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Review Article

A Review of Plastics Use in Winemaking: HACCP Considerations

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Abstract: Use of plastics is ubiquitous in food and beverage industries, with expanding usage in wine production. Common plastic additives, used to modify and improve applicability and durability of plastics, include phthalate plasticizers and bisphenols. Phthalates are used in many products from polyvinyl chloride (PVC), lubricants, and emulsifying agents. Bisphenols such as bisphenol A (BPA) and related BPA non-intent (BPA-NI) alternatives are used to harden plastics and are commonly used in polycarbonate plastics and epoxy coatings. Migration of bisphenols and plasticizers into wine from plastic containers and closures has been studied through the utilization of analytical tools such as GC-MS and LC-MS. Foodstuffs can become contaminated with plastic additives through food-contact processing and packaging materials, leading to environmental and human health concerns. This work reviews current food product use and regulations regarding plastic additives and potential leachates, particularly in wines, hazard analysis and critical control points (HACCP) approaches, alternative plasticizers, and bio-based plastics.

Key words: bisphenol, epoxy, leachate, packaging, phthalate, plasticizer

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1 Introduction

Due to their malleability, versatility, and low cost, plastics have become ubiquitous in present day products. Food stuffs may contact plastics through many pathways including food packaging, long-term product storage, and food transportation. Plastics are increasingly used in wine processing and packaging materials. Annually, about 8% of the world's oil production goes toward producing the approximately 250 million t_m of plastics used globally, of which roughly 30% of plastics are used for packaging (Robertson 2013). Plastics are considered to be biochemically inert and unable to penetrate through cell membranes because of their large molecular size, preventing them from interacting with the endocrine system. Nevertheless, additives, unreacted feedstock monomers or oligomers of the component plastics, or non-intentionally added substances (NIAS), which could include plastic degradation products and other potential chemical side reactions from the manufacturing process, could potentially migrate into the wine, may have biological consequences (Paseiro-Cerrato et al. 2017, Teuten et al. 2009), and may pose a food safety risk or otherwise be of concern to the quality or marketability of wine.

The goal of food safety practices is to limit the presence of food-borne hazards in food at the point of consumption. Food safety hazards are usually the result of physical, chemical, or biological factors. Since food safety hazards can occur at any stage in the food chain it is essential that adequate controls be in place. Hazard and Critical Control Points (HACCP) and quality assurance systems like the International Standardization Organization (ISO) 9000 series, and its food safety derivative ISO 22000 have been developed to prevent food safety risks and consequently provide a competitive advantage to producers that implement such systems (ISO

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2005). The purpose of this review is to point out potential plastics related hazards in wine that could be addressed by HACCP principles. Scalping, sorption, permeation and effects on product quality by plastic additives and wine are not the focus of this work and will be expanded upon in a follow-up review.

2 Review of Plastics

Plastics are synthetic or semi-synthetic polymers made from a wide range of moldable organic polymers set into a rigid or semi-elastic solid. The geometric structure, including conformation, configuration, and branching of polymeric chains, and degree of crosslinking with itself or other molecules determines the physical and chemical properties of the plastic. Properties of polymers, including density, thickness, and transition temperatures are determined by their molecular composition and structure, molecular weight, and degree of crystallinity, which affects optical transparency in plastics (Krimm and Tobolsky 1951, Robertson 2013). Molecular orientation of polymer chains determines whether plastics are amorphous or semi-crystalline (Robertson 2013, White and Spruiell 1981). Amorphous polymer chains are disordered, have no melting point, and gradually soften with increasing temperature. Examples of amorphous polymer chains include polystyrene (PS) and polyvinyl chloride (PVC). In contrast, semi-crystalline polymers usually exhibit distinct phase transition temperatures: a sharp melting and glass transition temperature (T_g) such as polyethylene (PE) and polypropylene (PP).

Plastic polymers can be divided into three categories: thermoplastics, theromosets, and elastomers (Klein 2011). Thermoplastics consist of long, linear, saturated carbon-carbon chains that extend in one dimension. Molecular chains of thermoplastics can move independently because they are not crosslinked. Thermoplastics can be reused because they can be repeatedly

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melted and solidified by heating and cooling (Robertson 2013). Unlike thermoplastics, thermosets form irreversible crosslinks between chains during processing and cannot be re-melted and reprocessed (Lithner et al. 2011, Robertson 2013). Elastomers share properties of thermoplastics and thermosets. Elastomers have wide crosslinks between molecules, which allow mobility of molecular chains resulting in soft and elastic properties. Rising temperature increases elasticity, but like thermosets they cannot be melted without thermal decomposition (Shanks and Kong 2013). Examples of types of plastic are given in Figure 1.

Due to their mechanical properties, thermoplastics are the most widely used plastics, accounting for more than two-thirds of all polymers used globally (Robertson 2013). Common thermoplastics belong to a few generic plastic resin families identified by the Resin Identification Codes (RICs) that aid sorting and recycling in the waste stream (Table 1) (D20 Committee 2010). The current ASTM D7611 gives codes for the six most commonly found resin types, in order from numbers 1-6: polyethylene terephthalate (PETE, PET); high density PE (HDPE); PVC (V); low density PE (LDPE); PP; and PS. All other resins, including PC, acrylonitrile butadiene styrene (ABS), nylon and other materials made with more than one type of resin from Nos. 1-6, are marked with a No. 7 (ASTM International 2013).

3 Plastic Ingredients

3.1 Additives to Plastics

Additives are inorganic or organic substances that enhance the processing and properties of plastics (e.g. stabilizers, plasticizers, biocides, flame retardants, pigments, and others); or act as filler (e.g. carbonates and silicates) to extend the volume of material and reduce production costs (Deanin 1975). Stabilizers may include hydrogen donating antioxidants (e.g. hindered

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phenols and secondary aromatic amines), hydroperoxide decomposers (e.g. organosulfur compounds), heat stabilizers (e.g. lead, tin and mixed metal compounds such as calcium-zinc), light stabilizers (e.g. hindered amine light stabilizers [HALS]), and UV absorbers (e.g. benzophenones) that inhibit the formation of free radicals and photo-oxidation reactions such as those catalyzed by UV irradiation (Ceresana 2011a, 2012, 2013). Pigments are used as colorants and may also confer additional properties such as UV protection (e.g. titanium dioxide and carbon black) (Ceresana 2011b, Lithner et al. 2011). Biocide examples include halogen, metallic and organosulfur compounds (Ceresana 2012). Fire retardants may be added to reduce flammability (e.g. organic halogen compounds and magnesium hydroxide) (Ceresana 2011c). Various lubricants such as paraffin and other petrochemical waxes and oils may also be added to the polymers or surfaces of machine processing parts during plastics manufacturing.

