ABSTRACT

CADWALLADER, DYLAN. Sensory and Instrumental Analysis of Flavors due to Scalping and Migration from Packaging in Fluid Milk (Under the direction of Dr. MaryAnne Drake).

Few studies have addressed the effects of packaging material in the absence of light on contributions to fluid milk flavor. The objective of this study was to compare the sensory and chemical properties of fluid milk packaged in paperboard cartons, low-density polyethylene (LDPE), high-density polyethylene (HDPE), polyethylene terephthalate (PET), linear low-density polyethylene (LLDPE), and glass. Pasteurized (high temperature short time, 77 °C for 25 sec) skim and whole milk were filled (280 mL ±10 mL) into paperboard cartons, LDPE, HDPE, PET, LLDPE, and glass (control). Milks were stored at 4˚C in the dark and sampled at days 0, 5, 10, and 15. Descriptive analysis (DA) was applied to document sensory profiles at each timepoint, and volatile compounds were extracted and identified by Solid Phase Micro-Extraction (SPME) with gas chromatography mass spectrometry (GC-MS) and gas chromatography-olfactometry (GC-O). Tetrad tests with consumers were conducted at day 10. Both skim and whole milks packaged in cartons had noticeable paperboard flavor by day 5 and higher levels of hexanal than skim and whole milks in other package types at day 5 ($p < 0.05$). Skim milks packaged in paperboard cartons and LLDPE had distinct refrigerator/stale flavor compared to milks in the other package types concurrently with increased levels of refrigerator/package-related compounds including styrene, acetophenone and 2-ethyl-1-hexanol ($p < 0.05$). Milks packaged in glass, PET and HDPE were not distinguished by consumers at day 10 post processing ($p > 0.05$). Package type influences fluid milk flavor, and these effects are greater in skim milk compared to whole milk. Paperboard cartons do not preserve milk freshness as well as PET, HDPE or glass due to flavor migration and scalping. Glass remains an ideal

barrier to preserve fluid milk flavor, but in the absence of light, HDPE and PET provide additional benefits while also maintaining fluid milk flavor.

Sensory and Instrumental Analysis of Flavors due to Scalping and Migration from Packaging in Fluid Milk

by
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BIOGRAPHY

Dylan Cadwallader was born on November 17th, 1992 to Karen and Keith Cadwallader. He was born in Baton Rouge, Louisiana but grew up and spent most of his school years in Champaign, Illinois with his two brother Myles and Ryan. Dylan attended high school in Champaign, Illinois, at Centennial High School. After high school Dylan pursued a B.S. degree in Food Science at the University of Illinois at Urbana-Champaign. The last 2 years of undergrad Dylan worked as an undergraduate worker for Dr. John Erdman as a lab and research assistant which cemented his love for research in food science. After obtaining his degree in Food Science Dylan left Champaign and moved to Columbus, Indiana to work as a Quality Assurance/Microbiologist for Rose Acres Farm where he learned how to manage a lab and be productive in a fast paced production environment. Dylan felt limited by his environment in Columbus and was compelled to further his education and obtain an M.S. degree. During his search he discovered Dr. Maryanne Drake's lab, which immediately felt like home. Dylan plans to pursue his PhD with Dr. Maryanne Drake.

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CHAPTER 1: LITERATURE REVIEW. FLAVOR SCALPING/FLAVOR ADSORPTION AND MIGRATION FROM PACKAGING IN DAIRY PRODUCTS

INTRODUCTION

The purpose of food packaging is to preserve the quality of the food and the acceptance of the consumer (Dainelli et al., 2008). The most common reason for a consumer to discard food or a beverage is from an unacceptable flavor (Reineccius, 1991; Mottram, 1998). Off-flavors are commonly thought of as a result of microbial spoilage but they are not the only offender (Reineccius, 1991; Risch, 2000). Sanitation practices and advances in thermal processing technology have provided a platform for off-flavors generated from deficiencies in packaging (Arvanitoyannis, 2004; Azzara, 1992). Formation of off-flavors as a result of packaging and the surrounding environment have become an extensively studied subject (Sajilata et al., 2007). The majority of consumer complaints to companies are unacceptable flavor complaints (Whitfield and Shaw, 1985). These complaints are usually the sign of a major problem with the product because it is estimated that for every negative complaint made to a manufacturer another 20 to 200 consumers have also had a similar experience (Hubbard, 2012).

BRIEF HISTORY OF DAIRY PRODUCTS AND PACKAGING

Milk has been a dietary staple for families across the world for over 10000 years (Bollongino, 2012). Milk has mostly been a fresh commodity directly from the cow to the table, simply consumed within a few days or made into longer lasting products such as butter and cheese (Burnett, 1999). Milk consumers before the turn of the 20th century were people who owned their own livestock and could get fresh milk daily (Tunick, 2009). Consumers who did not have their own livestock would normally get it brought to them in large earthen jars or metal

jugs (Lockhart, 1999), but the problem was that raw milk has a short shelf and is susceptible to spoilage (Tunick, 2009). It is only in the past 100 years that milk has become a regular consumer product due to the advent of pasteurization to extend shelf life and increase safety. Milk was first individually packaged into glass bottles to distribute to customers. Glass bottles were the beginning of conventional packaging of milk for consumers. It wasn't until the 1930's that paperboard or cardboard packaging became commonplace (Harper and Hall, 1976). The first forms of high-density polyethylene (HDPE) packaging emerged in the mid to late 1960's but only gained popularity as production processes became cost effective (Richmond and Stine, 1982).

Today, milk is packaged in many different package formats from paperboard cartons to different types of plastic bottles with the most common being HDPE and polyethylene terephthalate (PET). HDPE is the most common package because of its malleability, barrier properties and ease of transport (low weight) (Crosby, 1981). Paperboard and PET are also commonly used; paperboard is the cheapest. PET is similar to HDPE but is less expensive and a less effective barrier than HDPE (Brody, 2009). Other dairy products utilize other packaging formats. Butter is conventionally packaged in wax paper and covered in a paperboard box, but because of its longer shelf life, flavor migration occurs over time from its packaging and environment (Lozano et. al., 2007). Sour cream is packaged in polypropylene or polystyrene plastic tubs with a seal adhered to the lip of the tub protecting it from the surrounding atmosphere and a plastic lid on top (Costello, 2009). For all dairy products and especially milk, packaging plays a role in freshness and flavor stability.

FLUID MILK PROCESSING AND FLAVOR

Processing parameters plays a key role on the flavor of fluid milk. The two most important aspects of milk flavor are the quality of the raw milk and the heat treatment used for pasteurization. The quality of raw milk is normally measured by the microbial load and the somatic cell count (SCC) of the milk (Barbano, 2017). Microbial contents in raw milk are almost always due to sanitary conditions of the milking parlor and/or farm bulk tank. Somatic cells in milk are mainly leukocytes which become abundant as a response to pathogenic bacteria in the mammary gland of the cow (Ma et al., 2000). Pathogenic bacteria can causes mastitis, an inflammation of the udder. Poor microbial quality and/or high SCC can result in a host of fluid milk off flavors as well as decreased shelf life even following pasteurization.

The heat treatment step in processing fluid milk is also important for finished product quality. Several common heat treatments (pasteurization) are used in the dairy industry (from lowest to highest heat load): batch or vat, high temperature short time (HTST), ultra-high temperature (UHT), and sterilization (Holsinger et al., 1997). Vat or batch pasteurization is uncommon in today's dairy industry but is commonly used by small or hobby farms (Goff, 2019; Rankin, 2017). The FDA defines vat or batch pasteurization as milk that has been heat treated for 30 minutes at 140^{o}F (60^{o}C) (FDA, 2019). HTST pasteurization is the most commonly used heat treatment for fluid milk, particularly in North America (Goff, 2019; Rankin, 2017). The FDA defines HTST milk as heat treatment for 15 seconds at 161^{o}F (72^{o}C) (FDA, 2019). UHT milk is used for milk intended for a long shelf life and can be shelf stable depending on processes used when filling packages (Goff, 2019; Rankin, 2017). UHT milk is thermally processed at or above 280^{o}F (138^{o}C) for at least 2 seconds (FDA, 2019). When UHT milk is not aseptically packaged it is also called ultrapasteurized (UP) or extended shelf life (ESL) milk and it must be refrigerated.

Sterilization of milk can also be applied by retort processing in cans for shelf stable milk and the time/temperature used is 275ºF (135ºC) for 5 seconds (Goff, 2019).

The flavor effects of each heat treatment vary. Vat or batch pasteurization has the least effect on the flavor of fluid milk, demonstrated to have lower cooked and heated flavors when compared to other methods due to the lower heat load (Alvarez, 2009). HTST pasteurization provides a mild but higher cooked flavor to fluid milk than vat pasteurization (Alvarez, 2009; Potter and Hotchkiss, 1999). HTST is the most commonly used pasteurization method in the U.S. and has demonstrated through numerous studies to be widely accepted by U.S. consumers (Holsinger et al., 1997; Schiano et al., 2017). Ultrapasteurized (UP) milk flavor is also distinct from HTST fluid milk (Jo et al., 2018). UP milk has more cooked, caramelized, and sulfurous flavors than HTST milk (Potter and Hotchkiss, 1999; Lee et al., 2017; Jo et al., 2018). There are also flavor differences between UP milk pasteurized by direct steam injection ultrapasteurization (DSI-UP) and indirect steam ultrapasteurization (IND-UP) (Lee et al., 2017; Jo et al., 2018). The packaging of milk (and other dairy products) following processing and pasteurization also has a profound impact on the flavor.

MECHANISMS/CHEMISTRY OF BEVERAGE/FOOD INTERACTION WITH PACKAGING

Permeation is the diffusion of molecules absorbing and desorbing across the package into their respective internal and external environments (van Willige, 2002). Permeation in food and beverages is the transfer or transportation of surrounding environmental substances such as gasses (carbon dioxide, oxygen), volatile compounds (aroma active, contaminants), and/or microbes into the food and back into the environment (van Willige, 2002). Permeation or the permeability rate is dependent on the permeability of the package, thermal and mechanical

properties, and the properties of the surrounding environment, such as ambient vapor or condensation, which will eventually break down the package (Siracusa, 2012). This transfer of molecules from the environment can have adverse effects including oxidation, microbial growth and off-flavors in the product leading to degradation of the product and decreased shelf life (Nielsen and Jägerstad, 1994). A good example of permeation through packaging can be shown through the beneficial and adverse effects of the use of cork when bottling wine. The steady transfer of oxygen through the cork controls the aging process of wine, and eliminates undesirable compounds in wine. However, cork taint can be a problem because the cork is susceptible to microbial growth that can taint the wine through the same mechanism (Pereira, 2000). Migration and adsorption (scalping) of the package have increased effects on the process of permeation between the internal environment of the package and its surrounding environment (van Willige, 2002). There are two types of permeation that occur with food and packaging: migration and scalping.

Migration is when molecules desorb or absorb into the package or product from the environment or package and usually results in the formation of food off-flavors (Nielsen and Jägerstad, 1994). Migration in food and beverages is often described as flavor and aroma compounds of the environment and/or package materials moving and transferring to the food (Sajilata et al., 2007; Fu et al., 2003). It is important to clarify the differences between migration and permeation, permeation is exchange of gasses between the product and the surrounding environment, and this can involve flavor migration although permeation doesn't always involve flavor migration from the package (Siracusa, 2012; Fu et al., 2003). It is also possible through permeation that off-flavors from the outside environment migrate through the package to the product (migration) (Arvanitoyannis, 2004; Fu et al., 2003). The presence of stale off flavors in

butter stored at 4^{o}C were attributed to migration of styrene and toluene from the refrigeration environment through the package and into the butter (Lozano et al., 2007) Migration of volatile compounds from the package is a flavor defect in many products and is widely studied (Reineccius, 1991). Hazardous or toxic compound migration from plastics and dyes in food packaging may or may not cause off-flavors, but is considered a serious migration problem (Miltz and Rosen-Doody, 1985; Sajilata et al., 2007).

Dyes or printing inks are generally used with packaging, either as dyes for polymers of plastic packaging to protect from oxidation or on the outside of the package to convey information about the product (Arvanitoyannis, 2004; van Willige, 2002). It is a common problem for compounds in inks to migrate into the food product when the packaging degrades (van Willige, 2002; Lord, 2003; Sajilata et al., 2007). The FDA, in response, has made allowable limits for food packaging related compounds for foods, providing exemptions to food dyes and inks in foods as long as they are below their allowable thresholds, essentially giving them the same designation as a food additive (21CFR170.39: Food Additive Safety, 2019; Lord, 2003). A food additive is described by the World Health Organization (2020) as a substance that is added to food to maintain or improve its safety, freshness, taste, texture, or appearance. Inks can migrate from the package and accumulate in foods above allowable limits and create unwanted off-flavors and possibly cause safety concerns. To circumvent this problem, packaging is commonly selected based on its ability to limit this interaction. Different barriers and liners are also used with packaging to avoid migration as well as to prevent the product degrading the packaging and weakening the structure of the container (Riley, 2012).

The subject of scalping is a widely studied area of flavor and off-flavor research in food packaging. Scalping is defined as the loss of volatile aromatic flavors from the food that are

absorbed by the package (Sajilata, 2007). Most systems that illustrate scalping or off-flavor perception involve studies that measure the loss of flavor over time by solubility and diffusion factors (Landois-Garza and Hotchkiss, 1988). If a flavor compound is not soluble enough to enter the packaging, it will not be lost in the package matrix. If a flavor compound is soluble enough to diffuse into the package, the rate of loss of flavor depends on the rate of diffusion of the flavor compound into the polymer or packaging (Halek and Luttman, 1991; van Willige, 2002). The rate of diffusion of flavor compounds in a polymer is affected by a multitude of different factors. These include the pH, humidity, temperature, polarity, concentration, and molecular weight (Halek and Luttman, 1991; Landois-Garza and Hotchkiss, 1988). However, molecular weight and polarity of the flavor compounds are the primary determinations of permeability reduction (Reineccius, 2005).

Selection of packaging for a certain product involves shelf life studies and tracking migration of possible package related flavor migration over time through sensory and instrumental analysis (Lickly et al., 1995; Tawfik and Huyghebaert, 1998). During these studies, products are packaged in various packaging and over time, the products are sampled for sensory changes as well as for off-flavor defects and contamination. Many of these studies have been done with citrus, specifically orange juice, because citrus is particularly prone to flavor scalping problems with plastic polymers (Mannheim et al., 1987; Berlinet et al., 2008; Chamberlain and Kirwan, 2012; Peter, 2019). There is limited research on flavor absorption and scalping in respect to milk and other dairy products (Sajilata, 2007; Mestdagh et al., 2005).

Determination of off-flavors and scalping in food can be done using an assortment of analytical techniques that measure volatile flavor compounds and sensory analysis. Sensory analysis is used to confirm sensory perception, and instrumental techniques are applied to

measure volatile compound changes. Commonly, GC-MS (gas chromatography mass spectrometry) via headspace, dynamic headspace, or solid-phase micro-extraction (SPME), GC-O sniffing, SAFE (solvent-assisted flavor evaporation), and other techniques such as supercritical fluid extraction (SFE) are used (Yang and Peppard 1994; Zhang and Pawliszyn 1993; Thompson et al., 1996; Brattoli et al., 2013).

