European Regulations For Multilayer Food Contact Materials

Dr. Hermann Onusseit
Henkel KGaA
Düsseldorf, Germany

ABSTRACT

Articles and facilities which come into contact with food are subject to special demands. This applies to processing and also to transport and warehousing of food. These articles are requested to not influence the food they contact, neither toxicologically i. e. no health threat for the end-user, nor may they change the composition of the food and the properties like smell, taste and look. Most materials that come into contact with foodstuffs consist of different parts or components, which together form an article, eg, a package. Often adhesive is used for holding together the parts. In a multi-layer package, those layers not in direct contact with foodstuff may not be neglected, because their constituents may migrate through the food-contact layer. While there are already European regulations for plastics, for other components like printing inks or adhesives a preparation is intensively discussed. The establishment of a positive-list system for those components will require very much time and money and this surely cannot be accomplished for all formulation components used today. Thus in the future a number of formulations may no longer be legal for food contact materials. A positive-list system absolutely requires an individual compliance check with specific migration limits. It has to be considered that conventional migration test processes are time-consuming and costly and that there are no analytical methods for many components yet. As a consequence the only possible and useful way is a specific testing concept by a combination of extraction and migration tests and mathematical modelling, that guarantees the safe use of these components in multilayer food contact materials, and which can be utilized cost-efficiently by the industry, especially by small and medium-sized enterprises.

INTRODUCTION

Food contact materials are used in the production of articles intended to come into contact with foodstuffs, like cutlery, dishes, processing machines, containers and above all packaging [Fig. 1]. This applies to both the processing of food and to its transport and warehousing. The term also includes materials and articles that are in contact with water intended for human consumption, but it does not cover fixed public or private water supply equipment. Food contact articles shall be safe and shall not transfer their components into the foodstuff in unacceptable quantities. These articles are requested to not influence the food they contact, neither toxicologically ie, they should not cause a health threat for the end user, nor may they change the composition of the food or its properties like smell, taste and appearance.

THE EUROPEAN APPROACH

On the European level two authorities deal with questions of food safety. One is the European Union and the other is the Council of Europe. While the decisions of the European Union are obligatory for national legislations, those of the Council of Europe are only a recommendation.

THE EUROPEAN UNION

A first framework directive on materials and articles intended to come into contact with foodstuffs was adopted in 1976. It was replaced in 1989 by the Framework Directive 89/109/EEC. This basic law for food contact articles was laid down in the Council Directive of 21 December 1988, consolidating the laws of the member states relating to materials and articles intended to come into contact with foodstuffs. The framework directive applies to all materials and articles intended to come into contact with foodstuff. Detailed explanations about how these principal demands should be transformed are described in specific regulations within the framework directive. Hence in the past 15 years some specific directives have been prepared, eg, for plastic materials, the so-called Plastic Directive, first published in 1990 (90/129/EEC). The current version of the Plastic Directive is from 2002

(2002/72/EC). Others exist for regenerated cellulose films, ceramics, and vinyl chloride monomer. Due to technological development it was necessary to adjust this framework directive in 2004. Hence on 27 October 2004 the new Framework Regulation (EC) 1935/2004 was enacted and published in the European Official Journal on 13 November 2005. In contrast to a directive, which has to be converted after publication by the European Union within a certain period by the individual member states into national law, a regulation is automatically converted into the national law of all EU countries twenty days after publication. In this new framework regulation also some new articles and groups of substances were included for which specific regulations should be prepared. Some materials and articles to which future special directives or regulations will apply are active and intelligent materials and articles, adhesives, paper and board, printing inks, rubber, glass, metal and alloys, wood, cork, textiles, waxes, varnishes and coatings and ion-exchange resin [Fig. 2].

The regulation set some general requirements that must be met by all food contact materials: All materials and articles must be manufactured according to Good Manufacturing Practice (GMP) so that they do not transfer their constituents to foodstuffs in quantities which could endanger human health or "bring an unacceptable change in the composition of the foodstuff or a deterioration in the organoleptic properties". In article 3 the following general requirements are mentioned.

“Materials and articles, including active and intelligent materials and articles, shall be manufactured in compliance with good manufacturing practice so that, under normal or foreseeable conditions of use, 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.”

