

Chemical Migration from Plastic Types of Food Contact Materials

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Abstract

Foods are exposed to contact materials during all steps passed from farm to fork. Regulation (EC) No 1935/2004 was set in the European Union (EU) to provide safe FCMs and to explain the general requirements of the materials. Plastic materials and articles intended to come into contact with food are regulated by Commission Regulation (EU) No 10/2011. Annex I of Commission Regulation (EU) No 10/2011 contains the Union list of authorized monomers, additives, polymer production and other starting substances. There are 885 authorized food contact material substances in the list. These listed substances called as “*Intentionally Added Substances (IAS)*” can be used to manufacture plastic materials, with the restrictions and specifications established in the list. The contamination of foods due to the release of chemicals from packaging materials can be originated from the substances used in their formulation (IAS) but also from interactions between different ingredients, degradation products or from the presence of impurities in the raw materials (so called “*Non Intentionally Added Substances-NIAS*”). The components from food contact materials must not migrate into the foods in unacceptable quantities. Therefore, substances used in the manufacture of FCMs are regulated with maximum limits that may migrate into foodstuffs without causing any health concerns. There are two migration limit set for plastic based materials and articles: *Overall Migration Limit* and *Specific Migration Limit* (SML). SML are set for individual authorized substances based on toxicological evaluation. In the scope of this study, plastic type of food contact materials are classified, and migration concept is explained, the regulations about FCMs and analysis method on chemicals migrated are reviewed.

Keywords: Food contact materials, intentionally added substances, non-intentionally added substances, migration limits, and plastic regulations

INTRODUCTION

Foods are exposed to contact materials including cutlery and dishes, containers, processing machine, and packaging materials during all steps passed from farm to fork. (Simeneau, 2008; EC, 2004). Food industry has been conducting research and development activities on food packaging to increase shelf life, keep the food quality at optimum level, attract consumer interests, and reduce waste. A package material for any type of food should minimize aroma and flavor losses, constitute an excellent barrier for gas and water, provide a perfect hermetically sealed seam, as well as have a good mechanical properties.

Food contact materials including food packaging are generally based on paper, metal, ceramic, aluminum, lacquers and coating, and plastic (Krochta, 2007; Driscoll & Rahman, 2007; Robert, 2012).

Food packaging is used to increase shelf life, to keep food quality at optimum level, to attract consumer interest, to facilitate the sale and distribution (Robert, 2012). Foods

packaging provides information to consumers on product name, brand name, net weight, manufacturer information, price, production date, as well as the nutrient values in addition to keeping food at the desired amount in a single vessel and making it easier to bring a number of units to be moved into a single cluster and use (Çinibulak, 2010). Therefore, food industry makes expenditures on the research and development activities of food packaging systems. The degree of the final product quality and safety, and consumer expectations from the ergonomic features of the package affects the acceptance criteria of a package material. A package material for any type of foods should minimize aroma and flavor losses, constitute an excellent barrier for gas and water, provide a perfect hermetically sealed seam, as well as have a good mechanical properties and offer chemical and biological protection against contamination (Simeneau, 2008). Glasses, metals, paper, ceramic, and plastics are the most used materials for food packages. Glasses are inert packaging material and its shows heat resistance to thermal processing has advantages of providing good strength under compression and heat. Glasses as well as metals like steel and aluminum act a barrier to gases, water vapor and aromas. Paper based packaging materials produced from wood pulp, rags, and other waste have been reported to be used since the seventeenth century. Ceramic type packaging materials including glass and pottery are produced at high temperatures from non-metal inorganic material produced by high temperatures (Krochta, 2007; Driscoll & Rahman, 2007). Plastic packaging materials are made up from polymers by adding additives, processing aids, catalysts, and plasticizers.

Chemical components of packaging materials may migrate in to foods when they contact with them. This type of transfer is called as chemical migration, which is a mass transfer operation. Diffusion the macroscopic movement of molecules from high to low concentration is the main mechanism in migration. The migrated chemicals from packaging materials can be originated from the substances used in their formulation and also from interactions between different ingredients, degradation products or from the presence of impurities in the raw materials. The duration of the contact between the material and food, temperature profile during interaction and the physicochemical behavior of the packaging material are the main drives for the migration (Simeneau, 2008).

