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Nanotechnological Applications for Food Contact Materials

Summary

Nanomaterials can improve the properties of food contact materials. Innovations of this kind are of particular interest for food packaging made out of plastic materials. The purpose of their use is to improve food storage and so to guarantee both freshness and quality. A further goal is to improve the technical properties of materials in order to make them sturdier more resistant to abrasion, and easier to process. Food contact materials are subject to a number of EU consumer protection regulations. Nanomaterials require authorisation by the European Food Safety Authority (EFSA), being responsible for assessing their safety. For nanomaterials authorised for use in the EU, specifications and restrictions are laid down in order to prevent consumers being exposed to them or to keep exposure as low as possible, and so to rule out any danger to health. At the end of the product lifetime, workers of recycling and waste treatment facilities may be exposed to higher levels of ultrafine particles or particulate matter may also be released unintentionally. To date, however, it has not been demonstrated that recycling polymers containing nanomaterials leads to any increased exposure of employees. With regard to environmental protection, little is known at present about the specific behaviour of nanomaterials and composites during waste treatment processes. There is also a need for comprehensive research on how far nanomaterials can be recycled, in order to develop sustainable nanotechnology.

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Introduction

The term 'food contact materials' (FCMs) covers a large number of materials and objects that can come into contact with food. The objects include dishes, cutlery, saucepans, frying pans, drinking glasses, garlic presses, spatulas, and coffee machines. But all packaging materials, storage containers and processing machinery used in the food industry also belong to this extensive and important category of utensils covered by food law.[1] Not counted among food contact materials are public and private water supply installations that are legally required to remain in a fixed location, antiques, and coating materials that are not separate from the foodstuff and are consumed along with it.[2] Objects that can come into contact with food can be made of a range of materials, for example metal, ceramics, glass, or plastic materials. Nanomaterials can improve the properties of food contact materials. Innovations of this kind are of particular interest in the field of food packaging made out of plastic materials, as they can improve food storage and so guarantee both freshness and quality; in addition, they can improve the technical properties of materials in order to make them sturdier more resistant to abrasion, and easier to process.

In NanoTrust Dossier 004en, published in December 2010, we provided an initial, brief overview of nanotechnological applications in the field of food packaging. In the present Dossier we examine this issue in more detail, and in particular take a closer look at those nanomaterials that have already been authorised in the EU for use in food contact materials made of plastic materials. We also examine more closely the legal framework, environmental risks and possible dangers to health, and the questions of waste disposal and recycling.

The EU Legal Framework

For purposes of consumer protection, food contact materials (FCMs) are subject to a number of regulations. The framework directive is Regulation (EC) No 1935/2004, which is based on the principle that materials and objects intended to come into direct or indirect contact with food must be sufficiently inert. The purpose is to exclude any possibility of materials getting into food in quantities that would pose a risk to human health. The regulation also forbids any unacceptable change in the composition of food and any adverse effect on its smell, taste, colour, or appearance caused by a food contact material.[3]

This framework regulation also applies to what are known as '**active**' and '**intelligent**' **FCMs**. The purpose of 'active FCMs' is to prolong the shelf life of a packaged foodstuff, or to preserve it in a certain condition or improve that condition. They contain components which emit materials that are taken up by the packaged food or the environment surrounding it, or can absorb materials from the foodstuff or its environment. 'Intelligent FCMs' is a term used to refer to materials and objects which monitor the condition of a packaged foodstuff or of the environment surrounding it. These can include sensors which monitor and display, for example, the temperature of a foodstuff. At present there are a number of international research projects in progress for the purpose of developing such 'intelligent' packaging materials for future use.[4] In addition to the framework regulation, Commission Regulation (EC) No 450/2009[5] contains special requirements for the marketing of 'active' and 'intelligent' FCMs. They must undergo a safety assessment and an authorisation procedure. This regulation states explicitly that possible risks arising from nanoparticles must be assessed on a case-by-case basis, as the chemical and physical properties of these particles differ fundamentally from those of materials with larger structures. Until May 2017, no nanomaterial has been authorised for 'active' or 'intelligent' FCMs in the EU.