The transfer of components from the food contact materials into food is called migration. To ensure the protection of the health of the consumer and to avoid alteration of the foodstuff, two types of migration limits have been established in European law. For the first time these numerical values appeared in the Commission Directive of 23 February 1990 relating to plastic materials and articles intended to come into contact with foodstuffs:

- an overall migration limit (OML) of 60mg (of substances)/kg (of foodstuff or food simulants) that applies to all substances that can migrate from the food contact material into the foodstuff and

- a specific migration limit (SML) which applies to individual authorized substances and is fixed on the basis of the toxicological evaluation of the substance. The SML is generally established according to acceptable daily intake (ADI) or tolerable daily intake (TDI). These values formerly were set by the Scientific Committee on Food (SCF) and today are set by the European Food Safety Agency (EFSA). To set the limit, it is assumed that every day throughout his/her lifetime, a person of 60kg eats 1kg of food packed in plastics containing the relevant substance at the maximum permitted quantity.

The current approach for the authorization and control of substances used in food contact materials is considered to be cautious in relation to the estimation of potential exposure of the consumer to these substances. Approaches are being discussed which take into better consideration the actual exposure of the consumer to food contact materials in risk assessment.

The use of mathematical modelling for prediction of migration, which can reduce the amount of tests to be undertaken, has been recently introduced into the legislation. Practical examples for the application of this new concept are described in the Practical Guide.

THE COUNCIL OF EUROPE

Even though there is no formal agreement between the European Union and the Council of Europe concerning food contact materials, there are important and numerous contacts between the services of the Health & Consumer Protection Directorate-General and the Council of Europe in this field. The Council of Europe has prepared a number of documents, which are used as basics for many national regulations in Europe.

The Committee of Ministers has adopted the following resolutions:

- Resolution AP (89) 1 on the use of colourants in plastic materials coming into contact with food
- Resolution AP (92) 2 on control of aids to polymerisation for plastic materials and articles
- Resolution AP (96) 5 on surface coatings intended to come into contact with foodstuffs
- Resolution AP (97) 1 on ion exchange and adsorbent resins used in the processing of foodstuffs (superseding Resolution AP (89) 2)
- Resolution AP (99) 3 on silicones used for food contact applications
- Resolution AP (2002) 1 on paper and board materials and articles intended to come into contact with foodstuffs
- Framework Resolution AP (2004) 1 on coatings intended to come into contact with foodstuffs
- Resolution AP (2004) 2 on cork stoppers and other cork materials and articles intended to come into contact with foodstuffs
- Resolution AP (2004) 3 on ion exchange and adsorbant resins used in the processing of foodstuffs
- Resolution AP (2004) 4 on rubber products intended to come into contact with foodstuffs
- Resolution AP (2004) 5 on silicones used for food contact applications

Present activity programme of the Committee of experts:

- List of substances on paper and board products intended to come into contact with foodstuffs (concerning Resolution AP (2002) 1);
- Draft guidelines on the safety evaluation of food contact paper and board substances;
- Draft resolution on printing inks, primers, coloured lacquers and overprint varnishes applied to the non-food contact surface of food packaging and articles intended to come into contact with foodstuffs;
- Guidelines on lead leaching from glass tableware into foodstuffs;
- Guidelines on tissue paper kitchen towels;

All the above-mentioned topics will include toxicological and technological specifications and limits as well as inventory lists of chemical substances used for the manufacture of these substance groups.

MULTILAYER FOOD CONTACT MATERIALS

Most materials that come into contact with foodstuffs consist of different parts or components, which together form an article, eg, a package. Often adhesive is used for holding together the parts. In a multi-layer package, those layers not in direct contact with foodstuff may not be neglected, because their constituents may migrate through the food-contact layer. This thought was described for the first time in the so-called "Super Regulation".

The driving idea in writing the "Super Regulation" (former "Super Directive") was to collect in one text the rules set out in all the directives related only to plastic materials and articles ie, Commission Directive 2002/72/EC and its amendments, consolidation of 82/711/EEC and its two amendments, 93/8/EEC and 97/48/EC; 85/572/EEC; and the three vinyl chloride monomer directives, 78/142/EEC, 80/766/EEC and 81/432/EEC. This should be considered as an attempt to prepare a future commission directive on all types of plastics. As usual, the regulation or directive contains only general principles of the technical rules such as modelling, functional barrier etc. Details and guidance will be introduced at CEN level or in the Practical Guide.

