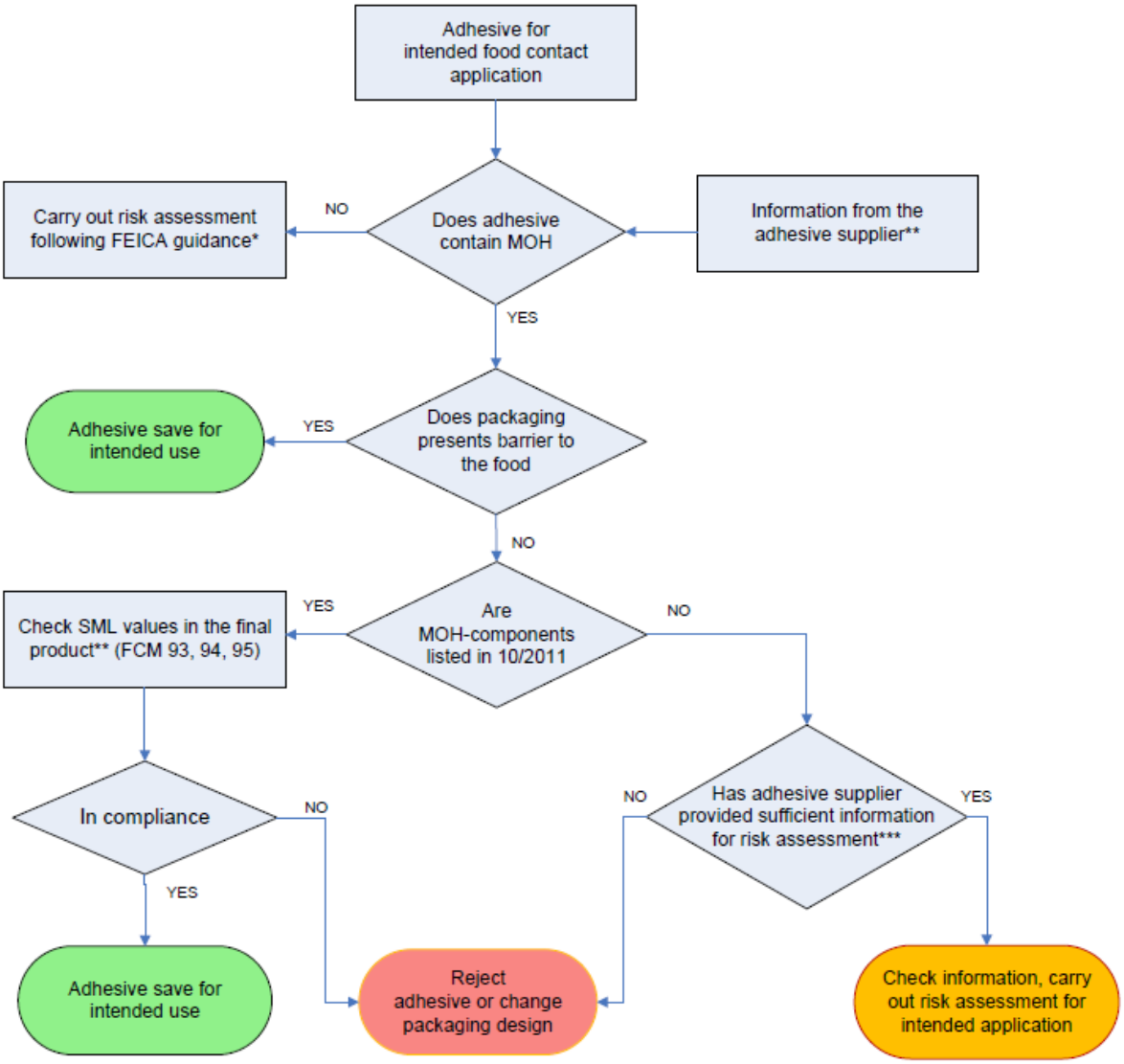
FCM substance No. 97: Petroleum hydrocarbon resins, hydrogenated

- Viscosity at 120 °C: > 3 Pa.s
- Softening point: > 95 °C as determined by ASTM Method E 28-67
- Bromine number: < 40 (ASTM D1159)
- The colour of a 50 % solution in toluene < 11 on the Gardner scale
- Residual aromatic monomer ≤ 50 ppm

Mineral Oil in Adhesives



Decision Tree for Downstream User Evaluation of Adhesives



** see FEICA decision tree, chapter 3.1, Guidance for a food contact status declaration for adhesives
** From food contact status declaration or other source
*** Tox data, e.g. ADI(acceptable daily intake) , LD (lethal doses) values, etc. / result of risk assessment

Contact for more information

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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.

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