Plastic materials are another group of materials and objects for which requirements already exist in the EU under the terms of Commission Regulation (EU) No 10/2011[6]; nanomaterials are already in use here. The only materials that may be employed intentionally in the manufacture of plastic coatings in materials and objects are those listed on what is known as the 'Union list' contained in Annex 1 of this regulation. Materials with a nanostructure may only be used when they are explicitly permitted and appear on the 'Union list'. The European Food Safety Authority (EFSA) carries out the safety assessment required for permission to be granted. The Regulation does not contain any definition of what exactly is meant by 'material with a nanostructure'. However, the 'novel foods' regulation[7] contains a definition that applies to all EU food legislation. As of May 2017, the 'Union list' contains 11 materials that fall under the definition of a 'technically manufactured nanomaterial' in the sense of the 'novel foods' regulation (see Table 1).

Nanomaterials for FCMs made of plastic materials

Any food that is not consumed immediately after manufacture requires packaging. Packaging is intended above all to protect food against dirt, oxygen, light or ultraviolet rays, microorganisms and moisture, and it also needs to be safe to use and cheap to produce. In addition, it should be light, able to stand up to processing, filling, storage and transport, and to present no problems for waste disposal or recycling. Many plastic materials, those known as polymers, meet these requirements. The materials most frequently used for FCMs are polyolefins, for example polypropylene (PP) and a range of kinds of polyethylene, and also polyethylene terephthalate (PET), polystyrene (PS), and polyvinyl chloride (PVC).[9] However, there are also problems with plastic materials: gases can permeate them, and they have low resistance to high temperatures and mechanical load. For this reason, additional materials known as additives or fillers are added to polymers in order to improve their properties. Nanomaterials, used as functional fillers, are playing an increasingly significant role here. For example, titanium nitride can improve the thermal and mechanical properties of PET bottles.[10] As outlined above, as of May 2017 11 nanomaterials have been approved for use in FCMs made of plastic materials (Table 1); these have a range of functions, and precise specifications have been laid down to protect consumers.

Table 1:
Nanomaterials permitted in FCMs made of plastic materials, as set out in the 'Union list' (EU No 10/2011, Annex 1, as of May 2017)[8]

FCM No.	Material	Restrictions and specifications
87	Silicon dioxide, silanated	For synthetic amorphous silicon dioxide, silanated: primary particles of 1–100 nm which are aggregated to a size of 0,1–1 µm and may form agglomerates within the size distribution of 0,3 µm to the mm size.
410	Kaolin	Particles can be thinner than 100 nm only if incorporated at a quantity of less than 12% w/w in an ethylene vinyl alcohol copolymer (EVOH) inner layer of a multi-layer structure, in which the layer in direct contact with the food provides a functional barrier preventing migration of particles into the food.
411	Carbon black	Primary particles of 10–300 nm which are aggregated to a size of 100–1200 nm which may form agglomerates within the size distribution of 300 nm – mm. Toluene extractables: maximum 0,1%, determined according to ISO method 6209. UV absorption of cyclohexane extract at 386 nm: < 0,02 AU for a 1 cm cell or < 0,1 AU for a 5 cm cell, determined according to a generally recognised method of analysis. Benzo(a)pyrene content: max 0,25 mg/kg carbon black. Maximum use level of carbon black in the polymer: 2,5% w/w.
807	Titanium nitride, nanoparticles	No migration of titanium nitride nanoparticles. Only to be used in polyethylene terephthalate (PET) up to 20 mg/kg. In the PET, the agglomerates have a diameter of 100–500 nm consisting of primary titanium nitride nanoparticles; primary particles have a diameter of approximately 20 nm.
859	(butadiene, ethyl acrylate, methyl methacrylate, styrene) copolymer crosslinked with divinylbenzene, in nanoform	Only to be used as particles in non-plasticised PVC up to 10% w/w in contact with all food types at room temperature or below including long-term storage. When used together with the substance with FCM No 998 and/or the substance with FCM No 1043, the restriction of 10% w/w applies to the sum of those substances. The diameter of particles shall be > 20 nm, and for at least 95% by number it shall be > 40 nm.
998	(butadiene, ethyl acrylate, methyl methacrylate, styrene) copolymer not cross-linked, in nanoform	Only to be used as particles in non-plasticised PVC up to 10% w/w in contact with all food types at room temperature or below including long-term storage. When used together with the substance with FCM No 859 and/or the substance with FCM No 1043, the restriction of 10% w/w applies to the sum of those substances. The diameter of particles shall be > 20 nm, and for at least 95% by number it shall be > 40 nm.
1016	(methacrylic acid, ethyl acrylate, n-butyl acrylate, methyl methacrylate and butadiene) copolymer in nanoform	Only to be used up to: a) 10% w/w in non-plasticised PVC; b) 15% w/w in non-plasticised PLA. The final material shall be used at room temperature or below.
1030	montmorillonite clay modified by dimethyldialkyl(C16-C18) ammonium chloride	Only to be used up to 12% (w/w) in polyolefins in contact with dry foods to which simulant E is assigned in table 2 of Annex III at room temperature or below. The sum of the specific migration of 1-chlorohexadecane and 1- chlorooctadecane shall not exceed 0,05 mg/kg food. Can contain platelets in the nanoform that are only in one dimension thinner than 100 nm. Such platelets shall be oriented parallel to the polymer surface and shall be fully embedded in the polymer.
1043	(butadiene, ethyl acrylate, methyl methacrylate, styrene) copolymer crosslinked with 1,3-butanediol dimethacrylate, in nanoform	Only to be used as particles in non-plasticised PVC up to 10% w/w in contact with all food types at room temperature or below including long-term storage. When used together with the substance with FCM No 859 and/or the substance with FCM No 998, the restriction of 10% w/w applies to the sum of those substances. The diameter of particles shall be > 20 nm, and for at least 95% by number it shall be > 40 nm.
1046	zinc oxide, nanoparticles, coated with [3-(methacryloxy)propyl] trimethoxysilane (FCM No 788)	Only to be used in unplasticised polymers. The restrictions and specifications specified for FCM substance No 788 shall be respected.
1050	zinc oxide, nanoparticles, uncoated	Only to be used in unplasticised polymers.