THE DEVELOPMENT OF OFF-FLAVOR FROM ENVIRONMENT TO PACKAGING

Off-flavors can be caused by degradation of the food, which includes enzymatic or non-enzymatic reactions, protein degradation, light oxidation, microbial growth and/or autoxidation of fat (Reineccius, 1991). Products can also be tainted or contaminated during production. Product taints are offensive odors or flavors passed on from external sources to the product (Kilcast, 1996). Tainting can involve residual chemicals left on the package during the packages production as well as contaminated water used in production of the product (Reineccius, 1991).

There are a multitude of ways for a product to develop an off-flavor taint by airborne and waterborne means (Reineccius, 1991). Off-flavors due to contaminated water can also indicate microbiological contamination (Dainelli et al., 2008; Bedient et al., 1999). There are many different types of water-borne microorganisms that could produce off-flavors but the most common is from algae (Ingram, 2004). Water can also be contaminated by piping and contaminated infrastructure. Off-flavors can also be created from plastic piping breaking down and diffusing into the water supply. Plastic piping is more permeable than metal piping so soil contamination around the piping can contribute significant changes to water flavor over time as the piping is contaminated (Tracy and Leonard, 1980; Goldenberg and Matheson, 1975). An uncommon but possible waterborne contamination could be from pollution (usually petrochemicals) next to the water supply or in the supply that a plant draws from (Goldenberg

and Matheson, 1975). Waterborne off-flavor taints occur because water is used in almost all aspects of manufacturing. A common example of an airborne environmental factor could be a factory renovating or painting near a production line and off-flavor taints from paint or other coating agents affecting the nearby product.

COMMON PACKAGING MATERIALS: FOOD AND BEVERAGES

The most used packaging types for food are plastics, cardboard/paperboard, metal, and glass (Paine and Paine, 1992). All of these package materials have their advantages and disadvantages ranging from differences in pricing, difficulty in manufacturing, and usability for different foods (Paine and Paine, 1992). Metal and glass are relatively inert and normally have very little interaction with most food as well as having excellent ability to protect from outside environmental factors (Marsh and Bugusu, 2007; Nielsen and Jaegerstad, 1994). The downside is that metal is significantly more expensive as a raw material and more expensive to manufacture (Nielsen and Jaegerstad, 1994). Glass is cheaper to manufacture than metal but is more expensive than plastic alternatives. Clear glass provides no light protection which is a detriment to dairy products due to light oxidation (Mestdagh et al., 2005). This issue can be remedied by pigmented glass but consumers do not prefer dark packaging (Krishna et al., 2017). Glass is also prone to breakage and is heavy and thus difficult to transport. Plastic packaging has become the most popular form of food packaging. It is relatively easy to manufacture and less expensive than glass and metal. As a lightweight material, plastic is easy to transport and can be made in various thickness and densities, based on the properties needed for the weight of the product (Paine and Paine, 1992). There are also many different types of plastics, which can also be used in combination with differing properties like strength, price, and ease of manufacture to make a suitable package for the product (Riley, 2012). Cardboard or paperboard is commonly used in

tandem with plastics because it is manufactured cheaply and an added layer of thin polyethylene as a liner can help with limiting absorption of product into the surrounding packaging (Riley, 2012).

Plastic Packaging

Plastic packaging has grown exponentially in the last few decades with improvements in production of new highly versatile products (Geyer et al., 2017). Developments in technology of durability and ease of use have made it the most versatile of all package alternatives for food products (Geyer et al., 2017; Gerlowski, 1989; Salame, 1986). There are many different types of plastics used in the food and beverage industry today. The most commonly used plastic is polyethylene (PE) which comes in three molecular arrangements: low-density polyethylene (LDPE), high-density polyethylene (HDPE), and linear low-density polyethylene (LLDPE) (Bhunia et al., 2013; Piringer and Baner, 2000). Other popular plastics used in the food industry are polypropylene (PP), polyethylene terephtalate (PET), polystyrene (PS), polyamide (PA) and ethylene vinyl alcohol (EVOH) (Bhunia et al., 2013; Risch, 2000).

Polyethylene is a densely linked network of ethylene (Zhong et al., 2017; Wang et al., 2004; Drumheller et al., 1995). Polyethylene is formed by making crystallized structures by polymerizing ethylene through chemical, thermal and mechanical processes (Zhong et al., 2017; Wang et al., 2004; Drumheller et al., 1995). The more impacted or dense the structural network of ethylene (i.e. higher crystallinity), the stronger and more rigid the final product will be (Zhong et al., 2017; Wang et al., 2004; Drumheller et al., 1995).

Polyethylene comes in a variety of different grades, all varying by molecular density and structure. Polyethylene is great for packaging applications because of its heat sealability, structural/temperature stability, high elasticity, and exothermic resistance as well as having very

good moisture resistance (Crosby 1981; Robertson 2012). Low-density polyethylene plastics are frequently found as bottles or as films for coatings on other containers such as paperboard/cardboard or aluminum cans. LDPE can act as a shield between the product and outer package, as it has an exceptional resistance, acids, alcohol/solvents, and bases and can be blended with other plastics to have mild heat resistance. (Nielsen and Jagerstad, 1992; Robertson 2012). Linear low-density polyethylene (LLDPE) is similar to LDPE in its physical properties though it is manufactured thinner and more malleable. LLDPE is used for plastic bags and food wrap that needs to be thinner than LDPE allows, while still maintaining rigidity and strength (Robertson 2012; Piringer and Baner, 2000). High-density polyethylene (HDPE) is harder and has greater density than LDPE. It also has barrier properties that LDPE does not have; greater cold resistance and more opacity, which makes it less susceptible to light transmission (van Aardt et al., 2001; Robertson 2012; Piringer and Baner, 2000). HDPE is generally used for gallon milk jugs because of its higher weight tolerance than LDPE and its resistance to light transmission (van Aardt et al., 2001). It is also used for films or plastic bags, such as heavy duty trash bags, and for heavier products such as meat because of its strength and resistance to high fat content (Crosby, 1981; Paine and Paine, 1992; Piringer and Baner, 2000; Robertson, 2012).

Polyethylene terephthalate (PET) is produced by polymerizing ethylene glycol with terephthalic acid (TPA), which creates a plastic that is interwoven similarly to polyester fibers that have high tensile strength (Kuczenski and Geyer, 2010; Duarte et al., 2004). Depending on the thermal and mechanical processes used when producing PET, its physical structure can range widely from polyester fabric threads used for clothing to clear and opaque bottles used in bottling beverages (Kuczenski and Geyer, 2010; Duarte et al., 2004). PET is lightweight and is known for its use in plastic soda bottles and ultra-pasteurized milk bottles. PET is used in

carbonated beverages that contain alcohol and oils because it is excellent for use in products that have a need for a package that has a great gas barrier and can prevent aromas from permeating in from surrounding environment (Risch, 2000; Marsh and Bugusu, 2007). PET can also be manufactured in colorless or white/opaque bottles so it is suitable for products that have light oxidation problems such as milk, though it is a more expensive plastic compared to HDPE to manufacture (Marsh and Bugusu, 2007).

Polypropylene (PP), like polyethylene, is made of a densely linked network but instead of ethylene it is made with the monomer propylene (Geyer et al., 2017; Wang et al., 2004; Drumheller et al., 1995). PP is formed by polymerizing propylene into a crystalline structure like polyethylene but with more rigidity and a higher degree of crystallinity (Geyer et al., 2017; Wang et al., 2004; Drumheller et al., 1995). Polypropylene (PP) is harder than polyethylene and it has a much better heat tolerance. This makes it a lot more usable in high heat applications such as hot filling for aseptic and sterilization purposes (Ansari and Datta, 2003; Risch 2000; van Willige, 2002).

The issue of flavor scalping can become a problem for paperboard and other packaging materials that have plastic coatings or liners, such as aluminum cans, and glass jar enclosures. This is because plastics can scalp flavors from the product (Reineccius, 2005; Fu et al., 2003). The larger the surface area of contact of the plastic to the food product, the greater the potential quantity of flavor absorption from the food (Sajilata, 2007; Hotchkiss, 1988).

Scalping in plastics is rarely consistent throughout a product and heavily depends on the interaction between the product and the polymer of plastic used (Sajilata, 2007). The severity of off-flavors or scalping of the product is increased by the potential off-flavor solubility (greater solubility, more migration) as well as the shelf-life of the product (Sajilata, 2007; Landois-Garza

and Hotchkiss 1988). Polyethylene draws large quantities of non-polar flavor volatile compounds due to it being extremely lipophilic (Brody et al., 2008). A longer shelf life product will inevitably have larger flavor scalping/migration problems as most packaging eventually will produce off-flavors from scalping or contaminate a product from migration (Sajilata, 2007; Brody et al., 2008). The compounds d-limonene and other terpenes, such as styrene and pinene are scalped in grapefruit and orange juice immediately in a paperboard polyethylene lined carton (Braddock and Goodrich, 2003). A similar study was done with fluid milk that showed that polyethylene and polypropylene were associated with a decrease in desirable milk flavors over time when compared to a control (Simon and Hansen, 2001). A long shelf life study (7 months) with table wine in PET bottles showed a decrease in concentration of esters when compared to wine filled in conventional glass bottles (Mentana et al., 2009)

Paperboard Cartons

Paperboard and cardboard packaging are the most common food-packaging material in the world (Marsh and Bugusu, 2007). Paperboard and cardboard packaging are used for a multitude of different products. Paperboard accounts for the largest share of global packaging sales at 39% as of 2003 with 70% of all consumer packaging used for food and beverages (WPO, 2013). Paperboard is generally produced from mechanically pressing and then drying pulped plant fibrous materials (Chamberlain and Kirwan, 2012; Ruth and Hanington, 1997). Plant material is formed into pulp through a chemical or thermal step to break down the lignin followed by a mechanical separation of the fibers to be reshaped for a specific purpose (Chamberlain and Kirwan, 2012; Ruth and Hanington, 1997).

Paperboard cartons for milk as we know today were first introduced to the public in 1929 (Richmond and Stine, 1982). Milk producers welcomed the change, because glass bottles had to

be moved carefully to avoid breakage and had high transportation costs because of their considerable weight (Richmond and Stine, 1982; Lockhart, 1999). Glass bottles also have a high susceptibility to light oxidation, which was a limiting factor to shelf life due to its effect on fluid milk flavor (Mestdagh et al., 2005; Johnson et al., 2015). These issues made the transition to use of paperboard cartons relatively easy because paperboard is not as susceptible to light oxidation as a clear glass milk bottle (Moyssiadi et al., 2009; Bradley et al., 1980). Milk cartons are also cheaper to make and easier to distribute because of their malleability. Cartons are more difficult to break than glass and have resistance or give when exposed to shock over a period during transportation (Marsh and Bugusu, 2007).

Paperboard comes in many different forms. The majority of paperboard cartons are made from solid bleached sulfate (SBS) paperboard (Duncan and Hannah, 2012). SBS paperboard is made from bleached virgin pulp (80%) and then the paperboard is usually colored with a synthetic dye (Duncan and Hannah, 2012). Coated unbleached kraft (CUK) paperboard is normally categorized by its strength and durability and is made from 80% unbleached virgin pulp (Duncan and Hannah, 2012). It can be used for frozen foods and soft drink beverages, even with a substandard printing surface, but is normally used for laundry detergents and other cleaning products due to its stronger rigidity and strength (Duncan and Hannah, 2012). Paperboard is separated into grades based on strength and its intended application (Riley, 2012). Riley (2012) classified paperboard into 4 different grades; solid bleached board (SBB) or SBS which is bleached virgin paperboard, solid unbleached board (SUB) or CUK which is unbleached virgin paperboard, folding boxboard (FBB), which is multiple layers of individual paperboard and is normally used for more acidic beverages or in some cases, hot filled, and white lined chipboard (WLC) which is paperboard made from multi-layers of uncoated and coated recycled

paperboard. There is specific nomenclature for degree of strength and application within each of these categories (Riley, 2012).

Paperboard cartons also have problems. Milk cartons are prone to flavor migration and permeation problems between the product and carton as well as the surrounding environment. These issues become much more pronounced the longer the product has been in the carton (Simon and Hansen, 2001). Milk will also eventually absorb (scalp) the flavors of the paperboard over time, losing both aroma and flavor intensity of the original product (van Willige, 2002). Flavors due to packaging become the limiting factor of shelf life (Simon and Hansen, 2001). In the past, paperboard was coated with wax to reduce the permeability of the carton but today paperboard is manufactured with thin layers of polyethylene that keep migration between product and beverage to a minimum (Chamberlain and Kirwan, 2012; Risch, 2000). Usually the polyethylene layer is so thin that paper packaging still has issues with off-flavors that are given off by the inks used in printing as well as the solvents used to coat the packaging (Sajilata et al., 2007; Marsh and Bugusu, 2007) . Resins that are used in production of wax and plastic liners for packaging can also impart off-flavors to the product inside (Chamberlain and Kirwan, 2012; Heydanek, 1979; Marsh and Bugusu, 2007). Paperboard is regulated by the FDA which has a specific list of all chemicals, dyes and resins that can be added to paperboard (21CFR176.180). Allowable limits of assumed carcinogenic or toxic substances that are used in the manufacturing of paperboard cartons and printing inks are also controlled by the FDA (21CFR176.180).

Paperboard becomes structurally weaker when liquids are encountered. The paperboard breaks down and the cellulose molecules/fibers that hold the paperboard together expand (Robertson, 2012). This breakdown also facilitates migration between the environment and the product (Sajilata et al., 2007; Marsh and Bugusu, 2007). To remedy this problem, multiple layers

of polyethylene can be used to reduce the scalping of the product to outer packaging (Marsh and Bugusu, 2007). The layers of polyethylene reduce moisture absorption and flavor scalping into the packaging and oxygen permeation from the outside environment (Marsh and Bugusu, 2007).

Cardboard or paperboard flavors in milk packaged in paperboard cartons can be perceived by the average consumer (Frandsen et al., 2007). The migration of these cardboard off-flavors are limited by the polyethylene layers and resins that are layered over the paperboard to protect the beverage from those off-flavors and to protect the paperboard from breaking down (Mezouari et al., 2015). Off-flavor migration problems will increase as container size decreases as a surface area to volume ratio (Leong et al., 1992; Sajilata et al., 2007). Cardboard flavor in dairy foods is caused by a group of different aroma active aldehydes and phenols mainly comprised of hexanal, octanal, nonanal, E-2-nonenal, E,E-2,4-decadienal, and alkyl- and methoxy- phenols (Czerny and Buettner, 2009; Ziegleder, 2001; Leong et al., 1992). Whitson et al. (2010) demonstrated that cardboard flavor in whey protein was composed of a similar combination of lipid oxidation compounds (a combination of pentanal, hexanal, heptanal, nonanal, and 1-octen-3-one) and dimethyl trisulfide (DMTS).

Flavor migration from paperboard carton to fluid milk takes place from low molecular weight compounds from the paperboard package diffusing into the product (Gnanasekharan et al., 1997). **Table 1.1** demonstrates that unwanted flavor volatiles from paperboard cartons and their plastic liners will diffuse into milk over the shelf life of the product. Off flavors will become more pronounced as the paperboard barrier weakens and saturates (Moshonas, 1989). Hazardous or toxic migration of compounds from plastics and dyes from packaging can also be a serious migration problem (Miltz and Rosen-Doody, 1985). This item could be significant if the

paperboard used for packaging degrades faster than the shelf life of the product, rendering it unsafe to drink.