3.1.1 Phthalate Plasticizers

One of the largest and most controversial groups of plastic additives is plasticizers. Phthalate ester plasticizers were first used commercially in the 1920s (Graham 1973, Guo et al. 2012). Plasticizers are a class of organic compounds added to plastics to improve flexibility and durability, without which a plastic may be too rigid and brittle for its intended use (Latini et al. 2004, Till et al. 1982). Plasticizers work by decreasing the polymer T_g, making the plastic softer. PVC polymers use almost 90% of plasticizers produced worldwide (Cadogan and Howick 2000). The most common types of plasticizers are the phthalate esters listed in (Table 2).

Specific phthalates used in food packaging materials such as plastic wrappers on candies, cooked meats, and cheese (Castle et al. 1988) are diethyl phthalate (DEP), dimethyl phthalate (DMP), diisobutyl phthalate (DiBP), di-n-butyl phthalate (DnBP) and di(2-ethylhexyl) phthalate

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(DEHP) (Fasano et al. 2012, Sendón et al. 2012). The Food and Drug Administration (FDA) restricts food use-approved plasticizers to 30% of the weight of food containers (US FDA 2013a). PVC is predominantly plasticized with DEHP. Due to health concerns and governmental regulatory changes (EU 2011), DEHP use is declining and being replaced with linear phthalates and non-phthalate plasticizers such as polyester (U.S. DHHS 2011). DEHP is classified by the Environmental Protection Agency (EPA) as a class B2 probable human carcinogen, and acts as an endocrine disruptor in the body (Zhou et al. 2011). Human exposure to DEHP is primarily through ingestion, whereas DMP and DEP are through inhalation; DBP and DEP can be absorbed transdermally (Guo et al. 2012).

Unlike some plasticizers, phthalates are not chemically bound to plastic products and therefore can leach into foodstuffs (Zhou et al. 2011). Majority of guidelines are set for drinking water, but not all phthalates used in food packaging are addressed. The EPA limits phthalates according to the Phthalates Action Plan due to their toxicity and evidence of pervasive human and environmental exposure pathways. Leaching into water sources can be toxic to terrestrial and aquatic animals (Russo et al. 2012, U.S. EPA 2012). The most common phthalate is DEHP (CAS 117-81-7), which is regulated under the EPA's Safe Drinking Water Act (SDWA) at a maximum contamination limit of 0.0056 mg/L (U.S. EPA 2017a). The solubility of DEHP in water is low (45 μg/liter), though, DEHP may form colloidal dispersions with higher solubility values (> 285 μg/liter) (IPCS 1992). Phthalates migrate into ethanol more readily than water (Karačonji et al. 2017) because they are miscible with most common organic solvents (IPCS 1992). The migration of phthalates is likely influenced by pH. Soft drinks with a pH of 3 had 5 to 40 times greater migration from plastic to liquid when compared to pH 5 mineral water (Bosnir et al.

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2007). Given wine ethanol content (7 to 14% v/v) and pH (3 to 4) it may be possible for greater migration to occur in wine when compared to water (Amerine et al. 1980), though no data are available to show whether there is greater migration of phthalates into wine compared to water. While few studies have investigated the migration of plastic materials into wine, there have been studies that evaluate food contact materials (FCM) migration into food simulants (Paseiro-Cerrato et al. 2017, US FDA 1977a), fruit juices (de Quirós et al. 2015), mineral water, and soft drinks (Bosnir et al. 2007). After 30 days of exposure in PET bottles, DMP was not detected in mineral water, DMP was abundant in soft drinks, varying with preservatives (sodium benzoate and potassium sorbate). DMP ranged from 18 – 2666 µg/L in soft drinks. However, they do not account for whether the products were contaminated prior to being in bottle, such as through exposure in the bottling line. If the source of contamination was the bottling line, a similar conclusion might be made for the bottling of wine.

Regulatory intake, defined as Tolerable Daily Intake (TDI) is not clearly defined across states, countries, or globally and differs for each type of plasticizer. Ideally, suggested TDI's should account for gender, age, duration of exposure and body mass. TDI set by the EPA for nine phthalates ranges from 0.02 to 0.8 mg/kg-day orally. The European Food Safety Authority's (EFSA) tolerable daily intake (TDI) for phthalates is 0.01 mg/kg-day for DBP, 0.05 mg/kg-day for BBP, 0.05 mg/kg-day for DEHP (Moreira et al. 2013). The US Consumer Product Safety Commission examined target subpopulations (women, infants, toddlers, and children), and for eight phthalates (DEP, DBP, DIBP, BBP, DNOP, DEHP, DINP, and DIDP) found ingestion of food, beverages, and drugs rather than children's toys and personal care products was the greatest source of phthalate exposure (Gennings et al. 2014). Estimated means of phthalate

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exposure ranged from 0.00015 to 0.0308 mg/kg-day. The EPA's reference doses for phthalates include 0.8 mg/kg-day for DEP, 0.1 mg/kg-day for DBP, 0.02 mg/kg-day for DEHP, and 0.2 mg/kg-day for BBP (U.S. EPA 2017b, 2017c, 2017d, 2017e). TDI limitations may not consider the isomeric mixtures of phthalates, with some studies only focusing on a few of the hundred potential isomers with varying physiological impact. Additionally confounding is the fact that few epidemiological studies have been conducted on humans. Though correlations between toxicity data on animal subjects can be made with human health, more work is needed to understand the physiological impacts on human health. The FDA's guidance for packaging, or Food Contact Substances (FCS) indicates a consumption factor (CF), for the fraction of content within a daily diet of a particular additive. The CF of plasticized LLDPE is 0.05 mg/kg under the assumption that migration is occurring in alcoholic beverages with alcohol concentrations ranging from 10 to 15% (v/v) ethanol/water, with no specific regulation applied to wine containers (US FDA 2007a). Despite the lack of global regulatory limits and study limitations, compelling evidence suggests the link between phthalates and negative effects on reproductive, fetal developmental, liver, kidney, heart, lung and hematologic health in humans (DiGangi et al. 2002) illustrating the need for HACCP systems when phthalate-containing plastics are used in food storage products.

3.1.2 Bisphenols

Bisphenols are primary constitutional monomers used in production of epoxy resins and polycarbonates used in food contact materials (FCM) applications (Table 2). Epoxies are used to line canned food containers, processing pipes, and concrete wine tanks, among many other uses (Pivnenko et al. 2015). Epoxy resins are produced through the reaction of epichlorohydrin and

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BPA to form bisphenol A diglycidyl ether (DGEBA or BADGE). Epoxy resins may be further reacted (cured) through catalytic homopolymerization or by forming a copolymer with hardeners or curatives to form thermosetting cross-linked polymers that exhibit strong mechanical properties with high temperature and chemical resistance. Hardeners include phenols, anhydrides, polyfunctional amines, and thiols in order of increasing reactivity.