Silicon dioxide [SiO_2], silanated

SiO_2, silanated, has been permitted since 1999 in the EU, without any restrictions, as an additive for all kinds of plastic materials. It is used, for example, to improve the stiffness and tensile strength of a polymer and to reduce its permeability to oxygen. SiO_2 can also be used for barrier layers that are added to foil by vapor deposition. Such multi-layer foils have been used for some time for food packaging, for example for potato crisps and cheese.[11]

The EFSA reassessed this substance in 2014,[12] on the basis that it has always been manufactured using synthetic amorphous silicon dioxide in nano form. Surface modification can be brought about by using dimethyldichlorosilane or another silane, in order to ensure that processing can be carried out with the polymer.[13] Primary particles are less than 100 nm in size, but in the powdered form in which it is sold larger aggregates and agglomerates can be found. When SiO_2 particles were incorporated into a polyethylene foil for test purposes, no migration of particles of any size took place.[14] The EFSA therefore sees no grounds for concern about consumer safety.

Kaolin

Kaolin, which is also known as china clay, is a white stone; its main component is kaolinite, which results from the erosion of feldspar.[15] Kaolin is treated with a surface layer of polyacrylic acid and sodium salt in order to make it easier to disperse the substance evenly. Kaolin platelets are less than 100 nm thin, and they are incorporated in a concentration of up to 12% into an inner layer made of ethylene vinyl alcohol copolymer (EVOH) in a multi-layer structure. This layer functions as a gas barrier in FCMs for drinks, sauces, dressings, and sausages, and also in pet food. The substance was evaluated by the EFSA in 2014.[16] No migration of individual platelets into food simulants was found. However, kaolin is composed of a number of metalliferous clay minerals, and it is possible for aluminium and magnesium ions to be released from it. The inner layer of EVOH containing kaolin must therefore be further isolated by an additional functional barrier to prevent the migration of these ions into the food or drink.