Examples of studies done on migration between paperboard and milk are limited, but several studies have been done on orange juice and paperboard. A study was conducted with orange juice stored in laminated carton packages which documented that aroma compound absorption was directly linked to the solubility of the absorbing layer of the package (van Willige et al., 2003).

In dairy products, specifically fluid milk, packaging flavor due to cardboard cartons develops by day 1 of storage (24 hours after processing) and is largely thought to be from the soluble compounds from the package and not just from the high temperatures used during heat sealing (Leong et al., 1992). Leong et al. (1992) evaluated parameters that covered a wide variety of fat contents in milk (skim 0.05%, low fat 2.0%, whole 3.25%) and used water as a control for each packaging type evaluated (polyethylene coated paperboard cartons, high density polyethylene (HDPE)). Leong et al. (1992) also assessed each package type at 3 different volumes (half-pint, quart, and half-gallon) and found that half-pint cartons had more intense packaging flavor due to the increase in contact area of milk to the package surface. The study was limited by the sensory terms and method used. Leong et al. (1992) used a pairwise ranking test (Meilgaard et al., 1987) which paired milk samples packaged in paperboard cartons vs. glass (skim, low-fat, whole) with the only comparison being made was which samples had more "packaging flavor". Leong et al. (1992) also did not conduct volatile instrumental analysis to delve deeper into causes of off-flavors.

Karatapanis et al. (2006) reported that by day 5 the sensory profile of fluid milk packaged in paperboard cartons (3.5% fat, 100mL) had a noticeable off-flavor associated with the

paperboard container with panelists describing a “slight stale” taste. The sensory profile correlated with increases in hexanal, pentanal, heptanal and dimethyl disulfide (Karatapanis et al., 2006). Karatapanis et al. (2006) also evaluated whole milk in 6 different package types (clear glass bottle, clear PET bottle, pigmented PET bottle, 3-layer pigmented coextruded HDPE bottle, monolayer pigmented HDPE bottle, and coated paperboard carton). Karatapanis et al. (2006) evaluated milk cartons over a short shelf life (6 days). Sensory methods were also limited similarly to Leong et al. (1992) with a limited lexicon of off-flavor descriptors evaluated. During shelf life, each sample was exposed to light for the entirety of shelf life, which may have had a confounding effect on the effects the package exclusively has on the aroma and flavor of fluid milk (Karatapanis et al., 2006).

Aluminum Cans and Steel Containers (Metal)

Metal food containers are generally made from non-corrosive and malleable metals such as steel or aluminum (Page et al., 2011). Metal is used in a variety of applications making it the most versatile of all food packaging. It is an excellent barrier against physical environmental factors. Depending on the package composition, metal can also be a good gas and aroma barrier (Page et al., 2011). It can be formed into thin shapes while still maintaining strength and durability (Marsh and Bugusu, 2007). Generally all metal food and beverage containers have a plastic liner made of polyethylene to prevent microscopic particles from contaminating the product (Page et al., 2011; Marsh and Bugusu, 2007).

Aluminum cans are the most common form of metal packaging for both carbonated and non-carbonated drinks (Page et al., 2011). Aluminum is a lightweight metal commonly found in the form of can or foil. It is resistant to most forms of corrosion and is an effective barrier against outside environmental factors such as gas, temperature and moisture (Page et al., 2011). It is also

flexible and has good resilience as well as being easy to form into seamless containers (Page et al., 2011; Davis, 1993). Though it is also difficult to shape into anything other than seamless containers or uniform foils (Page et al., 2011; Davis, 1993). The main drawback of aluminum packaging is that it has a high initial cost of production, which can be remedied by advocating recycling (Page et al., 2011).

Tinplated steel or tin-free steel is what is commonly used for packaging food and beverages (Page et al., 2011). Tin-plated steel is steel that has a small percentage of tin which is used as a liner because it is resistant to acidic environments, such as the canning of fruits and vegetables, and prevents corrosion (Page et al., 2011, Oldring and Nehring, 2007). Tin-free steel is commonly used for products that don't require the added barrier of tin (Page et al., 2011). Tin-plated and tin-free steel are both commonly lined with a plastic liner such as polyethylene (Page et al., 2011). Steel is frequently used for canning because of its excellent barrier properties and high resistance to heat. Steel also has high mechanical strength, which makes it resistant to most environments or shipping conditions (Marsh and Bugusu, 2007).

Metal, just like plastics and paperboard, can also impart off-flavors into products. Typically any type of can used to store milk, such as powdered formula, is lined with plastic to prevent lipid oxidation caused by direct contact with milk and a metallic surface (Page et al., 2011; Brown, 1940). Liners are also required because of the possibility of contamination of microscopic pieces of metal in the food as well as the possibility of low pH foods dissolving the can (Page et al., 2011; Cabezndo et al., 1986). Products can be tainted by flavors from the liners that are used when manufacturing cans. As such, selection of the plastic liner is vital (Cabezndo et al., 1986).

Glass

Glass is one of the oldest food packaging materials with examples being found that are over 5000 years old (Grayhurst and Girling. 2011). Glass is odorless and chemically inert with almost no interaction with most food products, though this is likely not the case as glass containers use some type or plastic or rubber seal (Brody et al., 2008; Grayhurst and Girling. 2011). Glass can sustain high processing temperatures making it viable for products to be sterilized within the container. Its disadvantages are that it is very breakable from sudden changes in temperature and repeated physical shock. It is also very heavy compared to other package options (Marsh and Bugusu, 2007). Fluid milk packaged in glass shows significantly less environmental or packaging related tainting compounds and a "fresher taste" by sensory analysis for a similar shelf life when compared to milk packaged in HDPE or PET containers or paperboard cartons (Karatapanis et al., 2006).

CONCLUSIONS/SUMMARY

Food packaging is critical to protect the flavor and aroma of dairy products since off-flavors can be produced from many different ways in the surrounding environment. Food packaging can mitigate off-flavors, but can also be a source of off-flavors from migration and/or scalping. Limited published work has addressed the role of packaging in the absence of light exposure on the flavor of fluid milk. The objective of this thesis was to address the role of packaging on the flavor of fluid milk.

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Table 1.1: Volatile compounds of whole milk (60 mL) stored in coated paperboard cartons (taken from Karatapanis et al., 2006)

Volatile compound profile (μg mL^{-1}) of whole pasteurized milk packaged in paperboard carton as a function of storage time at 4 °C

Volatile compound	t_r[a] (min)	Qulaity factor[b]	Days of storage				Trend[c]
			1	3	5	7	
Ethanol[d]	6.53	72	0.59[e]	0.79	0.45	3.98	↑↑
Acetone	7.52	86	128.61	132.97	134.51	121.34	
Dimethyl sulphide	8.72	97	1.60	1.42	1.16	13.57	↑↑
Methyl acetate	9.04	70	0.53	0.49	0.42	1.69	↑
Dichloro-methane	9.25	97	—	—	18.27	21.14	
Carbon disulfide	9.66	83	0.61	1.81	2.20	2.81	
2-Methyl-propanol	10.48	70	0.73	0.81	0.65	1.16	
3-Methyl-pentane	11.91	91	0.94	0.51	0.67	0.71	
2-Butanone	12.63	87	20.90	20.27	20.00	15.68	
Ethyl acetate	13.80	86	0.46	0.55	0.74	44.06	↑↑
Chloroform	14.13	96	2.50	3.23	7.56	6.38	
Propanoic acid ethyl ester	14.87	86	6.46	10.51	11.24	14.81	↑
3-Methyl-butanal	16.30	97	—	—	—	2.08	
3-Methyl-2-butanone	16.59	72	0.57	0.56	0.46	0.84	
Benzene	16.97	91	4.31	4.11	3.46	5.03	
2-Pentanone	18.19	72	2.53	2.08	1.69	2.64	
2,2,4-Trimethyl-pentane	18.41	83	5.55	17.14	37.75	17.57	↑↑↓↓
Pentanal	18.78	80	1.80	2.31	3.12	2.61	
Propanoic acid methyl ester	19.76	72	—	—	0.13	1.84	
Butyric acid methyl ester	20.27	97	4.24	3.09	3.06	6.25	
3-Methyl-1-butanol	20.86	78	—	—	—	2.96	
4-Methyl-2-pentanone (i.s.)	21.25	91	25	25	25	25	i.s.
Dimethyl disulphide	22.59	90	—	—	0.14	0.23	
Toluene	23.01	94	1.66	2.59	5.34	4.57	
Hexanal	24.32	90	0.60	0.61	1.12	—	
Butyric acid ethyl ester	24.35	97	—	—	—	10.74	
Ethyl benzene	27.67	94	2.08	1.08	1.21	1.87	
1,4-Dimethyl benzene	28.00	97	7.00	4.15	4.01	6.04	
2-Heptanone	28.52	87	—	—	—	0.54	
1,3-Dimethyl benzene	29.12	97	3.28	3.38	1.97	2.55	
Hexanoic acid methyl ester	29.87	90	0.41	0.19	0.14	1.44	
a-Pinene	30.95	97	0.70	0.60	0.67	1.38	
Hexanoic acid ethyl ester	32.61	97	—	—	0.12	3.80	
1,3,5-Trimethyl benzene	33.04	93	—	—	—	0.32	
Delta.3carene	33.65	97	0.38	0.32	0.41	0.76	
Limonene	34.19	98	—	—	0.09	0.52	
Octanoic acid methyl ester	36.40	75	—	—	—	0.22	

↑ = slight increase, ↓ = slight decrease, ↑↑ = large increase, ↓↓ = large decrease, i.s. = internal standard.

[a]Retention time.

[b]Quality factor = % matching of the experimental mass spectra against those found in the Wiley database.

[c]Trends are designated as follows:

[d]Tentatively identified on the basis of the Wiley275 library (J. Wiley & Sons Ltd., West Sussex, England).

[e]Values are the mean of three determinations ($n = 3$).

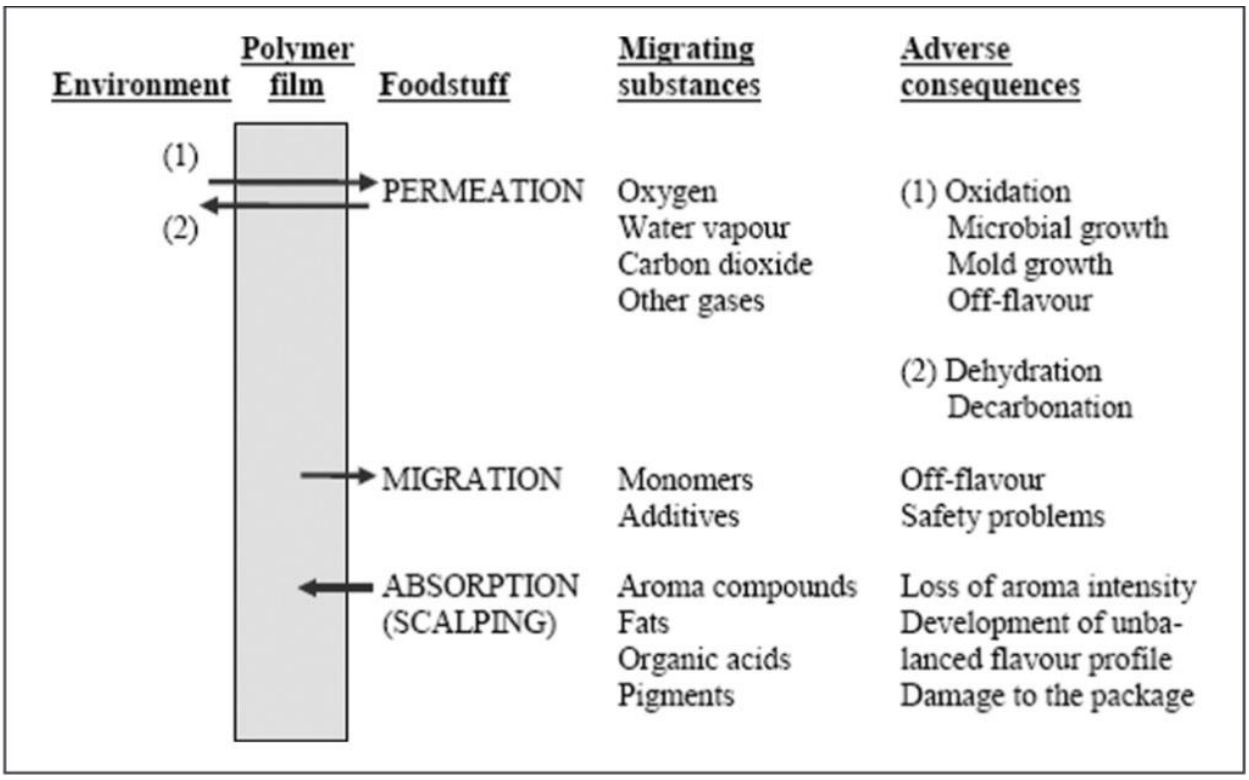

Figure 1.1: Interactions between product and packaging (taken from Nielsen and Jägerstad, 1994)]

CHAPTER 2: THE ROLE OF PACKAGING ON THE FLAVOR OF FLUID MILK

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ABSTRACT

Few studies have addressed the effects of packaging material in the absence of light on contributions to fluid milk flavor. The objective of this study was to compare the sensory and chemical properties of fluid milk packaged in paperboard cartons, low-density polyethylene (LDPE), high-density polyethylene (HDPE), polyethylene terephthalate (PET), linear low-density polyethylene (LLDPE), and glass. Pasteurized (high temperature short time, 77 °C for 25 sec) skim and whole milk were filled (280 mL ±10 mL) into paperboard cartons, LDPE, HDPE, PET, LLDPE, and glass (control). Milks were stored at 4˚C in the dark and sampled at days 0, 5, 10, and 15. Descriptive analysis (DA) was applied to document sensory profiles at each timepoint, and volatile compounds were extracted and identified by Solid Phase Micro-Extraction (SPME) with gas chromatography mass spectrometry (GC-MS) and gas chromatography-olfactometry (GC-O). Tetrad tests with consumers were conducted at day 10. Both skim and whole milks packaged in cartons had noticeable paperboard flavor by day 5 and higher levels of hexanal than skim and whole milks in other package types at day 5 ($p<0.05$). Skim milks packaged in paperboard cartons and LLDPE had distinct refrigerator/stale flavor compared to milks in the other package types concurrently with increased levels of refrigerator/package-related compounds including styrene, acetophenone and 2-ethyl-1-hexanol ($p<0.05$). Milks packaged in glass, PET and HDPE were not distinguished by consumers at day 10 post processing ($p>0.05$). Package type influences fluid milk flavor, and these effects are greater in skim milk compared to whole milk. Paperboard cartons do not preserve milk freshness as well as PET, HDPE or glass due to flavor migration and scalping. Glass remains an ideal barrier to preserve fluid milk flavor, but in the absence of light, HDPE and PET provide additional benefits while also maintaining fluid milk flavor

INTRODUCTION

The main concerns for the dairy industry for pasteurized fluid milk are preserving the quality and safety of the final product (Dainelli et al., 2008), while also preserving the characteristic pleasant flavor of milk (Alvarez et al., 2009). The characteristic flavor active compounds of pasteurized fluid milk are created during thermal processing (Shimamura and Uke, 2012). However, the flavor of milk has changed over time in part due to changes in packaging used to transport milk to the consumer (Dainelli et al., 2008; Azzara and Campbell, 1992). Light oxidation from transparent containers as well as scalping and migration from different packaging types affect the flavor of milk and have led to consumer dissatisfaction (Schiano et al., 2019; Schiano et al., 2017; Johnson et al., 2015; Potts et al., 2017; Potts et al., 2016).