To give an idea of the repartition of the rules, some examples or references to existing documents are given in the draft of the "Super Regulation", such as the extension of the rules to multilayers composed of different materials and the introduction of the new concept of "functional barrier". It has to be noted that the layer acting as "functional barrier" may be either authorized by the directive and listed together with the conditions of its validity, or used without any authorization by the manufacturer provided that the written declaration of compliance mentions the substances not subject to an authorization procedure.

A multilayer plastic food contact material shall comply with the following rules: Any layer shall be manufactured

from substances mentioned in a Community list subject to their restrictions. In absence of a Community list, substances which appear in a national list from a European country may be used provided they comply with article 3 of Resolution (EC) 1935/2004. In the absence of these lists, only substances in amounts which, in the practical use of the final materials or articles, can be proved to comply with article 3 of Resolution (EC) 1935/2004 may be used. These substances are hereinafter referred to as "Listed food contact substances". The finished articles shall comply with the OML, the SML and the other specific restrictions of the substances contained in the different layers; each layer maintains its own specifications.

A "Functional Barrier" is a barrier consisting of one or more layers that ensures that migration of adhesive components does not exceed their SML, and which reduces the migration of these components, their reaction products, and impurities into food to a "non-detectable" level when subjected to a validated test method. For the purpose of control, "non-detectable" means that a substance cannot be detected at a concentration appropriate for the nature of the substance, but in no case exceeding 0.01 mg/kg. This detection limit, which is currently discussed, applies to groups of structurally related compounds, eg, isomers.

DECLARATION OF COMPLIANCE

To guarantee the conformity of the used materials the Framework Regulation (EC) 1935/2004 demands in article 16 the preparation of a declaration of compliance. This thought is described in the draft of the ‘Super Regulation’ in detail.

The written declaration of compliance shall permit an easy identification of the materials or articles for which it is issued and shall be reviewed periodically. It shall contain the following information: Identity of material or article; its range of application; and the confirmation that the material or article complies with the requirements of the European directives and, when appropriate, with national law. When a functional barrier is used in a multilayer material, the following additional information shall be provided: The identity of the substances of the functional barrier, the date of latest use of the material or article; and the maximum heat treatment (temperature and time) for the article. At each stage of manufacture, processing, and distribution an appropriate technical documentation able to demonstrate the compliance of the material, article or substances with the relevant provisions shall be available. This documentation, hereinafter called "Supporting Documents", shall contain the description and the results of the analysis carried out to demonstrate the compliance of the material and article, and in particular the compliance with quantitative restrictions in the use of the substances such as OML, SML etc, plus the requirements of the layer(s) constituting a functional barrier, and the requirements set out in Article 3 of Framework Regulation (EC) 1935/2004 related to the substances migrating in detectable amounts and which are not listed in positive lists.

ADHESIVES IN MULTILAYER FOOF CONTACT MATERIALS

For the production of many articles that are intended to come into contact with foodstuffs, adhesives are used. There are non-packaging applications like refrigerators, microwave ovens, kitchen furniture or corks for beverage bottles, but of special importance is the use of adhesives in the production of mass articles like packaging [Fig. 3].

Packaging protects goods against damage and loss. In addition, packaging is essential for safe distribution and warehousing [Fig. 4]. Today’s variety of products would not be possible without modern packaging. The usefulness of modern packaging can be demonstrated very impressively with food. While modern packaging has eliminated the risk of contamination of food by bacteria or insects almost completely, the lack of suitable packaging and distribution results in dramatic losses due to deterioration. A comparison between industrialized and developing countries shows the impact of proper packaging on losses of food during storage and transport. Whereas in the industrialized countries only about 5% of all food decays thanks to the sophisticated packaging technology, in developing countries it is almost two thirds [Fig. 5].

For the production of many of these packagings, today a vast number of adhesive systems are available. Applications include laminations of flexible films to foil, paper/cardboard film combinations, rigid multi-layer plastic packaging, sacks and bags, and labelling – usually without, but sometimes also with direct food contact.