Keeping consumer health safe, components of food contact materials shall not migrate into the foods. Therefore substances used in the manufacture of the packaging materials are regulated with maximum limits that may migrate into foodstuffs without causing any health concerns. To analyze migrated chemicals, food simulants are used to test migration in the scope of compliance with regulations. Sophisticated equipment such as liquid and gas chromatography equipped with mass spectrometry and inductively coupled plasma mass spectrometry have been used successfully for migration analysis so far.

In the scope of this study, plastic type of food contact materials, the chemical migrations from packaging materials in to food and simulants, the analytical techniques and the recent international legislations which regulate packaging materials and chemical migration are reviewed and discussed in details.

Plastic Type Food Contact Materials

The starting substances of a plastic materials are mainly monomers. They react with other starting substances to make a large chain structure called as polymer. Polymers constitutes the main structural component of the plastics (EC, 2011). That process is known as polymerization (Selke, 2005). By application of heat and pressure, those high molecular weight polymers can be molded to get required final products and shapes including films, trays, bottles, and jars (Krochta, 2007). Additive such as antioxidant, solvents including printing inks, plasticizers, thermal stabilizers, light stabilizers are benefited to formulate

plastic resin along with the base polymer (Driscoll & Rahman, 2007; Lau & Wong, 2000; Arvanitoyanni & Kotsanopoulos, 2014) . In addition, organic or inorganic materials can be used as printing agents to coat plastic materials and articles. Polymer additives improves plastic flexibility, and polymer resistance to degradation by heat and light (Krochta, 2007; EC, 2011).

Depending on the heat resistance, polymers can be grouped in to two main group as thermoplastic and thermoset polymers. Thermoplastic polymers are linear or branched without crosslinks between polymer chains and can be softened and molten when heated, and can return to their original condition once cooling is applied. Despite of thermoplastic polymer, thermoset polymers cannot easily molten, therefore it plays an important role for making package closures, especially in rigid closure. If a plastic compose of one type of monomer with a regular repeating unit in the structure, it is called homopolymer. On the other hand, if it is made from multi type of monomer with different molecular place in the structure, it is called copolymer. (Krochta, 2007; Selke, 2005).

Plastics materials used in production of food contact materials

The common polymers used in food packaging include high density polyethylene (HDPE) and low density polyethylene (LDPE), polyethylene terephthalate (PET), polypropylene (PP), polystyrene (PS), and polyvinyl chloride (PVC), ethylene vinyl alcohol (EVOH), polyvinylidene chloride copolymers (PVDC), and ethylene vinyl acetate (EVA).

High density polyethylene (HDPE) is a linear polymer of ethylene, which can be represented as $-(CH_2-CH_2)_n-$. Since HDPE has a pigmented structure, it makes the containers opaque. It is commonly used in plastic milk bottles. Its structure prevents degradation caused by light, and enables the product more desirable for marketing (Selke, 2005; Lee, et al., 2008).

Low Density Polyethylene (LDPE) is also polymer of ethylene, compared to HDPE it is polymerized at high temperature and pressure. Therefore its polymer structure has branched structure. LDPE can be used in bread bags and squeezable drink bottles. Moreover it can be used as a heat seal layer and a moisture barrier (Selke, 2005; Lee, et al., 2008).

Polypropylene (PP) has high crystallinity, very low density and good clearness. Its monomer is propylene. The advantages of PP compared to PE, it has higher tensile strength, stiffness, hardness and a higher melting temperature (Luciano & Sara, 2016).

Polyethylene Terephthalate (PET), thermoplastic material, includes polymerized units of the monomer ethylene terephthalate. It is mostly used in plastic bottles for carbonated soft drinks and drinking water etc. (Selke, 2005).

Polystyrene (PS) is an aromatic polymer made from styrene with a benzene ring attached to every carbon. The brittleness and low impact strength are the characteristic features of PS. Since its thermal conductivity is low, it can be used insulating material. It is mostly used in disposable cups for hot beverages (Selke, 2005; Robert, 2012).

Polycarbonate (PC) is synthesized from its monomer Bisphenol A. Since its strength is high, it is successfully used for refillable water and milk bottles (Selke, 2005).

Polyvinyl Chloride (PVC) is polymer of vinyl chloride. It has resistance characteristics to chemicals including acids and bases. It can be used for blister packs for meat products (Marsh & Bugusu, 2007).