Pasteurized fluid milk is more susceptible to packaging related off-flavors than many other beverages because of its relatively bland taste. The dairy industry, as such, has focused on light blocking options in regards to packaging. Light oxidized flavor is a well-established and well-researched off flavor in retail fluid milk in transparent containers, and is caused by photo oxidation of riboflavin and other natural occurring photosensitive compounds in milk (Northrop-Clewes and Thurnham, 2012; Brothersen et al., 2016; Johnson et al., 2015; Walsh et al., 2015; Schiano et al., 2019). Paperboard cartons are generally considered to have good light barrier properties compared to other fluid milk package materials such as polyethylene terephthalate (PET), high density polyethylene (HDPE), or glass (Potts et al., 2016).

In addition to the light oxidized flavor, off odors and flavors in milk can be created and/or enhanced by flavor active compounds from packaging itself via migration and scalping (Bassette et al., 1986). Food migration is the transfer of unwanted chemical contaminants such as

trace amounts of solvents, byproducts, additives, and/or plastic monomers into a food from the surrounding environment or packaging (Kim-Kang, 1990). Food scalping is the transfer of desirable volatile food flavors and aromas to the packaging through absorption or release of undesirable flavors and aromas into the food from the surrounding packaging (Roland and Hotchkiss, 1991). Migration and scalping of flavor active compounds are heavily affected by the volatility and polarity of both the food matrix and packaging, lipophilicity of the product, physical structure (solid or liquid), and temperature of the product and storage conditions (Sajilata et al., 2007).

Paperboard cartons and HDPE are the most widely used packaging types for fluid milk (Brody, 2009). Paperboard cartons are widespread because they are malleable and cheaper to produce than many other options (Brody, 2009). Paperboard cartons are made from wood chips that have been chemically treated to a pulped consistency. The pulped wood is then poured into molds and left to cure until set in the desired shape. Commonly, paperboard cartons are lined with millimeter thin layers of low-density polyethylene (LDPE) as a barrier between the paperboard and the product. LDPE acts as a fluid barrier because of its resistance to liquids, pH, and alcohol (Nielsen et al, 1992; Simon and Hansen, 2001). Paperboard cartons have potential disadvantages because paperboard is highly permeable to the surrounding environment, and the paperboard itself is susceptible to off-flavors created through the oxidation of residual resins used during processing and the wood lignin that can migrate into a product (Ziegleder, 1998). HDPE, though more costly than paperboard, is commonly used because of its structural stability, moisture resistance, and barrier to the surrounding environment (Crosby, 1981; Nielsen et al., 1992). Polyethylene terephthalate (PET) is also an alternative for packaging of fluid milk (Brody, 2009). Polyethylene terephthalate (PET) when compared to HDPE is highly resistant to

acids, oils, and alcohols and generally a good barrier against moisture (Sajilata et al., 2007). PET is not as quite as rigid, durable, or resistant to heat as HDPE but is cheaper to produce and more malleable, making it easier to manipulate during production and recycling (Sajilata et al., 2007).

The role of flavors from packaging (scalping or migration) in fluid milks have not been fully investigated. Previous research has shown that salted and unsalted butter absorbed flavors from packaging and the surrounding environment at 4°C storage (Lozano et al., 2007a). Whole, low-fat and skim milk packaged in polyethylene coated half pint paperboard cartons had package off-flavor by day 1 at 4°C (Leong et al., 1992). However, different package materials for fluid milk have not been directly compared in the absence of light nor have volatile compounds been determined. The objective of this study was to determine the role of packaging on the chemical and sensory properties of milk in the absence of light exposure.

MATERIALS AND METHODS

Experimental Overview

Whole and skim milk were high temperature short time processed and filled into six different packages: amber glass (TraceClean® Wide Mouth Packers VWR, Radnor, PA), LDPE Low-Density Polyethylene (Wide Mouth, VWR), HDPE High-Density Polyethylene (Wide Mouth, VWR), PET polyethylene terephthalate (Storage Bottles, Corning®, Big Flats, NY), LLDPE Linear-Low-Density Polyethylene (Gusset Bag, Elkay Plastics Co., Austell, GA), or paperboard carton (Evergreen® Barrier Paperboard, Evergreen Packaging, Raleigh, NC) and stored at 4C. Sensory properties (descriptive sensory analysis (DA)) and volatile compound analysis (headspace solid phase micro extraction gas chromatography mass spectrometry (HS-SPME-GCMS)) were conducted at time 0 and days 5, 10, and 15. Consumer difference testing (tetrad tests) were conducted for whole and skim fluid milk packaged in **HDPE** vs **Glass**, **PET**

vs. **Glass**, and **Paperboard Carton** vs **Glass** at day 10. The entire experiment was repeated three times.

Milk Processing

Raw milk (<10000 CFU/mL aerobic plate count, <100,000 SCC) was standardized (whole milk 3.25+/-0.2 % fat and skim milk <0.10+/-0.05 % fat) and processed at the North Carolina State University Dairy Enterprise System (Raleigh, NC). Skim and whole milk were high temperature short time (HTST) processed (77 °C for 25 sec) and filled into 6 different 280 mL (±10 mL) packaging types; amber glass, LDPE, HDPE, PET, LLDPE, or paperboard carton. All packaging types were sanitized prior to filling in a 100 ppm (NaOCl) sanitizer/water solution (XY-12, Ecolab, St. Paul, MN). Processing and filling were done with minimal light to reduce light exposure. Packages were filled to the same volume: 230 mL (± 10mL) with 50 (±10mL) of headspace. Paperboard cartons were filled using an Evergreen EQ-2 gable top filler (Evergreen Packaging, Cedar Rapids, IA) system. All other packaging types were hand filled using a Micro Thermics Ultra-Clean Fill Hood (Micro Thermics, Raleigh, NC). Milks were stored at 4^{o}C and covered with black laser cloth to shield from light. Milks were sampled for sensory and instrumental analysis on day 0, 5, 10, 15 post processing. Total aerobic plate and coliform counts (coliform and aerobic plate count Petrifilms; 3M, St. Paul, MN) were conducted on milks at day 0, 10, 15 to monitor microbial quality.

Dissolved Oxygen

The dissolved oxygen of skim and whole milk of each package type was measured using a benchtop dissolved oxygen analyzer (YSI Model 5100, Yellow Springs, OH) at each time point (0, 5, 10, 15 days). The oxygen probe was placed in the sample, with both ends of the probe submerged, and stirred using the self-stirring probe BOD probe attachment. The reading was

then allowed to stabilize for dissolved oxygen and a measurement was taken. All measurements were done at 21^{o}C in triplicate for skim and whole milk on each package type and each experimental replication. Measurements were given in mg/L of oxygen in sample. Calibration for dissolved oxygen was done initially before each time point by placing the probe in a known oxygen environment (250 mL of water in a bottle aerated for 15 min with pure 0_2, the probe was stabilized for 15 min in the aerated water). The probe was adjusted if readings of known oxygen environment were out of specifications. Barometric pressure was adjusted to current elevation and temperature in the setting before each calibration

Headspace Oxygen

The headspace oxygen of skim and whole milk of each package type was measured using a benchtop oxygen analyzer (Quantek Model 905, Grafton, MA) at each time point (0, 5, 10, 15 days). To prevent oxygen leaking in or out of the packaging, the package was first fitted with a foam rubber septum (Foam Septum, Grafton, MA). The package was then punctured using a 20/2"/5 leur needle (N720 Hamilton Needle, Franklin, MA) with a side-slotted opening. The oxygen analyzer was then turned on until a stable oxygen reading was established. Readings were conducted at 21^{o}C. All measurements were done in triplicate for each package type and each experimental replication for skim and whole milk. A calibration was done initially before each time point by filling a heat sealed plastic bag (size 2"x2", thickness 0.0009", Gusset Bag, Elkay Plastics Co., Austell, GA) with pure N2 through a foam rubber septum then measuring the atmosphere in the bag. The reading was adjusted to 0% oxygen (+/- 0.1%) with the ZERO potentiometer on the back of the analyzer as needed. The atmosphere of the room was then used to check calibration between each sample and re-adjusted or re-calibrated if necessary.

Descriptive Sensory Analysis

Samples were prepared with overhead light off to minimize lights effects on milk. Sensory profiles of milks from each package type at each time point were evaluated by 7 experienced and trained panelists using an established sensory language for fluid milk (Lee et al., 2017; McCarthy et al., 2017; Lozano et al., 2007b). Each panelist had a minimum of 90 h of previous experience evaluating the sensory profile of different fat levels of HTST fluid milk. Attributes were scored using a 0 to 15 point scaling consistent with the Spectrum Method (Meilgaard et al., 2007). Each milk was evaluated at each time point (0, 5, 10, 15 days) in duplicate by each panelist. Milks were poured (40 mL) into soufflé cups (65 mL) numbered with randomized 3-digit codes. Panelists evaluated milks from each experimental replication of each fat type (6 milks) in a randomized order in one session. A 3 minute rest between samples was enforced. Paper ballots were used for data collection.

Volatile Compound Analysis of Fluid Milk

HS-SPME GC-MS

Volatile compounds were extracted from milks at each time point (0, 5, 10, 15 days) by headspace solid phase micro extraction (HS-SPME) followed by gas chromatography mass spectrometry (GC-MS) with a ZB-5ms (30m x 0.25mm ID x 0.25µm) (Phenomenex) and a ZB-Wax (30m x 0.25mm ID x 0.25µm) column (Phenomenex) with mass spectrometry (7820 GC 5975 MSD, Agilent Technologies Inc., Santa Clara, CA). Samples were injected via a CTC Analytics CombiPal Autosampler (CTC Analytics, Zwingen, Switzerland). Extraction methods were modified from Jo et al. (2018). Five (5g) grams of milk were measured in triplicate in SPME vials (Microliter Analytical, Suwanee, GA) with 20 uL of internal standard added 81 mg/kg 2-methyl-3-heptanone (Sigma-Aldrich, St. Louis, MO) in methanol and 167 mg/kg 2-methylpentanoic (Sigma-Aldrich) in methanol. Vials were equilibrated at 40^{o}C for 20 min with 3

sec of 350 rpm agitation. A single DVB/Carboxen/PDMS 1.0 cm fiber (Supelco, Bellefonte, PA) was used for all analysis. The SPME fiber was exposed to the samples for 25 min at depth 3.1 cm. The fiber was retracted and injected at 5.0 cm in the GC inlet for 7.5 min. The GC oven was held at 40C for 3 min with a gradient of 10°C/min to 90°C with no hold, the gradient was increased at a rate of 5°C/min to 200°C and held for 10 min, then increased to 20°C/min 250°C and held for 5 min at completion. Helium was pressurized at 7 psi in the column and maintained at a constant flow rate of 2 mL/min. A standard scan mode was used (30 to 350 m/z) to identify compounds of interest for initial evaluation. Selective Ion Monitoring (SIM) mode was subsequently utilized for compound identification using authentic standards.

HS-SPME GC-O GC-MS

HS-SPME gas chromatography olfactometry (GC-O) was used to identify aroma active compounds. GC-O was conducted on an Agilent 6850 GC (Agilent Technologies Inc.) with a ZB-5ms (30m x 0.25mm ID x 0.25µm) column (Phenomenex) or a ZB-Wax plus (30m x 0.25mm ID x 0.25µm) column (Phenomenex). Ten (10) ml of milk was added to 40 ml vials in duplicate. Vials were equilibrated for 30 minutes at 40^{o}C and SPME fibers were then exposed to the headspace of the vials for 30 min at 2 cm. Fibers were manually injected at 3 cm depth. The fiber was initially exposed to the inlet for 10 min at 250^{o}C. The GC program was held at 40°C for 3 min and increased gradually to 150°C over 11 min (10°C/min). The temperature was then increased to 250°C over 3.5 min (30°C/min) and then held for 10 min. Carrier gas used was helium at a flow rate of 1.5 mL/min. The splitter was utilized at a 1:1 ratio between the FID and sniffer port. Two experienced sniffers recorded aroma events, retention times and perceived aroma intensities using a 0 to 5 point post peak intensity scale.

Compound Identification and Quantitation

Compound identification was achieved by mass spectra (NIST, 2017) and odor properties of authentic standards. Retention indices of volatiles compounds were calculated (van den Dool and Kratz, 1963) using a C6-22 alkane series (Sigma-Aldrich) under identical GC-O/MS conditions. External standard curves for nine selected compounds (styrene, xylene, 2-ethyl-1-hexanol, hexanal, nonanal, p-cymene, acetophenone, toluene, octanal) were generated using skim or whole (HTST) milk packaged in glass as the matrix. Each stock solution was prepared using authentic standards (Sigma-Aldrich) and diluted (methanol, ethanol or water based on solubility) at separate points for a 5-point standard curve. Each matrix was also spiked with 20uL of an internal standard (81 mg/kg 2-methyl-3-heptanone in methanol) with the ratio of response calculated along with the external standard in the matrix.

DSE-SAFE

Direct solvent extraction solvent assisted flavor extraction (DSE-SAFE) was chosen to evaluate skim milk stored at 4C° for 15 days from paperboard cartons and glass (control) because of its more sensitive recovery of semi-volatile compounds and higher molecular weight compounds (Xie et al., 2013; Lozano et al., 2007b; Engel et al., 1999). DSE-SAFE was utilized for the identification and determination of specific compounds related to paperboard cartons that were identified at LOQ by SPME GCMS.

Fluid milk was extracted using a modified method of direct solvent extraction as described by Engel et al. (1999). Three hundred (300) mL of fluid milk was separated into three equal parts into three 250 mL Teflon bottles (Thermo, Rochester, NY). One hundred (100) mL of ethyl ether (Sigma Aldrich), 40 uL of each internal standard was added to each bottle (2-methy-3-heptanone, 81mg/kg, neutral/basic fraction, 2-methylpentanoic acid 167 mg/kg acid-fraction)

(Sigma Aldrich), and thirty grams (30g) of NaOH (Sigma Aldrich) to break the emulsion. Each bottle was sealed and then shaken vigorously for 2 min, untightened to let out excess pressure and then placed for 30 min on a platform mixer (Rotomix type 50800 Thermolyne, Willow Springs, NC) at the highest setting. Bottles were then centrifuged at 2500 xg (Thermo Lynx 6000, Thermo, Rochester, NY) for 30 minutes. The top aqueous layer (ethyl ether) was then removed and dried using anhydrous sodium sulfate (Na2SO4) (Sigma Aldrich) to remove excess water. All samples were concentrated to 100 mL under nitrogen gas.