Polycarbonate polymers are commonly used in water bottles and food storage containers because they are durable with high impact-resistance, temperature resistance, and optical clarity. Since the 1950s, BPA has been used as the monomer in polycarbonate plastic, resulting in global production estimated at 10 billion pounds per year (vom Saal et al. 2012). BPA was approved by the Food and Drug Administration (FDA) for products containing food in the 1960s (Grignard et al. 2012). Polycarbonate is typically produced by the reaction of bisphenol A (BPA) and phosgene $COCl_2$ but may be produced with other bisphenols e.g. bisphenol S (BPS) or bisphenol F (BPF) (Table 2). BPA may be used as an antioxidant for polymer and plasticizer use in PVC production (Grossman 2008). Leaching of BPA occurs when the molecules are hydrolyzed from polycarbonate as temperature increases at high or low pH (Fasano et al. 2012), although BPA is poorly soluble in water (Le et al. 2008). When BPA-containing plastics or epoxy-lined storage containers are scratched or damaged over time, BPA has the capacity to leach into food and beverages (Brede et al. 2003, Brotons et al. 1995, Howdeshell et al. 2003). Wine storage bags are often made of polycarbonate plastic (also called #7) which contain BPA.

BPA has received considerable attention as a suspected toxicant due to its weak estrogenic activity, which is suggested to disrupt endocrine and estrogen signaling, alter human development, and cause breast and prostate cancers, have led to usage restriction (Barrett 2008,

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Grignard et al. 2012, Matsushima et al. 2007, Timms et al. 2005). Nevertheless, migration of BPA into foods from packaging materials occurs at very low concentrations (Ackerman et al. 2010, Noonan et al. 2011). Few data are available on bisphenol migration in wine, though BPA and its curing agent methylenedianiline migrated through epoxy resin vats into model wine in a range of 0 to 30 mg/kg and 0 to 7.6 mg/kg resin, respectively (Larroque et al. 1988). New research would be needed to determine if contemporary tanks exhibit similar migration. BPA from plastic food containers is not expected to be a risk to consumers (Bang et al. 2012). US FDA and EFSA both agree that BPA poses no health risk to any age group under normal dietary exposures consumed, with women of childbearing age and men of comparable ages experiencing an exposure of up to 0.388 μg/kg-day, below the recommended TDI of 4 μg/kg-day. (EFSA 2015, US FDA 2014). No U.S. regulatory agency restricts levels of BPA in food, however twelve states in the US have policies to limit BPA exposure (Safer States 2017). For example, in 2015 California listed BPA on its Proposition 65 list, also known as the Safe Drinking Water and Toxic Enforcement Act of 1986, which prohibits companies and individuals from using chemicals known to the state to cause cancer or reproductive toxicity (Misko 2016, OEHHA 2015). As a result, many manufacturers are developing new formulations of non-BPA containing epoxies and other alternatives.

While many manufacturers have discontinued using BPA and claim to be “BPA-free,” or increasingly use BPA non-intent (BPA-NI) alternatives (BPA-NI means that no BPA was intentionally added), cross-contamination of trace amounts of BPA may be possible during the manufacturing process and contact with material still containing BPA that may be used on shared equipment. Additionally, they may instead be using BPS or BPF that also test positive for

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estrogenic activity (Gander 2016, Molina-Molina et al. 2013). Grignard et al. (2012) used two highly standardized transactivation assays, and found the estrogenic activity of BPA and BPS concentrations to be comparable.

BPA has been found in wine stoppers and wines stored in steel, wood and plastic vats, glass bottles and Tetra Briks (mean concentration 0.58 ng/mL) (Brenn-Struckhofova and Cichna-Markl 2006), in an unspecified brand of synthetic corks (Zapel 2011), and a small sample of beer and soda cans reportedly contained 1.7 to 3.5 μg of BPA per can attributed to the epoxy lining (Müller 2017). Ester bonds linking BPA to polycarbonate and epoxy resins of food storage material are hydrolyzed when exposed to heat and contact with acidic or basic foods, which releases bisphenols into foods (Fasano et al. 2012).

NIAS may also be an issue, especially where new, alternative polymers use is concerned and which despite FDA Guidance documents, may not be fully understood (Paseiro-Cerrato et al. 2017, US FDA 2007b). The FDA regulates food-contact "resinous and polymeric coatings," listing approved precursor materials and setting migration limits of total extractives from the coating to the food (US FDA 1977b).

4 Hazard Analysis Critical Control Points (HACCP)

As plastics use increases in wineries, little is known about the implications of plastic containing products on identified critical control points (CCP) and safety programs. To monitor the safety of food products, including their packaging, HACCP have been utilized by food producers, regulatory authorities, and inspection services (Bovee et al. 1997) and with increasing occurrence, winemaking. For example, the European Union set maximum concentration limits

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for ochratoxin A (OTA), a fungus-derived toxin in wines for all member states and HACCP have been proposed as a method to address that risk (Martínez-Rodríguez and Carrascosa 2009), which may also be applicable to plastic additive contamination. Though HACCP in wineries have not been required under the US Food Safety Modernization Act (FSMA) (Leake 2014), the FSMA requires FDA inspection of wineries since 2018. FSMA will be used to monitor the whole food production chain, so in addition to wineries, custom-crush operations, and mobile bottling operations will be under consideration (Smith 2013). Several control points (CP) and CCP lists and guides are already published in journal articles and through universities and are available for use in wineries. While CP are important, CCPs are crucial for product quality and manufacturing safety. CP and CCP can be used to develop Wine Standards Management Plans (WSMP) in a winery (N.Z. FSA 2017). CP and CCP occur in the vineyard, in transport of fruit from vineyard to winery, and in the winery. CP and CCP for grapes, must, and wine are related to physical, chemical, and microbial hazards and quality parameters such a product appearance, consumer acceptability, flavor, color, and aroma. Good manufacturing practices and vineyard management are key in maintaining CPs and CCPs (Christaki and Tzia 2002). In the United States, wineries must have a permit with the Alcohol and Tobacco Tax and Trade Bureau (TTB) and be registered with the FDA under the Bioterrorism Act of 2002. Wine is considered low in risk of food safety hazards according to the TTB and FDA. However, the FSMA imposes a few additional safety factors, such as enforcing continued registration with government agencies, recalls, product detainment, and import regulation (Smith 2013). One important consideration that is overlooked in CPs and CCPs is plastic usage. Plastic is either not mentioned or is not considered a biological, chemical, or physical hazard (N.Z. FSA 2017). However, based on

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research from other foods and beverages and the lack of published data on wines, there may be a need to re-evaluate and research potential hazards of plastics in winemaking.