Carbon black

Carbon black consists of nano-scale carbon particles that form larger aggregates and agglomerates. The substance is sold in powdered form, and has been in use for decades for a range of applications including car tyres, cosmetics, as black pigment, and also as protection against UV rays for FCMs made of plastic materials. An evaluation of carbon black by the EFSA in 2005 came to the conclusion that the substance itself does not migrate from plastic materials into food, but that it is quite possible for pollutants with aromatic hydrocarbons to migrate into foods such as cooking oil. For this reason, specifications to limit such migration were laid down (restrictions on toluene extractables and on the UV absorption of cylohexane extract).[17]

Titanium nitride nanoparticles

Titanium nitride nanoparticles are used as an additive for FCMs made of PET (for example bottles), in order to improve the thermal properties of the plastic material.[18] The substance was evaluated by the EFSA in 2008 to establish whether it could be used in PET bottles. An application to have its use extended to include foil made of PET was considered in 2012.[19] Titanium nitride nanoparticles are approximately 20 nm in size as primary particles. When they are incorporated into PET, agglomerates with a diameter of approximately 100-500 nm are formed. Titanium nitride is chemically inert and does not dissolve in any of the food simulants that have been tested. The EFSA's evaluation did not find any migration of titanium. The EFSA therefore considers the substance safe when used in a quantity of up to 20 mg per kg of PET plastic material.

Copolymers in nanoform

Copolymers of butadiene, ethyl acrylate, methyl methacrylate, and styrene may be used in the EU in a concentration of up to 10% w/w as an additive (impact modifier) in non-plasticised PVC.[20] Copolymers are made up of various kinds of monomer units, and under the terms of Regulation (EU) 10/2011 only certain monomers may be used for FCMs made of PVC. Since these monomers are incorporated into the polymer matrix having no reactive functional chemical groups, either no migration or only very limited migration of nanoparticles from the FCM into the food can be expected. The EFSA's 2014 evaluation therefore came to the conclusion that there were no grounds for concern about consumer safety.[21]

Montmorillonite clay [nanoclay]

Nano-scale montmorillonite is a natrium-aluminium silicate and is also known as nanoclay, since these layer silicates have at least one nanometer-scale dimension. The platelets are no more than between 1 and a few nanometers thick, and vary from several hundred to thousands of nanometers in length. Montmorillonite is a major component of bentonite (60-80%). The literature indicates that nanoclay is currently the nanomaterial in most frequent international commercial use, with a market share of about 70%.[22] Even small amounts of nanoclay (1-5% w/w) can improve the capacity of FCMs to function as a gas barrier, since gases and aroma materials (carbonic acid, food aromas, etc.) must take a longer route between the platelets positioned parallel to the surface of the plastic material matrix.[23] The gas barrier functions, for example, to prolong the shelf life and improve the aroma stability of food. In order to incorporate layer silicates like montmorillonite evenly into a plastic material matrix, however, the platelets must first be separated from one another and have their surfaces modified, since they would otherwise agglomerate immediately during processing. This surface modification is usually carried out by means of quaternary ammonium compounds, which turn the hydrophilic silicate surface into a hydrophobic (organophilic) one. Internationally, nanoclay is used in multi-layer packaging, for example in bottles for carbonated drinks (beer, soft drinks and thermoformed plastic containers.[24] The use of montmorillonite has been permitted in the EU since May 2017.

Montmorillonite was evaluated by the EFSA in 2015.[25] Tests using a transmission electron microscope (TEM) found instances of separated clay platelets. However, migration of platelets on the nanometer scale is not expected, since the platelets present are of a length and width of several hundred nanometers, they are positioned parallel to the surface of the barrier layer, and they are completely embedded in the polymer matrix. Also the migration of aluminium into 95% ethanol was tested, at a temperature of 40°C over a period of 10 days, at which no significantly higher concentration than in the control sample was found. Nor did the tests find any migration of the ammonium compound into the food test medium. The EFSA therefore considers that there are no grounds for concern on the part of consumers in relation to this substance, as long as it is only used up to 12% (w/w) in polyolefins for long-term storage of dry foods at room temperature or below.

Zinc oxide nanoparticles

The use of zinc oxide in nanoparticulate form as an UV-absorber has been permitted in the EU since 2016, both uncoated and, in order to improve its dispersive properties, coated with a silane. The substance is used in powdered form. Nanoparticles can be found in the final polymer, but mostly in aggregated form. Migration tests showed that zinc ions, but no nanoparticles, were released from the FCM into the test solutions.[26] These release tests thus indicate that, due to the effects of the environment, zinc ions are more likely to become detached from the nano-zinc oxide polymer than whole nanoparticles. However, the quantity released remains below the tolerable upper intake level (UL value) of 25 mg per person per day recommended by the EU's Scientific Committee on Food.[27] Nevertheless, in combination with zinc ion intake through food it is quite possible for this level to be exceeded.