Polyvinylidene Chloride (PVDC) is a homopolymer of vinylidene chloride. Eventhough the structure of this polymer is similar to PVC, there is an additional double chlorine substitution PVDC. It resists chemicals, moreover it shows low water, gas, aroma, and flavor permeability, and high strength (Driscoll & Rahman, 2007) .

Polyamides (PA) are usually produced by hydrolytic or anionic polymerisation of caprolactam. This form of polyamides known as PA6. There are other form of polyamide as PA66, formed by polycondensation of 1,6-diamino hexane and adipic acid. PAs are used as gas barrier in multilayer packaging for meat, fish or cheese. Moreover, it is commonly used as sausage casings and cooking utensils including spoons and spatulas due to its higher melting point (Heimrich, et al., 2012)

Multilayer Formulations

Plastic materials are either made up from one type of polymer called as monolayer plastics or from different layers of plastics held together by adhesives, called as multilayers. Despite of glass and metal materials, plastics do not provide a total barrier to gases, water vapor, and aromas. Therefore plastics are often combined in layers to increase the functionality of the final product (Krochta, 2007). Plastic paper, and/or aluminum can be combined to have efficient functions which is not possible with a single layer structure. Multilayer structures can be produced by polymer coating to a paperboard, lamination two or more plastic with an adhesive and co-extrusion. Layer levels in the multilayer materials were changing between 2 and 10. Examples for multilayer composition can be given as PE/PE/PE, PA/PP, PET/PE/PET, EVOH/PP/PP/PP/PE (Selke, 2005).

Mieth, et al. (2016) lists the most common polymers in multilayer packaging materials, their functions in the packaging and some applications. Polyethylene is used as heat sealable food contact layer moisture barrier which can be combined with gas/aroma barriers such as polyamide. Polypropylene moisture barrier to provide mechanical strength can be a coated with heat seal coatings (PVDC) can be combined with gas/aroma barriers such as PA. Polyethylene terephthalate (PET) can provide gas/aroma and moisture barrier to provide mechanical strength heat resistance. Polystyrene has a gas permeability and printability properties. Therefore it can be outer surface in the structure and can be combined with gas/aroma barriers (coextruded or laminated) (e.g PS/PVDC/PE). Polycarbonate can serve as heat resistant and moisture barrier with its mechanical strength. Ethylene vinyl alcohol (EVOH) is used in modified atmosphere packaging packing of oxygen-sensitive food. It serves as oxygen barrier and can be sandwiched (coextruded) between PE or PP, in some applications also sandwiched between PET, PA or PS (Mieth, et al., 2016).

Legislation

In the European Union (EU), Regulation (EC) No 1935/2004 sets up the criteria which aims to provide safely use of FCMs and articles (EC, 2004). The Regulation states that individual regulations may be required for different groups of materials including plastics, paper and board, metals and alloys, adhesives, printing inks, etc. can be adopted at EU level.

Plastic materials and articles intended to come into contact with food are regulated by Commission Regulation (EU) No 10/2011. Annex I of Commission Regulation (EU) No 10/2011 contains the Union list of authorized monomers, other starting substances, macromolecules obtained from microbial fermentation, additives and polymer production aids (EU, 2011). Annex I of Commission Regulation (EU) No 10/2011 contains the Union list of authorized monomers, other starting substances, macromolecules obtained from microbial fermentation, additives and polymer production aids. It is indicated that potential health risk from the polymers can be ignored, since their molecular weight are above 1000 Da, therefore they are excreted from the body. It can be important only if non- or incompletely reacted monomers, other starting substances, low molecular weight additives migrates from the plastic food contact material into foods. Therefore (EU) No 10/2011 stated that monomers,

other starting substances and additives should be risk assessed and authorized before their use in the manufacture of plastic materials and articles. There are 885 authorized food contact material substances in the list. These listed substances called as “Intentionally Added Substances (IAS)” can be used to manufacture plastic materials, with the restrictions and specifications established in the list (EU, 2011).