Regardless of the scientific or regulatory consensus about health risks, they may be irrelevant to market forces from negative public perception and the assumption that plastics are a hazard in a wine industry that increasingly uses plastics. For example, part of a "Chemical Fallout" article series by the *Milwaukee Journal Sentinel* outlined negative effects of BPA, to much praise (Rust et al. 2007). Brewer and Ley (2011) examined public response to this controversy across news media and determined that despite mixed opinions by the scientific community as to the confirmed link between risks and consumption, most people who had been exposed to even a small amount of information about BPA, were concerned and favored a ban on its usage. As outlined in Brewer and Ley, three main actors are involved in perception of BPA, which could also be expanded to other compounds like phthalates: science, business, and government. Irrespective of health effects, or lack thereof, even for benign compounds, negative marketing or public perception may have grievous consequences. While implementation of HACCP practices might be helpful in mitigating health risks and satisfying regulatory requirements, we suggest that HACCP approaches might also be beneficial if applied to other areas such as hazard analysis of critical control points to wine production processes that may influence potential public perception and marketing in addition to effects on wine flavor and quality.

4.1 Plastics and Plastic Additives in Wine

Plasticizers tend to be lipophilic, with limited solubility in aqueous alcohol solutions. However, many foodstuffs used to make alcohol (i.e. grapes, apples, grains, etc.) have some

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lipophilic substances in their skins that may accumulate plasticizers through contact (Buglass 2010). From the time they are picked, fruit used to make wine may contact plastics that potentially contain plastic additives. For example, fresh picked plums transported in plastic bags had detectable levels of DEP, DBP, and DIBP (Jurica et al. 2016). After entry into the winery, fruit and wine ingredients may be exposed to pumps, tubing, transport containers, pneumatic press material, additives such as flavorings, and finally storage, bulk shipping containers and consumer packaging materials which can all contain or be contaminated with plasticizers or bisphenols and possibly contribute cumulative increases of these chemicals to the wine (Buglass 2010, Del Carlo et al. 2008, Sendón et al. 2012). Even though the fruit, must, and wine residence time with any one of these plastics containing materials may be short, the cumulative exposure potential for leaching is unknown and a worthy area for additional research.

Regarding alcoholic beverages, plasticizers have been found in Chinese *baijiu*, a white spirit usually distilled from sorghum or other grains. The Jiungui liquor company found liquor samples containing 1.04 mg/kg DBP, which is higher than the 0.3 mg/kg standard set by the Ministry of Health in June 2011 (China.org 2012, Zhu 2012). Large-scale tests of China's liquor have shown almost all alcohol products contain an average level of 0.537 mg/kg of plasticizers (Yinan 2012). DBP and DIBP were found in more than 94% of food samples, but were significantly higher in wine and beer compared to other beverages (Guo et al. 2012). Other grain-neutral spirits and vodka have been found to contain phthalate plasticizers including DBP, DOP, and DEHP, however they were considered biologically insignificant (Leibowitz et al. 1995). In March 2013, three brands of French cognac were prohibited from entering China's market because they reportedly contained excessive levels of plasticizers (Global Times 2013).

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In terms of winemaking, plastics are used in the manufacture, transport, and storage of wine (Table 3). Just as in liquor manufacturing, various stages in winemaking may involve plastic products that contain leachable plasticizers and other additives. Wine may come into contact with extractible plasticizers such as DBP and DEHP, which are the most common phthalate contaminants in wine (Buglass 2010). In some cases, plastics are used because they offer advantages to traditional packaging. For example, PET bottles and Bag-in-Box containers weigh less than a glass wine bottle of the same volume, so shipping costs are lower, storage is easier, and they do not shatter (Scheer and Moss 2012). Several of these features are considered to be environmentally-friendly. Packaging used for boxed wine has some advantages because it supposedly prevents oxidation for longer once opened when compared to glass bottles and can keep wine fresh for up to six weeks after opening (Ghidossi et al. 2012). A drawback of using plastic is the potential for plastic materials in contact with wine to scalp volatile flavors from wine, or wine may absorb undesirable aromas from plastic (Peyches-Bach et al. 2012). Examples of plastic materials that may contact wine are stoppers, including those used to seal partially consumed bottles of wine, as well as aluminum cans and concrete fermenters, which were commonly lined with BPA based epoxy, although BPA-NI alternatives are available (Gander 2016, Scheer and Moss 2012, Sheftel 2000, Teichgraeber 2005). Wine in can consumer acceptance and sales share are increasing and is an area of great interest and concern for manufacturers (Johnston and Velikova 2016, O'Donnell 2016).

Alternatives to natural bark cork closures are also a concern for plasticizer contamination. Alternative closure use such as synthetic corks and screw caps has increased due to the rate of cork taint, estimated at 3-5% of bottled wine, caused by 2,4,6-trichloroanisole (TCA) that

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imparts musty, wet cardboard aromas (Butzke and Suprenant 1998, Jennings 2012). Synthetic closures comprise an estimated 19% of the closure market, with metal screw caps making up 11% of the market of approximately 20 billion wine bottles per year (Steeman 2010). A greater range of plasticizers occur in plastic closures compared to other plasticized plastic materials used in winemaking (Buglass 2010, Sendón et al. 2012). SARANEX™, used in both screw cap liners and synthetic closures, is a barrier film consisting of layers of SARAN™ resin (polyvinylidene chloride, PVDC) and thermoplastic polymer resins (Dow 2013). SARAN™ resin contains PVC, a source of plasticizer contamination. Plasticizers found in PVC-based films include DEHA and phthalates such as DBP and DEHP (Groth and Silbergeld 1998). LDPE has been used as a replacement for PVC in SARAN™ (SC Johnson), however it provides a poor oxygen barrier and can scalp flavors from foods (Smith and Hui 2004). In addition, use of artificial closures, plastic liners in screw caps, and other plastic closures may expose wine to plastic leachates that alter organoleptic properties in the wine as with other foods and beverages (Wagner and Oehlmann 2009).

In the environment, apart from a few fungal species and bacterial isolates, it is difficult for plastics to be broken down by microbes due to their absence of enzymes necessary to convert biochemically novel compounds such as plastic molecules into intermediates (Yoshida et al. 2016). Nevertheless, certain microbes are integral in the process of wine making and though microbes that can break down plastics have not been identified in wine, more work is needed to determine if microorganisms in wine promote the breakdown of plastics involved in wine processing, storage, and packaging and that may have human health or wine quality consequences. Also important is the identification of wine microorganisms potentially capable of

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degrading bio-based plastics. Microbes can degrade organic and inorganic compounds such as lignin, starch, cellulose, and hemicellulose, therefore storage in bio-based containers should be examined.