The evolution in materials and processes led to an intensive development of packaging adhesives in recent

decades, so that today there are tailor-made packaging adhesives for all kinds of applications and performance demands. The adhesives that are used for the production of these articles are requested to fulfil all technical requirements as well as the requirements of the European directives and regulations for materials and articles intended to come into contact with foodstuffs.

Adhesives are not yet treated as an individual regulated group, so that the European regulation of adhesives still is not harmonized. However in 2002 the European Commission (DG SANCO) started discussions to regulate adhesives. A first hearing about this subject took place in Brussels in 2001. The current draft (Rev. 5.2 dated 1 July 2004) of the so-called "Super Regulation" intends to include the migration contributions of the adhesives used in multilayer constructions, but without mentioning a specific regulation. In addition adhesives are mentioned as a group of substances to be regulated in the appendix 1 of the new Framework Regulation (EC) 1935/2004. In discussions with FEICA several different approaches were prepared.

From today's point of view the following possibilities for future regulations of adhesives can be imagined:

1. Inclusion in the positive-list system of the Plastic Directive 2002/72/EG or in the succession regulation of the Plastic Directive, called "Super Regulation", which is currently discussed.

2. A specific directive for adhesives with a special positive list for adhesives.

3. The industry works out its own concept which has to be acceptable by the enforcement, and which ensures the compliance of the products regarding Art. 3 of the Framework Regulation (EC) 1935/2004), namely the general demands (not to endanger human health).

POSITIVE-LIST SYSTEM

The positive-list system means that all raw materials and additives used for production of an adhesive have to be listed in a European guideline dealing with adhesives for food contact materials (2002/72/EG, "Super Regulation" or a specific directive). This procedure offers the advantage that finally all substances used have to be evaluated by the European Food Safety Agency (EFSA) and officially authorized. If necessary, limits have to be set in the European Adhesive Regulation. Some of the raw materials and additives used for adhesives are also used for plastics and so are already evaluated. However it has to be considered that short-chain polymers, such as are used in those adhesives whose viscosity is controlled by the length of the polymer chain, are now defined as polymer additives and are not regulated by the list of monomers and raw materials, but must be authorized specifically. However, most raw materials used for the production of adhesive formulations are not used in the already regulated production of plastic materials. These, as well as the short-chain versions of approved plastics, have to pass through an approval procedure before they are included in the list. For this, migration and toxicological studies based on the extent of migration are necessary. Depending on the knowledge of the raw materials these studies can require a lot of effort and be expensive. To get all necessary data for migration tests approximately 3,000–50,000 EUR and for the toxicological dossier approximately 50,000–500,000 EUR (range based on migration levels) are estimated. In addition, reactions and by-products have to be measured and taken into account. Depending on their amount and composition, migration toxicological data can also become necessary for them.

It is obvious that an inclusion in the positive-list system of all substances used in adhesives would be an enormously time-consuming and expensive process. To illustrate this amount of time one should look at the development of the positive list for plastic monomers. When the Plastic Directive 90/128/EEC was established, monomers and other raw materials that were used before 1990 for the production of plastics for food contact, but which had not yet been evaluated by the Scientific Committee for Food (SCF), were included in a B list of temporarily-usable substances pending a decision on inclusion in the positive list (Section A). This B list will presumably be cancelled by 31 December 2007, meaning that it will have taken 17 years until the last 'old' monomers and other starting substances have been evaluated and included in the A list of the approved monomers, or deleted.

It is assumed that some of the raw materials used for adhesives (and especially the additives) will get specific

migration limits based on toxicological evaluation. The compliance with migration limits has to be controlled on the finished food contact material. As adhesives consist of many components a migration test by the old procedure would be very expensive and time-consuming. For many or for most of the components today there are no, or at least no validated, analytical measurement methods.

THE INDUSTRY'S OWN CONCEPT

In contrast to the materials of which food contact articles like packages principally consist, adhesives are usually only a minor component. The tonnage of adhesives is but a minor percentage of the tonnage of, for example, paperboard and plastic. Thus the pressure for adhesive raw material producers to invest time and money in the approval of raw materials is limited, and because of this the adhesive industry will lose a substantial number of raw materials. The consequence could be that some of today's adhesive formulations will not conform to the food regulations anymore and therefore be forbidden for these applications.