Other nanomaterials not permitted in the EU

A number of further nanomaterials are mentioned in the literature that are used in FCMs, but which are not (yet) permitted in the EU.[28] These include **titanium dioxide** (TiO_2), which can be incorporated into polymers as an UV-absorber or to make use of its anti-microbial effect. There is also a discussion about the possibility of adding nanoparticles of **calcium carbonate** ($CaCO_3$), which can improve the strength and thermal resistance of plastic materials, to FCMs. On the international market (e.g. in the USA and Asia) FCMs containing **nanosilver**[29; 30] are also in use (e.g. for storage containers and chopping boards); the antimicrobial effect of this substance is said to prolong the shelf-life of food.

Protecting the health of consumers

There can only be a danger to consumers' health from nanomaterials if they are exposed to them, that is to say if the nanomaterial migrates into a foodstuff or drink, for example from the packaging. All nanomaterials permitted for use in FCMs in the EU therefore have to undergo a safety test in which, using standardised test methods, it is established whether such a migration is possible. A range of food simulants, for example alcohol or acid, are used to test the release behaviour of the substance. However, the testing methods that are laid down and standardised in the EU are not validated for nanomaterials. It is a major undertaking to demonstrate the presence of nanoparticles in food, as it is very difficult to distinguish quantitatively between nanoparticles that have been released and the ions produced as they dissolve. Such difficulties lead to some uncertainties, and so make it harder to interpret migration tests with nanomaterials. In many cases, the methods used in scientific studies are not adequately described.[31]

Where nanomaterials permitted in the EU are concerned, specifications for and restrictions on use have been laid down in order to ensure that consumers are not exposed to the materials or that, if they are, the exposure is as low as possible (see Table 1). For example, a plastic material layer into which kaolin has been incorporated must be prevented by means of an additional barrier layer from coming into direct contact with a foodstuff. In general, no migration of nanoparticles permitted in the EU for contact with food into food simulants, has been determined either by the EFSA or by other bodies[32]. If migration does occur, it has been found to be negligible in terms of any health risk. As already mentioned, though, it is possible for ions to be released from some nanomaterials, for example zinc oxide, nanoclay, and nanosilver, and for these to migrate into food. It is therefore very important that when the risks of such nanocomposites are assessed, such processes also have to be considered. At this point one must also mention the fact that the migration values for separated ions are very specific to the nanomaterial, and can vary considerably depending on where they are actually being used. The fundamental principle is that the limits that are laid down or recommended for intake through food, for instance, should not be exceeded. In an acidic environment, for example, it is possible for aluminium ions to be released from nanoclay. Tests of plastic bags for food sold in shops have shown that both aluminium ions and aluminium nanoparticles can be released. However, the quantity is very low, below the EFSA's tolerable weekly intake (TWA) level of 1 mg of aluminium per kilo of a person's body weight.[33]

The chemical substances used to treat the surfaces of nanoparticles are also a relevant consideration for possible health risks. If, for example, a nanoclay is to be incorporated into a polymer matrix, it is always necessary – except in the case of polyvinyl alcohol – to use a quaternary ammonium compound (QAC) as a 'mediator', since otherwise the hydrophilic clay will not be satisfactorily incorporated into the hydrophobic plastic material. The nanoclay and the QAC, as the organic modifier, form a unit that cannot be separated again without special chemical processes. However, tests have shown that under certain conditions the compound of plastic material, nano-scale additive and QAC can once again break down.[34] Since QACs have lipophilic properties, there is a particular risk of migration from food packaging where the packaged food is fatty. Unsuitable plastic materials (e.g. polyamide) can also be weakened by acidic foods, and there is a possibility of QAC migration into the foodstuff here too. In order to estimate how likely this is, one therefore needs to know exactly what the food to be packaged contains; in particular, information about its polarity and lipophilic properties is crucial. One way to minimise the release of QAC and thus prevent consumers' health being endangered is to ensure that the treated plastic layer does not come into direct contact with the foodstuff. The example of a nanomaterial with a surface layer modified by QAC shows that toxicity depends not only on the particle itself but also on its surface coating. A comprehensive survey of the literature on pollution caused by QACs also stresses that they are extremely toxic for aquatic organisms and take a long time to degrade biologically.[35] Plastic packaging that is thrown away is exposed to the weather, which means that QACs can be released from the polymer matrix and get into waters.