4.2 Exposure Considerations

Concentrations consumed by humans are an effect of many variables: storage conditions of the beverage influences amount of leaching, chemical properties of the beverage, packaging type, intake, gender, size of the person, and age all interplay. Moderate drinkers of alcoholic beverages are described as individuals who consume four drinks for men and three drinks for women in a single day, and a maximum of 14 drinks for men and seven drinks for women per week (Nordqvist 2018). In comparison to the estimated BPA consumed based on the National Health and Nutrition Examination Surveys, total adult intake ranged from 30 – 70 ng/kg-day between 2005 and 2010 and was mainly attributed to canned food consumption (Lorber et al. 2015). Estimated exposure of seven phthalate monoesters as measured by urinary metabolites ranged from 1.7 to 110 mg/kg-day for the 95th percentile of the population. Phthalates were based on total exposure, including consumption, absorption through skin, and inhalation (David 2000). For an adult man with an average weight of 89.6 kg (Gill 2018), given a standard glass of wine is approximately 148 mL (NIAAA) and he drinks the average 4 glasses a day with a potential BPA concentration of 0.58 ng/L, his exposure to BPA from this consumption factors to 343.36 ng, or 3.8 ng/kg-day, a tenth of the lowest total adult daily intake. Though many factors affect how much plastic additives are in a wine (e.g. storage conditions, manufacturing process), the average amount of BPA in wine from the papers reported in this review was 0.58 ng/mL (Brenn-Struckhofova and Cichna-Markl 2006, Lambert and Larroque 1997). Different phthalates

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are examined and reported in each study, but in general Carrillo et al. (2008) found total phthalates in wines ranged from 0.0027 to 0.015 mg/L. For the same man consuming the greatest of the range cited by Carrillo et al. (2008) in four drinks, he would consume 0.0089 mg, much lower than the 95th percentile of consumption according to David (2000). Just as alcohol affects each age, gender, and size of person differently, each person's ultimate exposure to additives may differ based on the same variables. Finally, it begs the question: how much of these plastic additives does a person have to consume before they experience health problems, if at all?

5 Analytical Methods

Due to the widespread use of bisphenols and phthalates, disposable laboratory plasticware such as pipette tips may be contaminated with or contain these additives, which can compromise laboratory experiments (Del Carlo et al. 2008). Additives such as oleamide and biocides have been found to leach from laboratory PP disposable plasticware, which affects protein function in biological research (McDonald et al. 2008). Laboratories can seek manufacturers that disclose information on additives used in the manufacture of plastic products for laboratory use, as well as leachable reaction components used in the manufacture of plastics. Regardless, researchers may still need to confirm the absence of effects due to additive contamination or to account for them in their assay methods and results.

Because bisphenols and phthalates are mostly found at trace levels (nanograms per milliliter or less), all analytical quantification methods in both solid and in liquid samples must start with concentration of the analytes prior to chromatographic analysis. Examples of concentration methods include liquid–liquid extraction (LLE) (Del Carlo et al. 2008), solid-

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phase extraction (SPE) (Del Carlo et al. 2008, Russo et al. 2012), multi-walled carbon nanotuble sorbents (Li et al. 2013), solid-phase micro-extraction (SPME) (Carrillo et al. 2008, 2007) and stir-bar sorptive extraction (SBSE) (Pfannkoch and Whitecavage 2002).

5.1 Determination of Phthalates

Several analytical methods have been utilized to determine phthalate concentration in various materials used in making and storing wine, however, detection of phthalates is challenging because of their ubiquitous nature in the laboratory environment (Del Carlo et al. 2008). Eliminating background traces of phthalates is important in order to report accurate limits of detection (Bradley et al. 2013). Phthalate analysis is based mainly on gas chromatography – flame ionization detection (GC-FID) and gas chromatography – mass spectrometry (GC-MS), however gas chromatography/ion trap – mass spectrometry (GC/IT-MS), high performance liquid chromatography – ultra violet visible detection (HPLC-UV) and liquid chromatography – mass spectrometry (LC-MS) are also utilized (Cao 2010, Russo et al. 2012). Using these analytical methods, DBP, BBP, and DOP have been found in wines, including DEHP at levels exceeding the EPA limit (0.0056 mg/L in water), particularly from wines with synthetic or agglomerated cork stoppers (Carrillo et al. 2008, Sendón et al. 2012).

5.1.1 GC-FID

GC-FID has primarily been used to examine other foods, but not alcoholic beverages. However, GC/FID was used to establish the effectiveness of single-drop microextraction (Batlle and Nerín 2004). Three aqueous food simulants containing trace phthalates were analyzed, including 15% (v/v) ethanol/water, 3% (w/v) acetic acid/water, and distilled water. In

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comparison to SPME, recovery was effective, ranging from 85 to 115% for most compounds. It was determined limits of detection levels were below those recommended by the EPA.

5.1.2 GC-MS

Solid-phase extraction-gas chromatography-mass spectrometry (SPE-GC-MS) was used to investigate the presence of six phthalate esters from commercial, private producers, and pilot red and white wines at low trace levels (Del Carlo et al. 2008). It was determined that all wine samples were contaminated with phthalates. The limit of detection (LOD) for the analysis was 18 ug/L, and the limit of quantitation (LOQ) was 29 ug/L. In comparison to current TDI amounts, which are specified based on mg/kg-d, the amount consumed, as well as factors such as gender an age play a role as to whether an individual is exposed to safe amounts.

Plastic wine tops held at “extreme conditions” (EC) of incubation in an oven at 40 °C for 10 days or in ultrasonic bath for 15 min and exposed to 15% (v/v) ethanol/water (Fasano et al. 2012). Eight compounds were examined with SPE-GC-MS, four of which were phthalates, and it was determined that all plastic wine tops receiving EC treatment were contaminated by all phthalates, and in the ultrasonic treatment were contaminated with 2 to 3 phthalates.

Carrillo et al. (2007) determined that the best fibers for examining phthalate esters in wine were polyacrylate (PA), carbowax-divinylbenzene (CW-DVB), and polydimethylsiloxane-divinylbenzene (PDMS-DVB). Further work utilized isotopically-labelled phthalate internal standards with HS-SPME-GC/MS and determined total phthalates in the wines analyzed ranged between 0.0027 to 0.015 mg/L (Carrillo et al. 2008).

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5.1.3 Electron Spin Resonance

Migration of DOXYL and TEMPO-phthalate from agglomerated champagne cork stoppers was examined using electron spin resonance (ESR) (Six and Feigenbaum 2003, Six et al. 2002). Paramagnetic probes were incorporated in the adhesive and cork during processing. To incorporate the probes, cork granules were sealed with probes in a hermetic poll box in an oven held at 70 °C for 2 hr for TEMPO-phthalate. Ten grams of cork granules were molded with 1.75 g adhesive plus 0.3 g Vaseline. Corks were immersed in alcoholic simulant of wine (12% v/v ethanol/water at pH 3) for 10 days at 40 °C. ESR spectra of slices of cork indicated simulant wine penetrated the whole structure of the finished cork.

5.1.4 GC/IT-MS

Pre-concentration is necessary for developing sensitive analytical methods of trace compounds such as phthalates. Russo et al. (2012) explored the pre-concentration step to optimize SPE-GC/IT-MS by using Carbograph 1 sorbent to improve recovery of phthalate by 78 to 105%. The method was both sensitive and reproducible in the red and white wines analyzed.