Concentrated samples were then distilled using a SAFE Apparatus, assembled as previously described **(**Lozano et al., 2007b; Engel et al., 1999). A round 1L flask was attached to the main SAFE head apparatus and submerged in a 40°C water bath to maintain temperature. The SAFE head apparatus was then attached to two glass traps, in series, for collection of sample. Both traps were submerged in liquid nitrogen filled Nalgene buckets that were constantly filled to maintain temperature. The SAFE Apparatus (Ace Glassware, Vineland, NJ) was then brought to 10^{-5} Torr vacuum and attached to the end of the second trap in series. Sample was introduced slowly (over 15 min) via a stopcock into the main SAFE apparatus head into the 1L flask, volatilizing the sample and pulling it through the SAFE apparatus and condensing it in the first trap. After all sample was fully introduced, the SAFE apparatus was maintained at constant vacuum (10^{-5} Torr) and liquid nitrogen was maintained in nalgene buckets for 2 h for maximum retention. The primary trap with condensed sample was collected and allowed to thaw at room temperature. Sample was then dried with anhydrous sodium sulfate (Na2SO4) in an amber glass jar (250mL) and then concentrated to 20 mL under a stream of nitrogen gas. Concentrated sample was then transferred to a screw-top glass tube for phase separation.

To aid in compound detection, all samples were separated into two separate fractions, neutral/basic and acidic. The concentrated sample was first washed with 3mL of 0.5M sodium bicarbonate (Fisher Scientific, Hampton, NH) and shaken for 10 min. The aqueous layer was removed (bottom layer) after each washing and collected. The sodium bicarbonate wash was repeated twice. The sample was then washed with 2mL of saturated NaCl solution (23% w/w) (VWR) and shaken for 10 minutes with the aqueous layer removed (bottom layer) and collected into the same glass tube. The ether phase remaining (upper layer) was the resultant neutral/basic fraction and was then concentrated to 2 mL under a stream of nitrogen gas, dried with anhydrous sodium sulfate (Na2SO4) into a new 2 mL glass vial, and concentrated to 0.5 mL under nitrogen gas.

The transferred aqueous layer (bottom layer) in the glass tube was acidified through the addition of 6M hydrochloric acid (Sigma Aldrich) to a pH of approximately 2.0. Five (5) mL of diethyl ether was then added to the tube and shaken for 10 min and the ether phase removed. This process was repeated three times. The collected ether phase was the acidic fraction. The acidic fraction was then concentrated to 2 mL under a stream of nitrogen gas, dried with anhydrous sodium sulfate (Na2SO4) and concentrated to 0.5 mL under nitrogen gas.

Compound Identification for DSE-SAFE

Ten µL of the acidic and neutral/basic fraction were injected in split-less mode in duplicate on a ZB-5ms (30m x 0.25mm ID x 0.25µm) column and a ZB-Wax plus (30m x 0.25mm ID x 0.25µm) column installed in a Agilent 7820-GC paired with a 5975-MSD. All injections had a 2.5 minute solvent delay to protect the instrument. The GC oven was at held at 40C for 3 min with a gradient of 10°C/min to 90°C with no hold, the gradient was increased to a rate of 5°C/min to 200°C and held for 10min, the gradient was then increased to 20°C/min

250°C and held for 5 min at completion. Selective Ion Mode (SIM) and scan mode were used in tandem to identify and quantify compounds of interest. Compound identities were confirmed with authentic standards. Internal standards were used to calculate relative abundance for compounds of interest.

Consumer Difference Tests (Tetrads)

Sensory testing was conducted in accordance with NCSU Institutional Review Board for human subject's regulations. Tetrad difference testing (ASTM E3009) was conducted for both skim and whole milks with 4 packaging types: Paperboard Cartons, High-Density Polyethylene (HDPE), Polyethylene Terephthalate (PET), and Glass (control) at day 10. Consumers were presented three separate tetrad tests for whole and skim milk (6 tetrads): **HDPE** vs **Glass**, **PET** vs. **Glass**, **Paperboard Carton** vs **Glass** at day 10 of shelf-life. Package types for tetrads were selected based on descriptive analysis and realistic practical applications. HDPE and PET packages were selected because they make up more than 85% of fluid milk packaging sales (USDA, 2013). Paperboard cartons contribute close to 14% of fluid packaging sales with the majority of that being used for the school lunch programs (USDA, 2013). Consumers (n=50) were recruited via emails from the North Carolina State University Sensory Service Center database with the qualifier being that they were self- reported regular consumers of fluid milk. During testing each consumer was instructed both verbally and provided with an instruction guide describing how a tetrad test is performed. They were asked to pair the 4 coded samples based on which ones they believed to be the most similar to each other. Testing was replicated with two of the experimental replications for skim and whole milk for all three tetrad pairs (n=100).

Milks (60 mL) were evaluated by consumers in 128 mL Styrofoam cups coded with three digit codes. Milks were served at 4ºC. Each panelist was given 4 coded samples at once and instructed to sample them in the order presented left to right. A 3 min enforced rest occurred between each tetrad during which consumers were requested to rinse their mouths with water and to take a bite of unsalted cracker. Consumers were asked to complete 3 sets of tetrads (one fat content level) in one seating with the order of presentation of each tetrad test randomized between each consumer. Consumers were compensated at the completion of the test with a $5 Target gift card. Compusense Cloud (Compusense, Guelph, Canada) was used for data collection.

Statistical Analysis

Data analysis was conducted at a 95% confidence ($p < 0.05$). Analysis software used included XLSTAT version 2019.1.3 (Addinsoft, New York, NY) and SAS version 9.4 (Cary, NC). Two separate analyses were conducted, one with whole milk and one with skim. Each experiment was designed as a randomized complete block design with a timepoint by package type factorial arrangement of treatments and evaluated as a mixed model analysis of variance. Principle component analysis was used to visualize differences among volatile compounds among package types within each fluid milk fat content. Consumer difference data were evaluated using the minimum correct judgement for significance ($p < 0.05$) (ASTM E3009).

RESULTS AND DISCUSSION

Milk microbial quality

No coliforms were detected in pasteurized whole or skim milks. All milks had an aerobic plate count of less than 10^2 CFU/mL initially and less than 10^4 CFU/mL at day 15.

Dissolved and Headspace Oxygen Analysis

Dissolved oxygen concentration decreased over storage time for all packaging types regardless of milkfat content ($p < 0.05$) (**Tables 2.1, 2.2**). Dissolved oxygen decreases were consistent with previous studies found for fluid milk (Karatapanis et al., 2006; Zygoura et al., 2003). Dissolved oxygen for skim and whole milks packaged in LLDPE decreased faster compared to milks in other packages, presumably due to the greater permeability of the LLDPE ($p < 0.05$; **Tables 2.1, 2.2, 2.3**). Headspace oxygen (%O2) concentration decreased over storage time for Glass, PET, HDPE, and LDPE regardless of milkfat content ($p < 0.05$) (**Tables 2.4, 2.5**). Headspace oxygen in milks packaged in cartons and LLDPE did not decrease over storage time due to the high permeability of both packaging types ($p > 0.05$; **Tables 2.3, 2.4, 2.5**).

Descriptive Analysis

Milks (whole and skim) packaged in different packages demonstrated distinct flavor differences initially and with storage time (**Tables 2.6, 2.7**). Skim milks packaged in paperboard cartons and LLDPE had lower cooked and sweet aromatic flavors with storage time and distinct refrigerator/stale flavor when compared to milks in the other packaging types ($P < 0.05$; **Table 2.6**). This flavor difference may be due to the high permeability of paperboard cartons and LLDPE (**Table 2.3**). High permeability of packaging has been shown to increase stale flavor and decrease overall flavor acceptability in UHT milks (Wadsworth and Bassette, 1985). Paperboard/cardboard flavor was only detected in milks packaged in cartons, and was detected within hours of packaging (day 0) and increased with storage time (**Table 2.6**). Previous studies have found that paperboard or cardboard off flavors associated with paperboard cartons are detected by sensory panelists from Day 1 to Day 5 of storage time (Leong et al., 1992;

Karatapanis et al., 2006). Skim milk packaged in LLDPE had higher refrigerator/stale flavor than skim milk packaged in cartons across storage ($P < 0.05$; **Table 2.6**).

Whole milks packaged in paperboard carton and LLDPE had lower sweet aromatic flavor across storage time compared to milks filled into glass, PET, HDPE, or LDPE ($P < 0.05$; **Table 2.7**). Similar to skim milk, paperboard/cardboard flavor was only detected in milks packaged in paperboard cartons (**Table 2.7**). Unlike skim milk, this flavor was not detected until day 5, but also increased through day 15 (**Table 2.7**). This effect has been previously documented in whole milk packaged in paperboard cartons by Day 5 (Karatapanis et al., 2006). Refrigerator/stale flavor was detected in milks packaged in LLDPE at day 0 and in whole milk filled into paperboard carton milk by day 10, and this flavor increased with storage time (**Table 2.7**). Though skim and whole milk cannot be directly compared, paperboard/cardboard and refrigerator/stale flavors in milk packaged in paperboard cartons or LLDPE were generally more intense for skim milk at a specific timepoint compared to whole milk (**Tables 2.6, 2.7**). This flavor difference between skim and whole milk is consistent with previous studies that off-flavors due to paperboard packaging were more pronounced in skim milk compared to whole milk (Leong et al., 1992). Off flavor problems in fluid milk due to package-related absorption or migration do increase with a decrease in container size due to the increase in the contact surface area with fluid milk. Comparisons have previously been made among various package sizes such as pint, quart, gallon, and liter (Leong et al., 1992; van Aardt et al., 2001; Bradley et al., 1980). Our packaging volume (230 mL) was selected to represent the largest contact area (surface area) or "worst case" scenario. This volume is also the serving size for school lunch milk in the school lunch program in the United States (8 fl oz, 236 mL) and that milk is also typically served in a paperboard carton (Sipple et al., 2020).

Volatile Compound Analysis

Skim and whole milks packaged in paperboard cartons or LLDPE had higher concentrations of packaging/refrigerator taint volatile compounds styrene, acetophenone and 2-ethyl-1-hexanol than milks filled into LDPE, HDPE, PET, or glass ($P < 0.05$; **Tables 2.8, 2.9**). Styrene concentrations increased across all packaging types except for the glass package. Styrene is a marker for degradation of plastic barriers over storage time (López et al., 2008), is present in refrigeration environments (Lozano et al., 2007a), and would be expected to migrate in from the environment with permeable containers such as cartons and LLDPE. Ethylbenzene is the main component in the production of styrene, and acetophenone is a byproduct of the oxidation of styrene and ethylbenzene (Ziegleder, 1998; Azzara and Campbell, 1992; Vera et al., 2020). The more permeable barriers of paperboard cartons and LLDPE (**Table 2.3**) may be the cause of increased concentrations of styrene and acetophenone, which migrates into the milk from the refrigeration environment. The compound 2-ethyl-1-hexanol does occur naturally in some foods at low concentrations but industrially it is used mainly used as a solvent for household cleaners and as a precursor to plasticizers, which are substances that make materials (in this case plastic) more pliable or flexible (Vera et al., 2020). The compound 2-ethyl-1-hexanol is also known/considered to be an indoor air pollutant coming from the condensation of refrigerators or air-conditioners (Wakayama et al., 2019) and may also migrate through permeable packaging material into fluid milk. Styrene is "generally recognized as safe" by the FDA though it is documented to cause a "chemical/plastic/stale" off flavor in most food products when at detectable levels (Piringer and Baner, 2000). Acetophenone is permitted by the FDA for human consumption (21CFR172, 2019) but is known to cause "stale" off flavors in milk (Arnold et al., 1966) and 2-ethyl-1-hexanol has a "chemical/cleaning agent" aroma (Drake et al., 2014).

Limonene and p-cymene increased in skim milk packaged in LLDPE or paperboard cartons compared to LDPE, HDPE, PET, and glass. A similar increase was documented in whole milk packaged in LLDPE but not paperboard carton (**Tables 2.8, 2.9**). Limonene is a compound found in fluid milk and is also used as a metric for assessment of environmental air quality (Subramanian, 2000). P-cymene is a degradation compound of limonene and correlates with the increase of limonene ($R2 = 0.74$, $P < 0.05$). The increased concentrations of limonene and the further degradation into p-cymene may be due to the more permeability of LLDPE and paperboard cartons (**Tables 2.3, 2.8, 2.9**). By day 5 of storage, toluene and p-xylene concentrations were higher in skim milks packaged in HDPE, LDPE, LLDPE, and paperboard cartons compared to skim packaged in PET or Glass (**Table 2.8**). According to the Agency for Toxic Substances and Disease Registry (ATSDR), toluene and all three isomers of xylene (m-xlyene, o-xlyene, p-xlyene) are used as solvents or intermediates in the production of polymers for plastics (Toxicological profile for toluene, 2017; Toxicological profile for xylene, 2007). The degradation of plastics may be the cause for increases detection of toluene and p-xylene over time. These volatile compounds are likely contributors to stale/refrigerator flavor in skim and whole milks, consistent with Lozano et al. (2007a) who associated these compounds with refrigerator/stale flavor in stored butter.

Skim and whole milks filled into paperboard cartons had increased levels of hexanal compared to milks in other packaging types ($P < 0.05$; **Tables 2.8, 2.9**). This difference in hexanal concentration may signify the degradation of the surrounding paperboard packaging, which may increase hexanal concentrations in skim milk across time. Hexanal is a degradation compound formed from the oxidation of linoleic acid during the processing of wood extracts or paper pulp and has been associated with off-flavor in food products packaged in paperboard and

cardboard (Czerny and Buettner, 2009). Ziegler (1998) found that hexanal can be used as a guide for tracking lipid oxidation of paperboard across time. Both of these studies and others have found the association of increased concentrations of aldehydes such as hexanal, heptanal, octanal, nonanal, and decanal to an increase in cardboard/paperboard flavor (Czerny and Buettner, 2009; Ziegler, 1998; Whitson et al., 2010; Caelenberg et al., 2013). The elevated level of hexanal in milks in paperboard cartons compared to whole and skim milks in the other five packaging types may be attributed to hexanal being the largest source of paperboard or cardboard off flavor (**Tables 2.8, 2.9**). Cardboard flavor in dairy products has also previously been sourced to lipid oxidation compounds including hexanal (Whitson et al., 2010). Skim milk packaged in paperboard cartons or LLDPE and whole milk packaged in LLDPE also had higher concentrations of nonanal compared to milks filled into other package types ($P < 0.05$; **Table 2.8**). This oxidation compound may indicate elevated lipid oxidation in milks packaged in these containers indicative of increased oxygen and greater permeability of these package types compared to the others in this study. Dynamic headspace analysis conducted on commercial milks revealed that milks with higher concentrations of accessible oxygen were correlated with higher concentrations of lipid oxidation compounds (Kim and Morr, 1996). Increases in lipid oxidation were also documented in UP and HTST milks stored at 4C for 14 days (Jo et al., 2018). Karatapanis et al. (2006) also documented lipid oxidation in HTST whole milk across 7 days of refrigerated storage (Karatapanis et al., 2006). Concentrations of volatiles compounds were generally lower in whole milk compared to skim milk (**Tables 2.8, 2.9**). This result may signify that packaging related compounds may be soluble in milkfat and thus less available for headspace extraction as well as sensory perception since refrigerator/stale and cardboard flavors by sensory analysis were also less prevalent in whole milks compared to skim milks. In a

previous study, fluid milk with higher fat content had lower recoveries of most volatile compounds via headspace extraction compared to skim milk (Jo et al., 2018).