The contamination of foods due to the release of chemicals from packaging materials can be originated from the substances used in their formulation (IAS) but also from interactions between different ingredients, degradation products or from the presence of impurities in the raw materials (so called “Non Intentionally Added Substances-NIAS”). Since it is impossible to list and consider all impurities in the authorization, EU 10/2010 gives the responsibility to business operator for risk analysis and taking further risks. Therefore they may be present in the material or article but not included in the Union list (EU, 2011). Substances used in the manufacture of FCMs are regulated with maximum limits that may migrate into foodstuffs without causing any health concerns. There are two migration limit set for plastic based materials and articles: *Specific Migration Limit (SML)* for individual authorized substances fixed on the basis of a toxicological evaluation and *Overall Migration Limit* – 10 mg of substances/dm² of the food contact surface for all substances that can migrate from food contact materials to foods (EU, 2011). Since, overall migration includes all kind of migrated substances, it sets out the general principles of safety and inertness for all Food Contact Materials (FCMs). In Turkey, the Minister of Food, Agriculture, and Animal was released the Turkish Food Codex Communiqué on food contact materials within the frame of compliance with EU (Turk Gıda Kodeksi Tebliği, No:2013/34; No:2012/30, No:2013/35).

Migration Analysis

To determine migrated chemicals into food either for specific or total migration, analysis is performed in food simulants, not actual foodstuffs. Food simulants are used as substitutes for food due to the complexity and variety of foodstuffs, simplification of chemical analysis, and make comparable results between different laboratories.

There are five simulants described in the legislation for plastic (EU 10/2011): 10% ethanol (v/v) in aqueous solution (simulant A), 3% acetic acid (w/v) in aqueous solution (simulant B), 20% ethanol (v/v) in aqueous solution (simulant C), 50% ethanol (v/v) in aqueous solution (simulant D1), vegetable oil (simulant D2), and poly(2,6-diphenyl-p-phenylene oxide, particle size 60-80 mesh, pore size 200 nm, commonly named as Tenax) (simulant E). Food simulants A, B and C are used for hydrophilic foods. Food simulant B is used for acidic foods with pH below 4.5. Food simulant C is used for alcoholic foods with an alcohol content of up to 20 %. Food simulants D1 and D2 are assigned for lipophilic foods. Food simulant D1, D2, and E are used for alcoholic foods with an alcohol content of above 20 %, for fatty foods, and for dry foods, respectively (EU, 2011).

Migration experiments shall be conducted under standardized conditions of contact time and temperature to get the worst scenario for the labelling information on the maximum temperature of use.

Total/Overall Migration Analysis

Overall migration analysis are based on gravimetric methods. For the determination of OM, the corresponding CEN standards are EN 1186 series (EN 1186 1-15). The representative for time and temperature conditions are provided in the Annex III (selection of

proper food simulant) and V (time/temperature conditions) of the Regulation EU 10/2010 (EU, 2011; EN, 1186-1:2002- EN 13130-1:2004).

Specific Migration Analysis

Specific migration analysis are based on chromatographic methods. Therefore, the analytical steps includes extraction and sample clean-up. The general information about specific migration are provided in EN 13130 series Part 1 (Kassouf, et al., 2013). The representative time and temperature conditions are provided in the Annex IV of the Regulation EU 10/2010 (EU, 2011). Simeneau (2008) summarized the official methods used for the specific migration analysis. Gas chromatography equipped with mass spectrophotometry (GC-MS), high pressure liquid chromatography, GC-MS equipped with head space and liquid chromatography-quadruple- time of flight, high pressure liquid chromatography (HPLC) equipped with diode array and fluorescence detector are the common sophisticated equipment used for specific migration. Headspace solid phase micro extraction (HS-SPME) coupled to gas chromatography/mass spectrometry (GC-MS) can be used for volatile substances (Heimrich, et al., 2013). HPLC with chemiluminescent nitrogen detection was used to determine cyclic oligomer of caprolactam (Darowska, et al., 2003).

Migration Studies on IAS and NIAS

It has been known that the technology of PET bottle production causes thermal degradation of the polymer and this process can lead to aldehyde formation. Bach et al. (2013) studied the aldehyde contaminations to mineral water stored in PET bottles. It was reported that the concentration of acetaldehyde in water stored in PET bottles changed with the base the concentration of acetaldehyde in PET material.

Carneado et al. (2016) also investigated the migration of aldehydes as well as trace metals and other compounds in to water bottled in PET type plastics and stored at 40, 50, and 60 °C.

It was observed that, migration of formaldehyde, acetaldehyde and antimony (Sb) were bound on the temperature and the presence of CO₂. Moreover, a degradation compound of phenolic antioxidants as 2,4-di-*tert*-butylphenol was detected.