Six and Feigenbaum (2003) analyzed champagne corks for potential migrants of concern, toluene diisocyanate (TDI) and methylene bisphenylisocyanate (MDI) in adhesives, lubricants, and surface treatments. Analysis was conducted spectroscopically and chromatographically. The composition was determined and verified by infrared spectroscopy, proton nuclear magnetic resonance spectroscopy (1H-NMR) and GC-MS. Interestingly, they found the presence of other solutes in wine, such as sugars, can decrease the migration of additives from cork. Simulant wine can overestimate this migration, providing an extra margin of safety when compared to levels found in actual wine. DEHP was the only migrant detected from the corks, with 90% of

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extraction occurring within 1 day in all tests. DEHP was specifically monitored in further tests, and found transfer from cork to simulant wine to be 50 µg/L. Existing regulations on silicone elastomers in France was used as the reference value for safe levels of migration from champagne corks, therefore migrated DEHP was less than the legal reference of 3 mg/L.

5.1.5 Liquid Chromatography

Agglomerated corks are made from natural cork granules and adhesives which contain esters such as phthalates and adipates. Sendón et al. (2012) utilized HPLC-MS/MS to examine the presence of phthalates in 21 agglomerated cork stoppers as well as their potential migration into 12% (v/v) ethanol/water, although, no corks yielded quantifiable levels of phthalate migration. Yano et al. (2002) also used HPLC to determine the presence of phthalates in Korean and Japanese retail beverages, including alcoholic beverages such as the Japanese distilled beverage *sho-chu*, beer, rice punch, red wine, and white wine. Levels of DBP in Japanese red wines were among the highest sampled at 0.275 µg/g, nearly 100% greater than in Korean wine. LC-GC/MS has been found to be an efficient method to examine phthalate residues in grain neutral spirits and vodka (Leibowitz et al. 1995). Six reported phthalates were quantitated in 50 samples, although detected levels were insignificant compared to the suggested threshold for long-term exposure, 15 mg/L, with concentrations as low as 20 µg/L. Most recently (Barciela-Alonso et al. 2017), developed a SPE-LC-MS method to determine 4 phthalates in water stored in plastic bottles and white and rosé wine stored in Tetra Brik packages. All phthalates were found in water, though DEP and DBP were the only phthalates recovered in both type of wine.

5.2 Determination of Bisphenols

Many solvent extraction methods as well as SPE are used to isolate BPA from samples,

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followed by analysis such as LC, GC, and immunochemical methods (Ballesteros-Gómez et al. 2009).

5.2.1 SPE with GC/MS

Fasano et al. (2012) conducted migration tests to examine levels in 11 types of common food packaging materials, including plastic wine tops made with elastomers and foams (ethylene, propylene, urethane, silicones or their copolymers with different additives). The migration test utilized liquid food contact materials to simulate different types of foods: distilled water for aqueous foods with pH above 4.5, 3% acetic acid in distilled water for acidic aqueous foods with pH below 4.5, 15% ethanol for alcoholic foods, and oil for fatty foods. Migration test conditions were 40 °C for 10 days, which was considered "extreme conditions" (Fasano et al. 2012). Analytes were concentrated with SPE and quantified with GC/MS. Plastic wine bottle tops showed the highest level of migration for one of the two alkylphenols and three of the four phthalates when compared to the ten other sources of food packaging materials which included items such as baby product food packaging, canned food, food bags, and glass jar caps. Of the three phthalates found, levels were 25 to 75 times greater than the lowest amount found in the other packaging materials. BPA was not recovered in wine bottle tops. The authors concluded risk due to exposure is primarily associated with potentially negative impact on health. In cases where wine is stored on its side, there may be potential migration issues from wine bottle tops.

5.2.2 HPLC

Lambert and Larroque (1997) utilized HPLC with fluorescence detection in wine and mineral water, which can be contaminated through exposure to epoxy resins lining wine storage containers, water towers, and drinking water pipes. Detection limits ranged from 5 to 2.5 μg/L in

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red and white wine and 0.25 to 0.70 μg/L in mineral water. Sol-gel immunoaffinity, HPLC, and fluorescence detection were used to examine BPA contamination in wine exposed to vats (steel, wood and plastic), glass bottles and Tetra Brik type carton packages (Brenn-Struckhofova and Cichna-Markl 2006). Plastic wine stoppers were immersed in 11% ethanol and detectable levels of BPA were leached from the stoppers. Wine samples consisted of a total of 59 wines (46 white, 13 red). In 13 of 59 wine samples, the BPA concentration was below the LOQ (0.2 ng/mL). Mean BPA for the wine samples was 0.58 ng/mL, below previously published BPA levels derived from migration experiments using wine simulants.

6 Alternative Plastics and Plasticizers: Bio-based Options

Alternative plastics and plasticizers from natural compounds that have no negative effect on human health and little to no impact on economic viability and product quality would likely be of interest to some producers in the wine industry, particularly those that desire to market more natural or organic winemaking approaches. Polysaccharides, proteins, lipids, and microbes are potential sources for natural plasticizers and bio-based polymers. Bio-based compounds (Table 4) may decrease the need for petroleum-based plastics and plasticizers as well as reduce their toxicological and environmental impacts. Alternative plasticizers are an option to reduce toxicity due to plasticizer leachate in plastic products (Lowell Center for Sustainable Production 2011). Biopolymer films and natural-based plasticizers are less toxic, leachable, and are biodegradable compared to phthalate plasticizers. However, biopolymers tend to have reduced mechanical properties and performance. Biopolymers currently only share 5 to 10% of the market and cost more than their non-biopolymer counterparts (Lowell Center for Sustainable

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Production 2011, Vieira et al. 2011). With BPA-free and phthalate-free labels on many products, the public is aware these substances are problematic, bio-based alternatives may improve consumer confidence and perception.

Though many petroleum-based plastics contain leachable additives that potentially cause health problems, many food contact approved plastic resins such as specific formulations of PE currently offer useful functional properties and do not contain phthalates, bisphenols or other potentially harmful substances (Dow 2014). Linear low-density polyethylene (LLDPE), high-density polyethylene (HDPE), medium-density polyethylene (MDPE), ultra-high-molecular-weight polyethylene (UHMWPE), and cross-linked polyethylene (PEX) that is made with HDPE, are generally considered to be plasticizer free. PE products available to the wine industry include wine bottle closures and wine tanks. Phthalate alternatives to DEHP and DINP-containing plastic corks and cap liners may include low volatility plasticizers such as Palatinol 10-P (Goth 2007), to those that are oil and plasticizer free (Elastocon 2015). Barrier films have also been applied to natural cork stoppers as a way to prevent TCA induced cork taint (Easton 2010). It is conceivable that barrier films may be developed that could prevent plasticizer leaching. It is worth mentioning that even polyethylene products can contain bioactive ingredients added for various purposes (e.g. UV stabilization), but not necessarily in all cases or products.