Skim milk packaged in paperboard carton or LLDPE were closely aligned across storage time with increasing concentrations of packaging related volatile compounds styrene, toluene, p-xylene, p-cymene, limonene, acetophenone, 2-ethyl-1-hexanol and hexanal (**Figure 2.1**). Skim milk packaged in LLDPE or paperboard carton after Day 5 of storage were distinct from milks filled into glass, LDPE, HDPE, or PET (**Figure 2.1**). Whole milks were not as distinct in volatile compound differences compared to skim milks but whole milk packaged in LLDPE was closely aligned across storage time with increasing concentrations of packaging related volatile compounds styrene, toluene, p-xylene, p-cymene, acetophenone , 2-ethyl-1-hexanol and hexanal (**Figure 2.2**). Whole milk packaged in LLDPE after Day 5 was distinct from milk filled into glass, LDPE, HDPE, and PET (**Figure 2.2**). Karatapanis et al. (2006) reported slight increases in packaging/storage volatiles in the low part per billion (μg/kg) for toluene and various benzene compounds (ethyl benzene, 1,4-dimethyl benzene, 1,3-dimethyl benzene, 1,3,5-trimethyl benzene) in whole HTST milk across 7 days of refrigerated storage time.

Relationship between volatiles compounds and sensory attributes

Principal component biplots provide a visualization of the changes in the volatiles and sensory attributes that characterize differences among the milks with storage time along with correlations between sensory attributes and volatile compounds (**Figure 2.3, 2.4**). All milks decreased in cooked/milky and sweet aromatic flavors with storage time with milks filled into paperboard cartons or LLDPE having the most distinct storage-related changes (**Figure 2.3**). Paperboard/cardboard flavor and the volatile compound hexanal were correlated ($R2 = 0.84$, $P < 0.05$) (**Figure 2.3**). Refrigerator stale flavor was correlated with the volatile compounds styrene

(R2 = 0.88, P < 0.05), p-cymene (R2 = 0.73, P < 0.05), acetophenone (R2 = 0.71, P < 0.05) and 2-ethyl-1-hexanol (R2 = 0.82, P < 0.05). Styrene has also been attributed to "refrigerator/stale" flavor in stored butter (Lozano et al., 2007a). Similar to skim milk, whole milk filled into cartons was associated with paperboard/cardboard flavor and astringency and the volatile compound hexanal (**Figure 2.4**). Whole milk packaged in LLDPE and paperboard cartons were also associated with higher overall aroma and refrigerator stale flavor (**Figure 2.4**).

Direct solvent extraction solvent assisted flavor extraction

Skim and whole milk packaged in paperboard cartons had distinct paperboard and refrigerator stale flavors. HS-SPME-GCMS analysis established significant migration of volatile compounds at Day 15 in paperboard cartons (whole and skim milk) associated with packaging and refrigerator tainting compounds (**Tables 2.8, 2.9**). In particular, headspace volatile analysis of skim milk filled in paperboard cartons at Day 15 revealed traces of plasticizers and ink related compounds at detectable limits but not above base line noise and thus not quantifiable. We decided a more direct and sensitive extraction process (DSE-SAFE) would be needed to get an increased recovery of these specific volatile compounds.

SAFE extraction resulted in a much clearer representation of tainting compounds associated with paperboard packaging. Relative abundance of packaging/refrigeration related compounds in skim milk stored in paperboard cartons versus glass for 15 days (ug/kg) were: styrene 283 +/- 23.3 vs 1.7 +/- 0.7, toluene 930 +/- 85.7 vs 8.6 +/- 0.5, p-xylene 46 +/- 12.4 vs 3.6 +/- 2.1, acetophenone 38.7 +/- 13.9 vs not detected, and 2-ethyl-1-hexanol 287.3 +/- 8.19 vs 25.1 +/- 0.90.). These values were higher in skim milk by SAFE extraction compared to headspace extraction (SPME GCMS) milk from paperboard carton, as expected, but it is also important to note that these values from SAFE were calculated based on internal standard

recovery rather than actual quantitation with standard curves. More importantly, as expected, additional compounds were detected in skim milk by DSE-SAFE extraction (but not by SPME-GCMS) including n-propylbenzene (dye solvent), benzophenone (photoiniators, plasticizer), dibutyl phthalate and diethyl phthalate (plasticizers and softening agents) (Czerny and Buettner, 2009; Frostling et al., 1984; Leja and Lewandowicz, 2010). The compounds n-propylbenzene, benzophenone, diethyl phthalate and dibutyl phthalate were detected in skim milk packaged in paperboard cartons at 210 +/- 28.6, 6 +/- 0.3, 56 +/- 18, and 300 +/- 1.79 parts per billion (ug/kg) respectively via relative abundance. Comparatively, n-propylbenzene, benzophenone, dibutyl phthalate and diethyl phthalate were not detected in skim milk packaged in glass. The compound n-propylbenzene is a compound found in plastic packaging for milk (Abrantes, 1993), though it is not recognized in the indirect food additives section of the CFR by the FDA and is considered nontoxic. Benzophenone was previously used as photoinitator to cure ink on packaging materials, namely paperboard, but is no longer authorized as a food additive or plasticizer by the FDA as of 2018, because of its carcinogenic potential (21CFR172: Food additives permitted for direct addition to food for human consumption, 2019; 21CFR177: Indirect food additives: Polymers, 2019). Benzophenone and n-Propylbenzene have been previous found in skim milk powders (Shiratsuchi et al., 1994; Sanches-Silva et al., 2008), possibly due to these products being packaged in plastic and using photoinitators to cure the ink on the packages. Dibutyl phthalate is designated as an indirect additive by the FDA (21CFR177: Indirect food additives: Polymers, 2019). Diethyl phthalate is classified as a plasticizer by the FDA and considered a prior-sanctioned food ingredient, which means the FDA sanctions it for a specific use in food (21CFR181.27: Prior-sanctioned food ingredients, 2019). Dibutyl phthalate and diethyl phthalate have been previously found in milk and other dairy products (Sørensen, 2006; Fierens et al.,

2013). Determination of exact concentrations of these specific tainting compounds in all skim and whole milks filled into paperboard and or other packaging types at larger packaging volumes would require additional solvent extractions and merits future work.

Consumer Difference Tests

Consumers detected differences between skim and whole milk filled into paperboard cartons and glass ($P<0.05$; **Tables 2.10, 2.11**). Consistent with minimal to no differences by trained panels, consumers could not detect differences between the PET and glass pairings and HDPE and glass ($P>0.05$; **Tables 2.9, 2.10**). These results are consistent with a previous study where consumers reported similar flavor and acceptability of PET and HDPE (Potts et al., 2017). The results are also consistent with our trained descriptive panel and volatile compound results. Skim or whole milks filled into PET, HDPE or glass were similar in overall flavor profile with no paperboard/cardboard or refrigerator stale off-flavors present by Day 15 (**Tables 2.6, 2.7**).

CONCLUSIONS

Off-flavors due to packaging and refrigeration environment were detected in fluid skim and whole milk over refrigerated storage time. Off-flavors in milks detected by sensory analysis were correlated with the development of increased specific volatile compounds. Milks packaged in paperboard cartons and LLDPE had the highest intensities of off-flavors due to permeability and migration with off flavors present by Day 0 in skim milk. Consumer differences tests were comparable with trained panel and volatile compound analysis suggesting that HTST milks packaged in HDPE, PET, or glass in the absence of light exposure have no discernable differences by Day 10 post processing. In contrast, milks filled into paperboard cartons were differentiated by consumers compared to milks filled into glass. Paperboard cartons are the most widely used packaging type for school lunch programs in the U.S., and milks packaged in

paperboard cartons had distinct off flavors and presence of specific migration volatile compounds. Our findings suggest that industry and policy makers may need to find new packaging alternatives for school lunch fluid milk. The consequences of using packaging that has significant off-flavors over time could affect how young children, and those children as adults, perceive fluid milk.

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Table 2.1: Concentration of dissolved oxygen (mg/L) in skim milk in different packages across 4 time points (day 0, 5, 10, 15).

	Time points (d. 0, 5, 10, 15)			
Package Type	Day 0	Day 5	Day 10	Day 15
Glass	10.31 ± 0.20 b	8.68 ± 0.50 c	5.71 ± 0.66 c	3.90 ± 1.20 c
PET	10.59 ± 0.08 ab	9.26 ± 0.40 b	7.77 ± 0.43 ab	5.12 ± 1.35 b
HDPE	10.66 ± 0.10 ab	9.37 ± 0.16 b	7.70 ± 0.35 ab	4.36 ± 0.50 bc
LDPE	10.61 ± 0.13 ab	9.00 ± 0.66 bc	7.04 ± 0.67 b	3.90 ± 0.32 c
LLDPE	10.46 ± 0.35 ab	7.08 ± 0.61 d	4.73 ± 0.96 d	0.96 ± 0.25 d
Carton	11.15 ± 0.13 a	10.06 ± 0.26 a	8.31 ± 0.59 a	7.02 ± 0.99 a

Means in the same column followed by a different subscript are significantly different ($p < 0.05$)

Table 2.2: Concentration of dissolved oxygen (mg/L) in whole milk in different packages across 4 time points (day 0, 5, 10, 15).

	Time points (d. 0, 5, 10, 15)			
Package Type	Day 0	Day 5	Day 10	Day 15
Glass	11.94 ± 0.08 b	10.63 ± 0.37 bc	7.89 ± 0.48 c	5.78 ± 0.36 b
PET	12.19 ± 0.11 a	10.09 ± 1.04 c	8.80 ± 1.01 b	4.72 ± 0.62 d
HDPE	12.18 ± 0.16 a	11.04 ± 0.44 ab	8.94 ± 0.56 b	5.37 ± 0.62 bc
LDPE	12.16 ± 0.14 a	10.57 ± 0.36 bc	7.75 ± 0.65 c	5.09 ± 0.59 cd
LLDPE	11.93 ± 0.07 b	7.57 ± 0.34 d	3.80 ± 0.43 d	1.16 ± 0.18 e
Carton	12.10 ± 0.06 a	11.47 ± 0.24 a	10.51 ± 0.26 a	8.38 ± 0.37 a

Means in the same column followed by a different subscript are significantly different ($p < 0.05$)

Table 2.3: Oxygen transmission rates of packaging types over 24 h[2]

Packaging	Glass	PET	LDPE	HDPE	LLDPE	Carton
Oxygen Transmission Rate[1]	<0.5	5	195	72	1009	801
Film/Packaging Wall Thickness (mm)	1.1 ± .1	1.1 ± .1	1.3 ± .2	1.3 ± .2	.019 ± .01	0.9 ± .2

[1]Oxygen Transmission Rate - (cc*mm/m2*24h*bar)
[2]Oxygen transmission rates were reported from packaging type manufacturer

Table 2.4: Concentration of headspace oxygen (%O2) in skim milk in different packages across 4 time points (day 0, 5, 10, 15).

	Time points (d. 0, 5, 10, 15)			
Package Type	Day 0	Day 5	Day 10	Day 15
Glass	20.2 ± 0.3 b	18.6 ± 0.4 cd	18.5 ± 0.4 b	18.4 ± 0.3 b
PET	20.2 ± 0.2 b	18.4 ± 0.3 d	17.7 ± 0.3 c	17.5 ± 0.3 c
HDPE	19.7 ± 0.1 c	19.0 ± 0.4 c	17.8 ± 0.6 c	17.8 ± 0.7 c
LDPE	19.7 ± 0.1 c	18.6 ± 0.3 cd	17.7 ± 0.3 c	17.9 ± 0.4 c
LLDPE	20.8 ± 0.1 a	20.9 ± 0.2 a	20.9 ± 0.2 a	20.9 ± 0.3 a
Carton	20.6 ± 0.2 a	20.5 ± 0.3 b	20.6 ± 0.3 a	20.7 ± 0.3 a

Means in the same column followed by a different subscript are significantly different ($p < 0.05$)

Table 2.5: Concentration of headspace oxygen (%O2) in whole milk in different packages across 4 time points (day 0, 5, 10, 15).

	Time points (d. 0, 5, 10, 15)			
Package Type	Day 0	Day 5	Day 10	Day 15
Glass	20.2 ± 0.4 b	19.3 ± 0.3 b	19.3 ± 0.1 c	18.8 ± 0.5 c
PET	20.1 ± 0.4 bc	18.7 ± 0.1 c	18.6 ± 0.4 d	18.3 ± 0.3 d
HDPE	19.7 ± 0.7 c	19.0 ± 0.8 bc	18.5 ± 0.2 d	18.5 ± 0.3 cd
LDPE	19.7 ± 0.7 bc	18.8 ± 0.7 bc	19.0 ± 0.7 cd	18.5 ± 0.2 cd
LLDPE	20.9 ± 0.1 a	20.9 ± 0.2 a	21.0 ± 0.1 a	21.1 ± 0.1 a
Carton	20.2 ± 0.2 bc	20.5 ± 0.1 a	20.1 ± 0.3 b	20.3 ± 0.4 b

Means in the same column followed by a different subscript are significantly different ($p < 0.05$)

Table 2.6. Trained panel profiles of skim milks filled into different packages with storage time (day 0, 5, 10, 15).

		Sensory Attributes[1]							
Time	Packaging Type	Overall Aroma	Sweet Aromatic	Cooked	Paperboard/Cardboard	Refrigerator Stale	Sweet	Salty	Astringency
Day 0	Glass	2.6 a	1.9 a	3.4 a	ND[2]	ND	2.0 a	1.6 a	1.9 b
	PET	2.6 a	1.6 b	3.3 a	ND	ND	2.1 a	1.7 a	2.0 b
	HPDE	2.6 a	1.5 b	3.3 a	ND	ND	2.0 a	1.7 a	2.0 b
	LDPE	2.6 a	1.4 bc	3.3 a	ND	ND	2.0 a	1.5 a	2.0 b
	LLDPE	2.6 a	1.2 cd	3.2 ab	ND	0.5 d	2.1 a	1.7 a	2.0 b
	Carton	2.7 a	0.9 c	3.2 ab	1.3 c	ND	2.0 a	1.5 a	2.0 b
Day 5	Glass	2.3 b	1.9 a	3.3 a	ND	ND	2.2 a	1.5 a	1.9 b
	PET	2.2 bc	1.5 b	3.3 a	ND	ND	2.0 a	1.7 a	1.9 b
	HPDE	2.0 cd	1.0 c	3.3 a	ND	ND	2.1 a	1.7 a	2.0 b
	LDPE	2.1 bcd	1.0 c	3.2 ab	ND	ND	2.1 a	1.6 a	2.0 b
	LLDPE	2.3 b	ND	3.0 b	ND	1.9 c	2.2 a	1.5 a	2.0 b
	Carton	2.2 bc	0.7 d	3.0 b	1.8 b	1.1d	2.0 a	1.5 a	2.1 ab
Day 10	Glass	2.1 bcd	1.5 b	3.3 a	ND	ND	2.0 a	1.7 a	1.8 b
	PET	2.1 bcd	1.0 c	3.2 ab	ND	ND	2.1 a	1.7 a	1.8 b
	HPDE	1.9 cd	0.8 cd	3.3 a	ND	ND	2.0 a	1.5 a	1.9 b
	LDPE	1.8 d	0.6 d	3.0 b	ND	ND	2.1 a	1.6 a	2.0 b
	LLDPE	2.3 b	ND	3.0 b	ND	2.4 b	2.0 a	1.5 a	2.2 ab
	Carton	2.2 bc	ND	3.0 b	2.0 b	1.9 c	2.0 a	1.5 a	2.2 ab
Day 15	Glass	2.1 bcd	1.0 c	3.0 b	ND	ND	2.0 a	1.5 a	1.8 b
	PET	2.2 bc	0.9 cd	3.0 b	ND	ND	2.2 a	1.7 a	1.9 b
	HPDE	2.0 cd	ND	3.0 b	ND	ND	2.2 a	1.7 a	1.8 b
	LDPE	1.8 d	ND	3.0 b	ND	0.5 e	2.0 a	1.5 a	2.0 b
	LLDPE	2.6 a	ND	2.6 c	ND	3.4 a	2.0 a	1.5 a	2.1 ab
	Carton	2.6 a	ND	3.0 b	2.8 a	2.4 b	2.0 a	1.7 a	2.3 a

Means in the same column followed by a different letter are significantly different ($p < 0.05$).
[1]Sensory attribute intensities were scored on a 0 to 15 point universal intensity scale (Meilgaard et al., 2007).
[2]ND = Not Detected

Table 2.7. Trained panel profiles of whole milks filled into different packages with storage time (day 0, 5, 10, 15).