The alternative to the positive-list system would be to prepare a praxis-orientated testing concept to guarantee the safe use of adhesives in multilayer food contact materials. This test could be developed by the adhesive industry, including small and medium-sized enterprises, cost-efficiently and without compromise to consumer protection requirements and thus to their own product image. Such a concept finally should be given to the European authorities to be written in a legal format. Most likely, the general principles would be embedded into a European regulation. The necessary details would be laid down in the so-called Practical Guide for food contact materials of the Commission.

The practical guide is based on the fact that adhesives are usually between two other materials, ie, that there rarely is direct contact of adhesive and foodstuff. Hence the components of the adhesives cannot migrate into the foodstuff directly, but have to migrate through a barrier layer [Fig. 6]. For the first time, this view has been described in the so-called "Super Regulation", too. The practical guide also takes into account the relatively small quantity of adhesive compared to the material that forms the food-contact article itself, eg, slim glue lines of folding boxes [Fig. 7].

The evaluation of the migration of adhesive components into foodstuff could be based on the principle of the so-called "Functional Barrier" (FB), which will presumably be considered within the regimentation of the "Super Regulation". The basic idea behind the FB principle is that the food contacting layer(s) (of a multilayer) may act as a barrier for migration of adhesive components (ie, a functional barrier). The performance of a functional barrier depends on the properties of the food contacting layer and the migrating substance. As a consequence, substances that are immobilized in the adhesive or that cannot migrate into the food because of the functional barrier properties of the interior layers under the presumed use conditions, can be omitted from this concept of approval. Then a toxicological evaluation would only be necessary for those substances that can migrate in significant or large amounts.

In reactive systems, by-products are formed, often of unknown composition. Usually it is very difficult to receive sufficiently detailed information about the composition and purity of raw materials from raw material suppliers. This should be considered in the test concept by utilizing practical validated screening methods on components able to migrate. The test should be usable even when one does not have exact knowledge of all individual substances used in the adhesive formulation.

Tests of migration and migration kinetic tests for barrier materials are very time-consuming and expensive. Usually, the adhesive exists in a very thin layer, and the amount of components able to migrate is small, so that such sophisticated migration tests often are not necessary. The test concept hence should be a fast, cost-effective, and relatively easy method of determining compliance. This should be achieved by a combination of extraction tests to determine the potential of components able to migrate, mathematical modelling, and but a few verifying migration tests of the finished food contact materials. Conclusions should be drawn from a tested system with defined layers and layer thicknesses about the compliance of other systems with the same adhesive, without causing the need for new tests.

Regardless of which of these approaches will be incorporated into the regulations, in the future the European idea,

which is characterized by consumer and environmental protection, will lead to a clear expansion of the necessary documentations and surveys of adhesives and adhesive raw materials used for food contact materials. Especially for raw materials it will be necessary to disclose many ingredients, so that processors can execute corresponding surveys. The adhesive industry, too, will have to give much more information to its customers, so that customers can ensure the compliance of their articles with the European food contact material regulation.

MATHEMATICAL MODELLING

Adhesives are considerably more complex systems than mono plastics, so that the modelling methods used for plastics until now cannot be simply transferred to the testing of adhesives. Because of this there is yet no scientifically stable basis for simplified testing of adhesives. Different research institutes have prepared the ground in this field of research and from these preparations such a concept can be developed. The mathematical modelling of migration [Fig. 8] from mono plastics and the estimation of diffusion coefficients as proof (Piringer model) is validated for many plastics and has been included as a possibility to test the compliance in the European Plastic Directive 2002/72/EG (article 8/4) and Practical Guide. The model has been published among others as a guide in the Practical Guide for Food Contact Materials of the European Commission and as a CEN Report. It has been extended to the applicability on multilayer materials in connection with a recently finished research project**.**

To prepare the necessary scientific data a European project is being planned by several research institutes. The project will be assisted by FEICA and by a number of national European adhesive associations, as well as by small and medium-sized adhesive manufacturers.