7 Conclusion

Though HACCP are not required for wineries in the United States, additional benefits of developing a program include addressing health, wine quality, and public perception concerns in using plastics during winemaking. Though manufacturers may state that a finished product may

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not contain certain additives such as bisphenols or phthalates, other additives may be present. Furthermore, consumers and wine producers alike may not be aware that some plastics, such as PE, could contain additives synthesized from animal extracts such as fatty acids produced by the hydrolysis of animal fats (tallow) (Dow 2014). Therefore, labelling of these plastics may be warranted to satisfy situations in which individuals desire to comply with various religious dietary laws (e.g. kosher) or for personal reasons (e.g. vegan).

Concerned consumers and wine producers who use plastic might want to contact manufacturers for details about the plastic resins and additives used, notwithstanding nondisclosure of proprietary information. Manufacturer resin codes and supporting Regulatory Data Sheet, or independent lab tests, may be needed for definitive details. However, results from independent lab tests can be compromised due to ubiquitous use of plastic products in the laboratory (McDonald et al. 2008).

Naturally, consumers and wine producers concerned about leachate contamination can seek products that use traditional materials, such as wood, clay, stainless steel, glass and cork to avoid potential sources of petroleum or animal-based chemicals. Additional action plans for winemakers specifically include developing CPs and CCPs for their winery which would include identifying key points in which fruit, must, wine and its ingredients are in contact with plastic-containing substances. For quality concerns, if plastic-containing items are used, conditions that encourage potential leaching and scalping should be avoided. If plastic is used in the final product, labelling to note any specific precautions taken (e.g. bisphenol-free) could be used to improve public perception. HACCP approaches that identify and prevent potential hazards from

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occurring in winemaking and its marketing, may lead to safer products and better consumer confidence, marketability, and ultimately wine sales.

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Figure 1 Classification of Plastics (modified from Klein 2011).

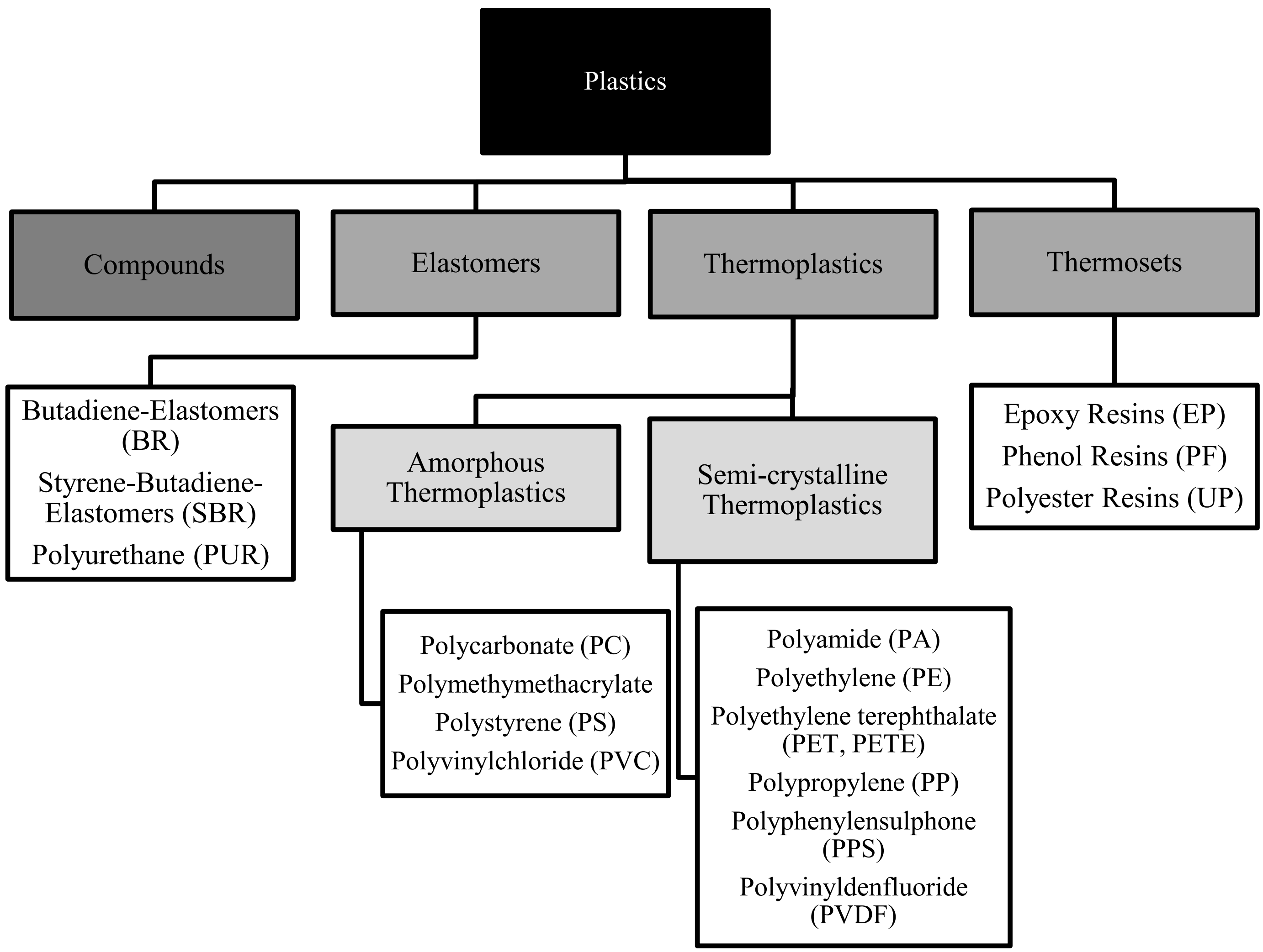

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Table 1 Resin Identification Code (D20 Committee 2010).

Resin Code[1]	**Polymer Name**	**Uses**
1 PETE	Polyethylene terephthalate	Drink/water and soda bottles, juice boxes, liquor bottles, food trays, condiment jars, plastic film, microwavable packaging.
2 HDPE	High-density polyethylene	Milk, juice, water jugs, detergent bottles.
3 V	Polyvinyl chloride	Bottles for cooking oil, salad dressing, mouthwash, and liquor. Plastic wrap, “blister packs”, plastic pipes.
4 LDPE	Low-density polyethylene	Produce, frozen food, and bread bags, trash bags, squeezable bottles.
5 PP	Polypropylene	Condiment and medicine bottles, drinking straws, yogurt containers, margarine tubs.
6 PS	Polystyrene	Egg cartons, disposable plastic table ware (cups, plates, cutlery), packaging foam/"peanuts", “clamshell packaging”, food carry-out containers.
7 Other	Other (includes polycarbonate)	Injection molded drinking bottles, glasses and food containers.

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Table 2 Common Phthalate Esters and Bisphenols.