		Sensory Attributes[1]								
Time	Packaging Type	Overall Aroma	Sweet Aromatic	Milkfat	Cooked	Paperboard/Cardboard	Refrigerator Stale	Sweet	Salty	Astringency
Day 0	Glass	2.4 a	1.9 a	3.3 a	3.9 a	ND[2]	ND	2.2 a	1.7 a	1.7 b
	PET	2.3 a	1.8 a	3.3 a	3.8 a	ND	ND	2.2 a	1.7 a	1.7 b
	HPDE	2.3 a	1.6 ab	3.2 a	3.8 a	ND	ND	2.0 a	1.5 a	1.6 b
	LDPE	2.2 ab	1.6 ab	3.2 a	3.8 a	ND	ND	2.2 a	1.7 a	1.7 b
	LLDPE	2.2 ab	1.2 cd	3.3 a	3.6 ab	ND	0.7 d	2.2 a	1.5 a	1.6 b
	Carton	2.3 a	1.4 bc	3.2 a	3.8 a	ND[2]	ND	2.0 a	1.5 a	1.7 b
Day 5	Glass	2.0 b	1.6 ab	3.2 a	3.7 ab	ND	ND	2.0 a	1.7 a	1.7 b
	PET	2.1 ab	1.5 bc	3.3 a	3.6 ab	ND	ND	2.2 a	1.7 a	1.7 b
	HPDE	2.0 b	1.1 cd	3.2 a	3.6 ab	ND	ND	2.3 a	1.5 a	1.8 b
	LDPE	2.0 b	1.1 cd	3.2 a	3.6 ab	ND	ND	2.2 a	1.7 a	1.8 b
	LLDPE	2.2 ab	0.6 e	3.0 a	3.0 c	ND	1.7 c	2.0 a	1.5 a	1.8 b
	Carton	2.0 b	0.9 de	3.2 a	3.4 b	1.2 c	ND	2.0 a	1.5 a	1.8 b
Day 10	Glass	2.1 ab	1.6 ab	3.2 a	3.6 ab	ND	ND	2.0 a	1.5 a	1.8 b
	PET	2.1 ab	1.5 bc	3.3 a	3.5 b	ND	ND	2.2 a	1.7 a	1.8 b
	HPDE	2.1 ab	1.2 cd	3.0 a	3.4 b	ND	ND	2.2 a	1.7 a	1.8 b
	LDPE	2.1 ab	1.0 de	3.2 a	3.4 b	ND	ND	2.0 a	1.7 a	1.9 ab
	LLDPE	2.4 a	ND	3.3 a	3.0 c	ND	2.0 b	2.3 a	1.5 a	2.0 ab
	Carton	2.1 ab	0.6 e	3.0 a	3.4 b	1.7 b	0.5 d	2.0 a	1.5 a	2.1 ab
Day 15	Glass	2.1 ab	1.5 bc	3.2 a	3.7 ab	ND	ND	2.2 a	1.7 a	1.7 b
	PET	2.0 b	1.3 cd	3.3 a	3.6 ab	ND	ND	2.2 a	1.5 a	1.8 def
	HPDE	2.0 b	1.0 de	3.2 a	3.4 b	ND	ND	2.2 a	1.7 a	1.8 def
	LDPE	2.0 b	1.6 ab	3.3 a	3.0 c	ND	ND	2.2 a	1.7 a	1.8 cde
	LLDPE	2.3 a	ND	3.0 a	2.9 c	ND	2.5 a	2.0 a	1.5 a	2.0 ab
	Carton	2.1 ab	ND	3.2 a	2.9 c	2.2 a	1.5 c	2.0 a	1.5 a	2.2 a

Means in the same column followed by a different letter are significantly different ($p < 0.05$).
[1]Sensory attribute intensities were scored on a 0 to 15 point universal intensity scale (Meilgaard et al., 2007).
[2]ND = Not Detected

Table 2.8: Selected volatile compounds in skim milk packaged milk packaged in glass, HDPE, PET, LLDPE, and paperboard carton at 4°C across 4 time points (day 0, 5, 10, 15).

		Volatiles Compounds (µg/kg)[1]									
Container	Day of Shelflife (Time point)	styrene	toluene	p-xylene	p-cymene	hexanal	octanal	nonanal	limonene	acetophenone	2-ethyl-1-hexanol
Glass	0	ND	0.51 ij	ND	ND	1.49 l	ND	0.57 lm	ND	ND	3.09 i
	5	ND	1.11 h	ND	ND	3.26 jkl	ND	1.96 klm	0.72 hi	ND	4.00 i
	10	ND	2.06 ef	0.46 h	ND	10.2 ghij	ND	2.42 ijkl	0.98 efg	ND	4.11 i
	15	ND	1.26 gh	0.40 h	ND	22.1 e	0.92 e	3.86 hijk	0.88 fghi	0.51 h	4.16 i
PET	0	ND	0.44 j	ND	ND	1.35 l	ND	1.58 lm	ND	ND	1.05 i
	5	0.59 h	1.11 h	ND	ND	5.23 ij	ND	2.42 ijkl	0.75 ghi	ND	4.70 hi
	10	ND	0.97 hi	0.44 h	0.41 h	29.7 d	0.79 ef	13.7 e	0.96 fgh	ND	3.7 fg
	15	1.18 gh	1.68 fg	0.44 h	0.42 h	21.2 e	1.06 de	4.58 hi	0.87 fgh	0.72 gh	4.78 hi
HDPE	0	ND	0.97 hi	ND	ND	2.5 kl	0.78 ef	ND	ND	ND	3.72 i
	5	1.00 gh	2.87 d	0.89 f	0.85 f	12.8 fgh	0.93 e	4.28 hij	0.48 k	0.81 gh	15.8 efg
	10	0.99 gh	2.42 de	1.15 e	0.96 f	8.48 ghi	0.87 e	7.12 g	1.30 d	1.07 g	15.4 fg
	15	1.40 fgh	4.51 b	1.92 c	1.71 d	15.3 efg	1.36 d	4.69 h	1.02 ef	1.62 f	13.4 fgh
LDPE	0	ND	1.62 fg	0.85 fg	ND	1.45 l	ND	1.52 lm	0.41 j	ND	1.24 i
	5	0.77 h	2.53 de	1.28 de	0.47 h	8.89 ghi	0.61 f	7.73 g	0.70 i	0.62 h	15.4 fg
	10	1.17 gh	1.91 f	1.39 d	0.68 g	1.61 l	1.44 d	2.05 jklm	1.24 de	1.09 g	24.2 e
	15	2.21 f	4.91 ab	3.16 a	0.91 f	12.9 fgh	1.38 d	10.8 f	1.01 ef	1.66 f	19.8 ef
LLDPE	0	1.75 fg	0.90 hij	ND	ND	1.61 l	ND	4.44 hi	0.40 j	ND	1.06 i
	5	8.93 e	2.77 d	0.62 gh	1.29 e	9.86 ghi	0.44 g	19.1 d	0.89 fghi	3.20 d	36.4 d
	10	12.3 d	2.06 ef	1.38 de	1.90 c	10.9 fghi	ND	9.03 fg	2.19 c	5.65 c	59.1 c
	15	15.3 b	5.29 a	2.99 a	2.88 a	18.4 ef	0.52 g	34.6 a	4.48 a	20.0 a	119 a
Carton	0	0.74 h	0.53 ij	ND	ND	7.17 hijk	ND	0.70 lm	ND	ND	2.81 i
	5	11.7 d	3.42 c	0.78 fg	1.28 e	133 b	4.71a	24.1 c	0.80 fgh	1.57 f	60.3 c
	10	13.9 c	3.67 c	1.27 de	1.87 c	115 c	2.78 b	17.2 d	1.28 d	2.24 e	70.7 b
	15	18.5 a	4.90 ab	2.37 b	2.72 b	159 a	2.22 c	28.8 b	3.78 b	6.54 b	58.3 c

[1]Volatile compounds were quantified using 6 point standard curves ($R > 0.95$)
Means in the same column followed by a different subscript are significantly different ($p < 0.05$)
ND = Not Detected

Table 2.9: Volatile compounds in whole milk packaged milk packaged in glass, HDPE, PET, LLDPE, and paperboard carton at 4°C across 4 time points (day 0, 5, 10, 15).

		Volatiles Compounds (µg/kg)[1]									
Container	Day of Shelflife (Time point)	styrene	toluene	p-xylene	p-cymeme	hexanal	octanal	nonanal	limonene	acetophenone	2-ethyl-1-hexanol
Glass	0	ND	0.82 jkl	ND	ND	0.84 j	ND	2.85 bc	7.74 b	ND	ND
	5	ND	0.98 ijk	ND	ND	1.30 ij	ND	1.24 efg	3.04 cde	ND	0.71 de
	10	ND	1.26 fghi	ND	ND	1.38 ij	ND	ND	2.67 def	ND	0.46 e
	15	ND	1.44 efg	ND	ND	3.26 defgh	ND	ND	2.32 def	ND	0.41 e
PET	0	ND	0.66 lm	ND	ND	2.98 efghi	ND	1.64 de	7.45 b	ND	ND
	5	ND	0.62 lm	ND	ND	3.17 efgh	ND	0.66 fgh	1.69 ef	ND	ND
	10	ND	0.78 jkl	ND	ND	4.11 de	0.505 a	0.75 efgh	2.08 def	ND	0.83 d
	15	ND	0.85 jkl	ND	ND	2.28 fghij	ND	ND	1.65 ef	ND	ND
HDPE	0	ND	0.69 klm	ND	0.484 c	2.14 ghij	ND	0.80 efgh	8.44 b	ND	ND
	5	ND	ND	ND	ND	1.87 hij	ND	ND	1.48 ef	ND	ND
	10	0.69 f	1.04 hij	ND	ND	3.68 defg	ND	0.64 fgh	2.11 def	ND	0.52 de
	15	ND	1.35 efgh	ND	ND	4.89 d	ND	0.57 gh	2.01 def	ND	1.32 c
LDPE	0	ND	1.67 e	ND	ND	2.92 efghi	ND	1.52 def	8.89 ab	ND	ND
	5	0.53 f	1.58 ef	ND	ND	3.34 defgh	ND	0.60 fgh	2.56 def	ND	ND
	10	0.63 f	3.64 b	0.42 e	ND	3.94 def	ND	1.00 efg	3.01 cde	0.46 de	0.45 e
	15	0.47 f	5.09 a	0.95 c	ND	2.57 efghi	ND	0.85 efgh	3.12 cde	ND	ND
LLDPE	0	2.11 e	1.53 ef	ND	0.66 b	3.54 defgh	0.357 b	2.27 cd	9.41 ab	0.98 d	0.62 de
	5	3.39 c	1.18 ghi	0.40 e	0.51 c	2.78 efghi	ND	2.67 c	2.83 def	2.64 c	1.62 c
	10	7.45 b	2.05 d	1.59 b	0.94 a	4.09 de	ND	3.72 b	3.78 cd	6.63 b	8.06 a
	15	12.7 a	2.41 c	2.16 a	0.94 a	3.54 defgh	ND	7.88 a	4.83 c	8.95 a	7.67 b
Carton	0	0.71 f	0.69 klm	ND	ND	6.85 c	ND	1.28 efg	10.8 a	ND	ND
	5	0.88 f	0.45 m	ND	ND	7.07 c	ND	0.87 efgh	0.96 f	ND	0.46 e
	10	2.66 d	1.40 efg	0.60 d	ND	13.2 b	ND	0.69 fgh	1.86 def	1.05 d	0.69 de
	15	3.50 c	1.43 efg	0.62 d	ND	19.2 a	ND	0.99 efg	2.13 def	1.59 d	1.71 c

[1]Volatile compounds were quantified using 6 point standard curves ($R > 0.95$)
Means in the same column followed by a different subscript are significantly different ($p < 0.05$)
ND = Not Detected

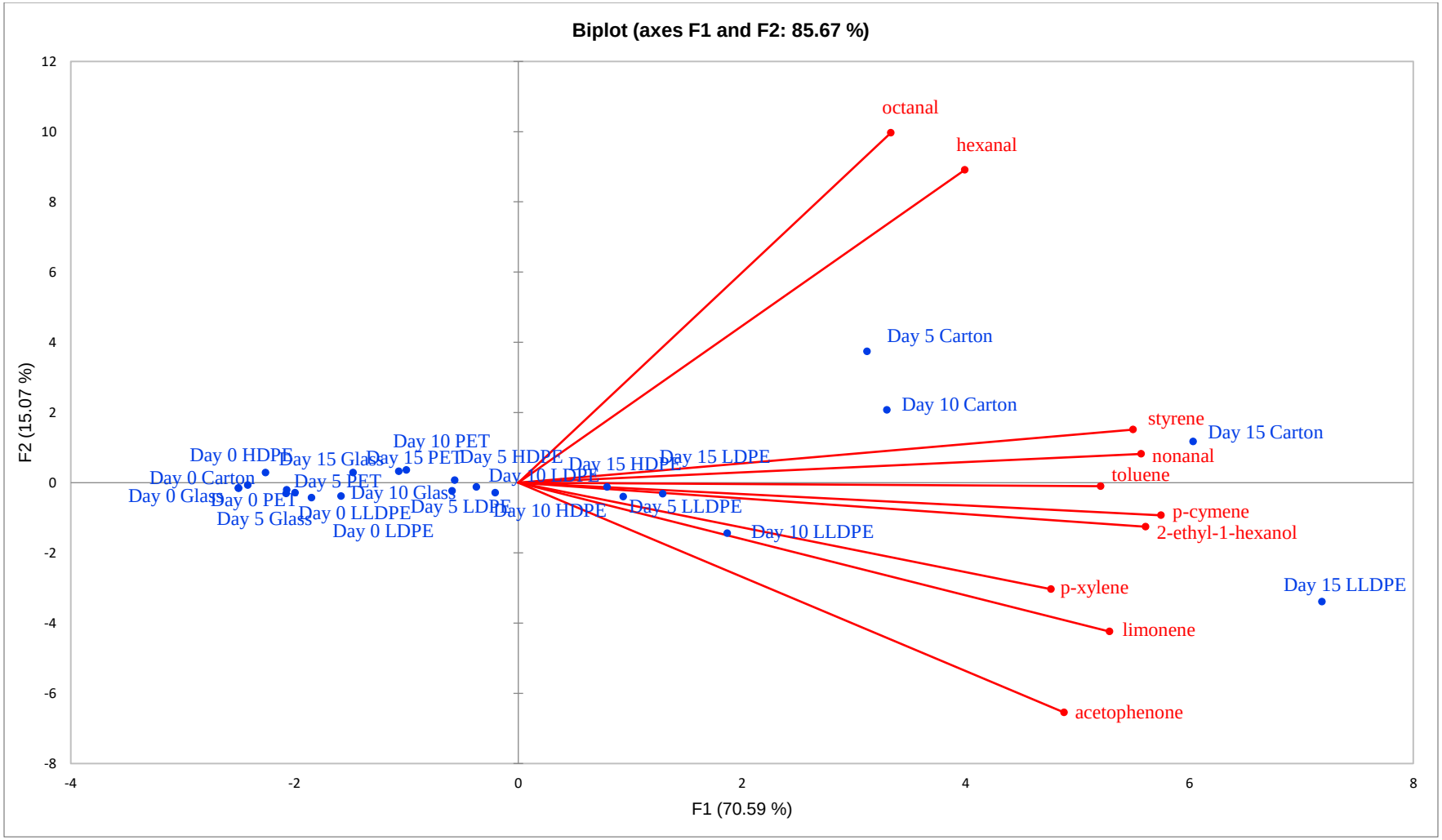

Figure 2.1: Principle component biplot of volatile compound concentrations of skim milk packaged in glass, HDPE, PET, LLDPE, or paperboard cartons at 4°C across 4 time points (day 0, 5, 10, 15).