SUMMARY

Without multilayer food contact materials modern life would not be possible. While there are already European regulations for plastics, for other components like printing inks or adhesives a preparation is intensively discussed. The establishment of a positive-list system for those components will require very much time and money and this surely cannot be accomplished for all formulation components used today. Thus in the future a number of formulations may no longer be legal for food contact materials. A positive-list system absolutely requires an individual compliance check with specific migration limits. It has to be considered that conventional migration test processes are time-consuming and costly and that there are no analytical methods for many components yet.

As a consequence the only possible and useful way is a specific testing concept by a combination of extraction and migration tests and mathematical modelling, that guarantees the safe use of these components in multilayer food contact materials, and which can be utilized cost-efficiently by the industry, especially by small and medium-sized enterprises.

ACKNOWLEDGEMENTS

The author would like to thank Bill Leach, Henkel Adhesives, Hayward, USA, for his valuable assistance.

Fig. 1 Food Contact Articles

Regulation of the European Parliament and of the Council on materials intended to come into contact with food: (EC) 1935/2004

Annex 1: List of groups of materials and articles which may be covered by specific measures

Active and intelligent materials and articles	Plastics
Adhesives	Printing ink
Ceramics	Regenerated cellulose
Cork	Silicones
Rubbers	Textiles
Glass	Varnishes and coatings
Ion-exchange resins	Waxes
Metals and alloys	Wood
Paper and board	

Fig. 2 Specific Regulations

Fig. 3 Packaging

Fig. 4 Distribution with Packaging

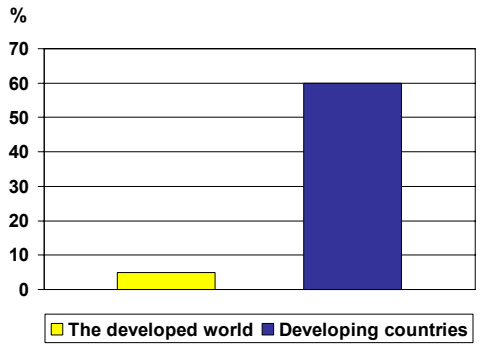

Fig. 5 Loss of Food During Transport and Warehousing

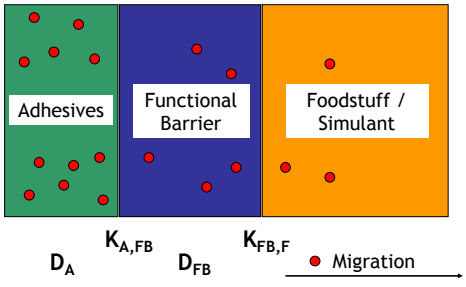

Fig. 6 Migration in Multilayer

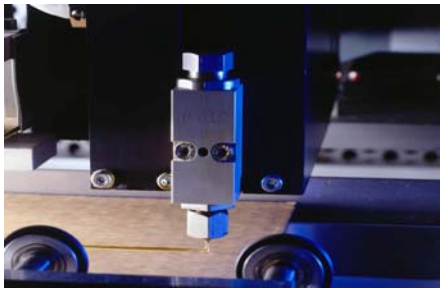

Fig. 7 Adhesive Application

$$-\left|\frac{\partial c}{\partial t}\right|_{total} = -D\left(\frac{\partial^2 c}{\partial x^2}+\frac{\partial^2 c}{\partial y^2}+\frac{\partial^2 c}{\partial z^2}\right)+\left(v_x\frac{\partial c}{\partial x}+v_y\frac{\partial c}{\partial y}+v_z\frac{\partial c}{\partial z}\right)+k_r c^n+k_e\sigma$$

$$\frac{m_{L,t}}{A} = c_{P,0}\rho_P d_P\left(\frac{\alpha}{1+\alpha}\right)\left[1-\sum_{n=1}^{\infty}\frac{2\alpha(1+\alpha)}{1+\alpha+\alpha^2 q_n^2}\exp\left(-D_P t\frac{q_n^2}{d_P^2}\right)\right]$$

$$\alpha = \frac{V_L / V_P}{K_{P,L}}$$

Fig. 8 Migration Modelling (single layer) *Fick's 2nd law*