Chemical class	Compound	Abbr.	CAS number	Formula	SIM	MW	Limits
Phthalate	Dimethyl phthalate	DMP	131-11-3	$C_{10}H_{10}O_4$	163 [a]	194	
	Diethyl phthalate	DEP	84-66-2	$C_{12}H_{14}O_4$	149-177 [a]	222	
	n-Dibutyl phthalate	DBP	84-74-2	$C_{16}H_{22}O_4$	149-205 [a]	278	
	Butyl cyclohexyl phthalate	BcEP	84-64-0	$C_{18}H_{24}O_4$	149-223 [a]	304	
	Butyl benzyl phthalate	BBP	85-68-7	$C_{19}H_{20}O_4$	149-206 [a]	312	
	Bis-(2-ethylhexyl) phthalate	DEHP	117-81-7	$C_{24}H_{38}O_4$	149-167 [a]	390	0.006 mg/L [b,c]
Bisphenol	Bisphenol A	BPA	80-05-7	$C_{15}H_{16}O_2$	(213.10, 228.10, 119.05, 216.10, 234.10, 121.05) [d]	228	0.05 mg/kg-day [e,f]
	Bisphenol F	BPF	620-92-8	$C_{13}H_{12}O_2$		200	
	Bisphenol S	BPS	80-09-1	$C_{12}H_{10}O_4S$		250	

[a]Russo et al. 2012, [b]U.S. E.P.A. 2009, [c]U.S. F.D.A. 2013b, [d]Macek and Burkhardt 2011, [e]U.S. E.P.A. 1993, [f]EU 2008.

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Table 3 Potential sources of plastic exposure in the production of wine.

Activity	Type	Item	Material	Potential Additive
Transport	Container	Flexible transport tank	PE, PP, polyester, PVC	DBP, DEP, DEHP, DMP [a], BPA
		Intermediate Bulk Containers (IBC's), drums, totes, and bins	PE, HDEP, PP	DBP, DEP, DEHP, DMP [a]
Equipment	Crush	Press bladder membrane	Nylon, rubber, polyester, PVC, silicone	?
		Conveyor belts		
	Pumps & hoses	Seals, impellers, bearings, lines, hoses	PE, polyacetal, PVC	DBP, DEP, DEHP, DMP [a], BPA
	Tanks/ containers/ barrels	Poly tanks	PE	DEHA, DBP, DEHP [b]
		Fiberglass tanks	Fiberglass-epoxy/PVC	BPA
		Concrete	May be lined with epoxy [c] which may contain PVC [d,e,f]	
Ingredients	Fining	Resins	?	?
Filtration	Media & housings	Pads and resins		
		Reverse osmosis membranes	PP, Nylon, polyethersulfone, silicone elastomer [g,h]	DBS, DEP, DIBP
		Ultrafiltration media		
Packaging	Bottle stoppers	Synthetic "cork"	#4 LDPE or #7 mixed plastics	DBP, DEP, DEHP, DMP [a,c], DEHA [b]
		Agglomerate corks	PUR	DEHP [i]
		Natural cork coatings	Paraffin, waxes, silicon, other polymer coatings [j]	?
		Screw cap liners	#4 LDPE, PVC, PVDC	DBP, DEP, DEHA; DEHP, DMP [a,b,k]
	Containers	Bag-in-Box	PE, #7 mixed plastics/PC	DBP, DEP, DEHP, DMP [a], BPA [d]
		PET bottles	PET	DBP, DEP, DEHP, DMP [a]
		Aluminum can lining	Epoxy/PVC, #7 Mixed plastics [d] /PC [l]	BPA

[a]Buglass 2010, [b]Groth and Silbergeld 1998, [c]Zapel 2011, [d]Scheer and Moss 2012, [e]Sheftel 2000. [f]Teichgraeber 2005, [g]Pall Corp. 2014, [h]3M 2011, [i]Six and Feigenbaum 2003, [j]Amerine and Joslyn 1970, [k]Nara et al. 2009, [l]Natural Resources Defense Council 2011.

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Table 4 Bio-based plastics and plasticizers.

Material	Name	Abbr.	Sources
Plastic	Cellulose acetate [a]	CA	Cotton fibers and wood
Plastic	Cellulose acetate butyrate [a]	CAB	Cotton fibers and wood
Plastic	Cellulose acetate propionate [a]	CAP	Cotton fibers and wood
Plastic	Cellulose nano-composites [b]		Obtained by chemical treatments and steam explosion of cellulose materials
Plastic	Corn zein [a]		Corn
Plastic	Lignin [a]		Plants and wood
Plastic	Natural fiber reinforced composites [a]		Kenaf, hemp, ramie, flax, sisal, jute, pineapple leaf
Plastic	Polyhydroxyalkanoate [c]	PHA	Whey, lignocellulosic raw materials, molasses, glycerol, fats, wastewater
Plastic	Polylactic Acid [a]	PLA	Corn, sugar beets, sugar cane, wheat, sweet potatoes, rice
Plastic	Polysaccharide nanocomposites [d]		Heparin, chitosan, cellulose, hyaluronan, starch, alginate, pectin, guar, starch/chitosan, chitosan/heparin, chitosan/hyalurona, hyalurona/heparin, cellulose and chitin whiskers, platelet-like starch
Plastic	Poly(trimethylene terephthalate) [e]	PTT	Sugar from corn with terephthalic acid (PTA) or dimethyl terephthalate (DMT) derived from petroleum [f]
Plastic	Soy protein [a]		Soybeans
Plastic	Starch derived plastics: Thermoplastic starch [a]	TPS	Corn, potato, rice, wheat, tapioca
Plastic	Urethanes: Polyol [a]		Soy oil/soybean, castor oil, rapeseed, sunflower, linseed
Plasticizer	Acetyl tributyl citrate	ATBC	Citric acid derivative
Plasticizer	Acetyl triethyl citrate	ATEC	Citric acid derivative
Plasticizer	Acetyl trihexyl citrate	ATHC	Citric acid derivative
Plasticizer	Acetyl trioctyl citrate	ATOC	Citric acid derivative
Plasticizer	Acetylated monoglycerides		Edible fats and triacetin
Plasticizer	Butylated hydroxytoluene	BHT	Phenol derivative
Plasticizer	Butyl Stearate		Stearic acid and butyl alcohol
Plasticizer	Butyryl trihexyl citrate	BTHC	Citric acid derivative
Plasticizer	Epoxized soybean oil	ESBO	Soybeans
Plasticizer	p-tert-Butyl phenyl salicylate		4-tertiary-butylphenol and salicylic acid [g]
Plasticizer	Tributyl citrate	TBC	Citric acid derivative
Plasticizer	Triethyl citrate	TEC	Citric acid derivative
Plasticizer	Trihexyl citrate	THC	Citric acid derivative
Plasticizer	Trioctyl citrate	TOC	Citric acid derivative

[a]Wool and Sun 2005, [b]Chirayil et al. 2014, [c]Du et al. 2012, [d]Zheng et al. 2015, [e]DuPont 2007, [f]Álvarez-Chávez et al. 2012, [g]Sommerfield and Stoesser 1953.