Safety testing of nanomaterials for FCMs permitted in the EU also extends to substances used to modify the surfaces of such materials. The only substances that may be used for this purpose are those that are themselves permitted, and/or those for which a specific migration limit has been laid down, for example montmorillonite (see Table 1).

Safety during waste disposal

Although we know a considerable amount about potential effects on health when polymers containing nanomaterials (nanocomposites) are in use, not much research has been done so far on possible effects on the environment during waste disposal. EU regulations only relate to health risks during use. Dangers to the environment or to human health during waste disposal are not relevant to whether or not a substance is permitted. A survey of the literature carried out by the Organisation for Economic Cooperation and Development (OECD) points out, for example, that nano-scale additives in PET bottles can be released again when the bottles are recycled or regranulated.[36]

Recycling and waste treatment facilities may expose their workers to higher levels of ultrafine particles or particulate matter, and this may also cause nanomaterials to be released unintentionally as a result of mechanical or chemical stress.[37] To date, however, it has not been demonstrated that recycling polymers containing nanomaterials lead to any increased exposure of workers. In order to rule out any increase in workplace pollution, it would be necessary to carry out a larger number of studies and to measure levels of exposure in workplaces.

Initial laboratory tests, for example tests on polymers containing CNTs, have shown that no increased exposure to individual CNT fibres occurs when these polymers are ground or crushed.[38] Another study came to similar conclusions for epoxy resin and polycarbonate compound materials containing CNTs.[39] This study also found that the dust released consists of larger agglomerates in different forms, but never of single released nanoparticles. However, a study that simulated abrasion processes has shown, by using electron microscopy, that material abraded from polymer composites contained a significant proportion of individual CNTs and CNT agglomerates.[40] All these studies, though, agreed that any potential release depends very much on the actual application, on the carrier matrix itself, and on the processes involved, for example abrasion, crushing, grinding, and so on. Other studies that simulated the ageing processes of nanocomposites have already attempted to standardise testing protocols as a way of making it easier to com-

Conclusion

Nanomaterials can significantly improve the properties of plastic materials, and this is why materials known as 'nanocomposites' are increasingly interesting for manufacturers, especially for food packaging. Nanomaterials used in food contact materials in the EU require authorisation, and the European Food Safety Authority (EFSA) is responsible for assessing their safety. This is to ensure that nanomaterials do not pose any danger to the health of consumers. At present, little is known about the environmental behaviour of nanomaterials during the end of life phase of FCMs. Nor have many studies been carried out to investigate the possible release of nanoscale additives during waste treatment processes or the question of the exposure of workers during such processes. Further studies and exposure assessments are needed here. If nanotechnology is to be made sustainable there is a need to ensure that, in the framework of a circular economy, plastic materials with nano-scale additives can also be recycled or used for energy recovery in a secure way.

pare such studies and to make their findings more conclusive.[41; 42] Nevertheless, significantly more studies would be needed in order to draw general conclusions in the field of FCMs.

With regard to questions of environmental protection, we know just as little at present about the specific behaviour of nanomaterials and composites during waste treatment processes (see NanoTrust Dossier Nr. 044en). There are still large gaps in our knowledge of the ultimate environmental fate of nanomaterials that were originally processed in FCMs or composite materials (that is to say knowledge of exact application and input quantities, form, possible transformation processes, toxic endpoints, sinks, etc.). In general, there is a lack of comprehensive studies of the recyclability and usability for energy recovery of composite materials, which can only be re-used as FCMs in some exceptional cases, for example PET bottles. At the moment, the subject about which we know least is the precise role of nanoscale additives and filler materials in composite materials during solid waste treatment processes and landfilling.

Notes and References

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