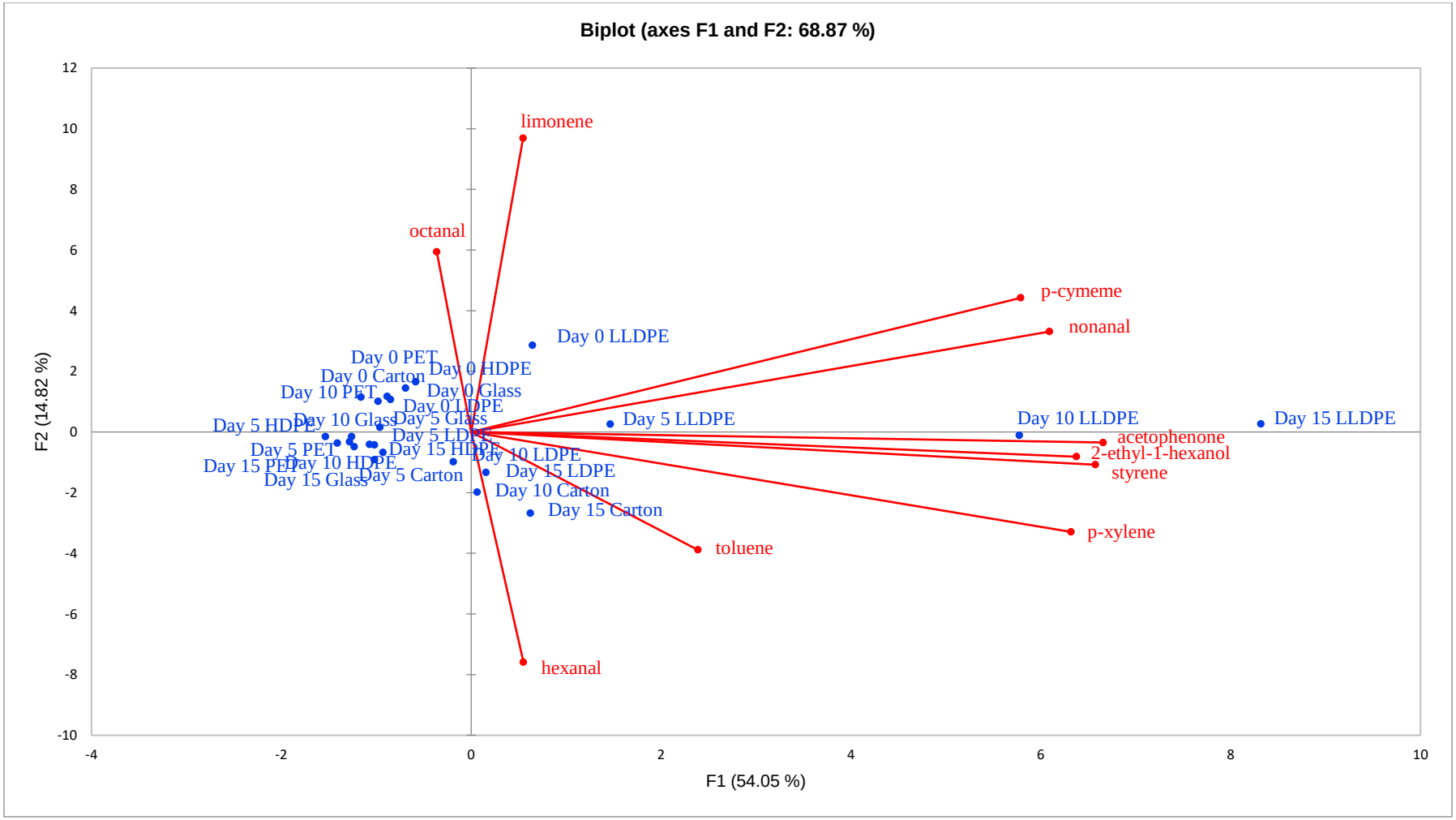

Figure 2.2: Principle component biplot of volatile compound concentrations of whole milk packaged in glass, HDPE, PET, LLDPE, or paperboard cartons at 4°C across 4 time points (day 0, 5, 10, 15).

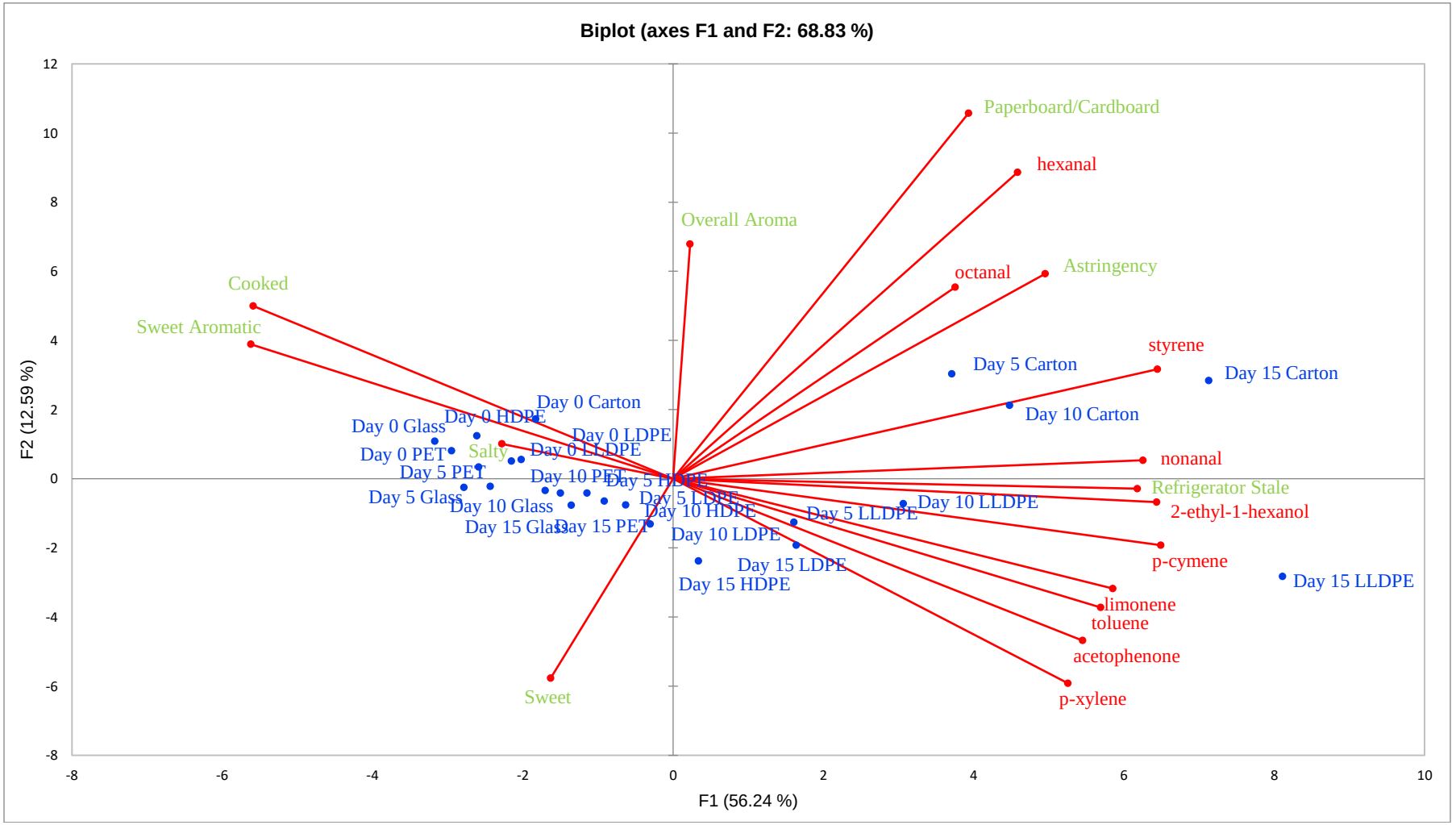

Figure 2.3: Principle component biplot of volatile compound concentrations and trained panel sensory attribute intensities of skim milk packaged in glass, HDPE, PET, LLDPE, or paperboard cartons at 4°C across 4 time points (day 0, 5, 10, 15).

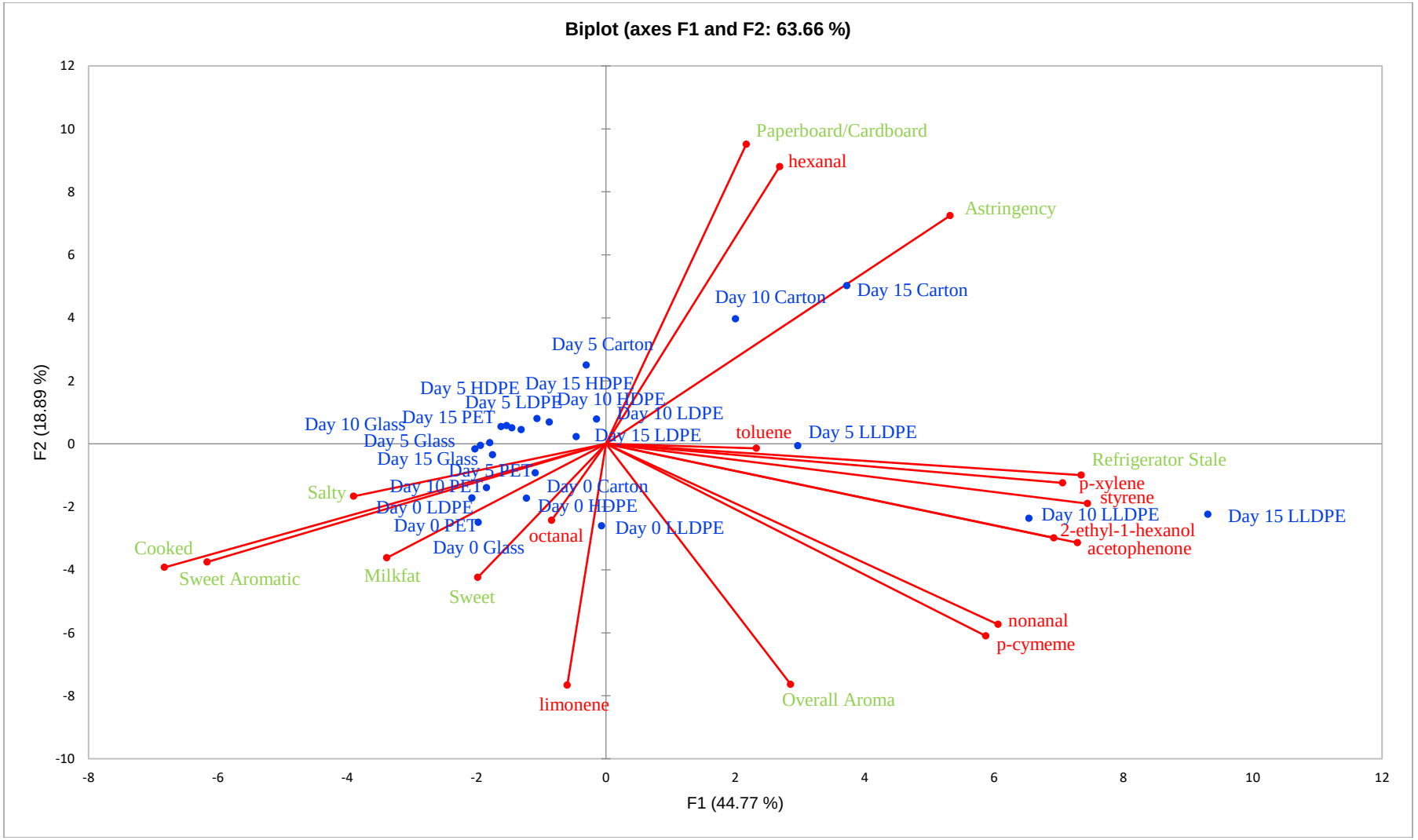

Figure 2.4: Principle component biplot of volatile compound concentrations and trained panel sensory attribute intensities of whole milk packaged in glass, HDPE, PET, LLDPE, or paperboard cartons at 4°C across 4 time points (day 0, 5, 10, 15).

Table 2.10. Consumer tetrad difference tests of skim milk in three packaging types (paperboard, PET, and HDPE) compared to the control (Glass) after 10 days at 4°C.

		Fluid Skim Milk	
Packaging Comparisons		No. Correct	Significant at $\alpha = 0.05$
Paperboard Carton vs. Glass (Control)	n=101	56	Yes
PET vs. Glass (Control)	n=103	28	No
HDPE vs. Glass (Control)	n=103	36	No

Table 2.11. Consumer tetrad difference tests of whole milk in three packaging types (paperboard, PET, and HDPE) compared to the control (Glass) after 10 days at 4°C.

		Fluid Whole Milk	
Packaging Comparisons		No. Correct	Significant at $\alpha = 0.05$
Paperboard Carton vs. Glass (Control)	n=104	47	Yes
PET vs. Glass (Control)	n=103	39	No
HDPE vs. Glass (Control)	n=103	37	No