Migration of antimony trioxide (Sb), widely used in the polycondensation reaction step of PET bottles as a catalyst in to water stored at 4 and 20 °C was found as insignificant. However its concentration was increased if the storage temperature increased to 60 °C (Chapa-Martínez, et al., 2016). Fang et al. (2017) concluded that migration of antimony increased with the storage temperature as well as pH of the water.

The other study reported by Ohno et al. (2001) reveals that extrusion, storage, UV treatments, and sunlight exposure significantly affected concentrations of Irgafos 168 and the degradation products from PP. 2,4-Di-*tert*-butylphenol was the major degradation product produced by UV irradiation, but tris (2,4-di-*tert*-butylphenol) phosphate was the major degradation product produced by extrusion, storage, and sunlight exposure. On the other hand it was concluded that the degradation products showed little health risk.

During the polymerization process of PS, styrene oligomers may occur. On the other hand, Nakaia et al. (2014) concluded that these substances have no estrogenic activity. The findings by Marć & Zabiegała (2017) supported that as well. They stated that the risk of the genotoxicity of styrene oligomers that migrate from polystyrene food packaging into food is very low.

Disposable containers with PS lids are used for hot beverages including coffee and tea. Those types of lids have been reported to release low molecular weight organic compounds

such as styrene and ethylbenzene into the gaseous phase causing direct exposure of consumers to these compounds during drinking (Biedermann-Brem, et al., 2008).

Bisphenol A (BPA) is the monomer of PC. Specific migration limit of BPA was set by The European Commission as 600 µg/kg food (EU, 2011). The studies showed that BPA is an endocrine disruptor (Krishnan et al. 1993). There are valuable studies which proved that small amounts of BPA migrates from polycarbonate material into foods (Brede, et al., 2003-Guarta, et al., 2014). Based on those type of studies, European Union forbidden the use of PC feeding bottles for infants (EU, 2011).

Fang, et al. (2017) reported that BPA was detected in 77% of samples (40 water sample in PC bottles). After one year storage of the same samples, BPA was detected in % 60 of the fresh water. It was concluded that Bisphenol A may originated from the non-polymerized BPA during PC production and degradation of the PC during the reused of the bottles.

Drowska et al. (2003) highlighted that caprolactam, cyclic monomer of PA and its oligomers are the major migrating substances from the materials. Their concentration increased in final product of PA6 after the thermal extrusion of the PA6 film from a granulate. They proved that 95% ethanol and isooctane were not suitable as D2 substitutes for overall migration analysis.

Plasticizers are used in plastic production to increase the functionality of the material by improving plasticity, durability, fluidity, flexibility [54]. There are only five Phthalate plasticizers, namely di(2-ethylhexyl) phthalate (DEHP), di-n-butyl phthalate (DBP), benzyl-butylphthalate (BBP), -di-isononylphthalate (DINP), di-isododecylphthalate (DIDP), which are allowed to be used in plastics with restrictions (EU, 2011). They are commonly used for PVC. On the other hand, it finds application in PP production as well.

Therefore, Fang et al. (2017) studied to determine the level of phthalate migration from polypropylene food containers into food simulants under different heating time at microwave oven. Their findings indicated that pH as well as heating time affect DEHP and DBP migration. Yang et al. (2016) concluded that the concentration of DEHP and DBP was highest under strong acidity and prolonged heating time. Moreover the highest migration of DBP from the PP food container exceeded specific migration limit.

CONCLUSION

Chemical constituents from all type of food contact material may migrate in to foods in small amounts when they contact with certain type of food. This type of transfer is called as chemical migration. The migrated chemicals from packaging materials can be originated from the substances used in their formulation and also from interactions between different ingredients, degradation products or from the presence of impurities in the raw materials. Substances used in the manufacture of the food contact materials are regulated with maximum limits that may migrate into foodstuffs without causing any health concerns. The recent concern of the scientists are to define and evaluate the toxicities of the specific migrated chemicals including non-intentionally added chemicals (NIAS) rather than evaluation of total migration. However, there are some challenges which is needed to be solved by scientist. Those includes the lack of commercial reference standards and a need for rugged multi residue method. Moreover, more studies are needed to find out the effect of combination of different type of plastics as multilayer formulations as well as different usage conditions.

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