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THE EFFECT OF PASTEURIZATION TEMPERATURE ON CONSUMER ACCEPTABILITY, SENSORY CHARACTERISTICS, VOLATILE COMPOUND COMPOSITION, AND SHELF-LIFE OF FLUID MILK

By

April Lynne Gandy

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in the Department of Food Science, Nutrition and Health Promotion

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THE EFFECT OF PASTEURIZATION TEMPERATURE ON CONSUMER ACCEPTABILITY, SENSORY CHARACTERISTICS, VOLATILE COMPOUND COMPOSITION, AND SHELF-LIFE OF FLUID MILK

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The relationship among consumer acceptability, descriptive sensory attributes, and shelf-life was determined for 2 % milk pasteurized at 77, 79, 82, or 85°C. Pasteurization temperature had no effect ($p>0.05$) on shelf-life. Consumers preferred ($p<0.05$) 79°C over other treatments on day 0; however, six days post-pasteurization 79°C milk was only preferred ($p<0.05$) over 77°C. Consumers were grouped into eight clusters based on product liking for both day 0 and 6 evaluations. The largest cluster liked all pasteurization treatments, and 79°C was highly acceptable to all consumers that liked milk. Similar sensory descriptors indicated the end of shelf-life for all pasteurization treatments even though treatments could be differentiated by descriptors

on day 0. This research reveals that altering pasteurization temperature from 79°C may cause a decrease in consumer acceptability to some consumers. Altering pasteurization temperature does not affect shelf-life or sensory descriptors and volatile compound profiles at the end of shelf-life.

DEDICATION

This work is dedicated to my family in honor of the support, prayers and encouragement they have given me as I have pursued my education. Thank you for demonstrating dedication and a solid work ethic, showing me the importance of a good education, instilling in me a love of learning, and, for the most part, patiently putting up with my inquisitive nature.

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Vi Jackson, Julie Wilson, and Dr. Youngmo Yoon are offered my deepest gratitude for their assistance with this project. Thank you all for your willingness to assist me any way you could, your positive attitudes, your patient explanations, and your contribution of personal knowledge as well as reference materials and additional resources. I feel that you made it possible for us to conduct all of the components of this research.

I am thankful that during my time as a graduate student I was given the opportunity to learn life lessons from Joseph Andol and Dr. Mike Martin. You both share a passion for working with students, and the students notice and appreciate it. One common concept you both try to ingrain in students is the importance of treating others with respect, and I will carry your motto "praise in public, correct in private" with me throughout my career.

Appreciation is given to David Hall and the Custer Dairy Processing Plant at Mississippi State University for assistance with this project. Sincere thanks are also extended to the staff, including Mary Andol, Donna Bland, and Coy Farley, in the Department of Food Science, Nutrition and Health Promotion.

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CHAPTER I

INTRODUCTION

There has been increased interest in recent years in raising the minimum standard temperatures for fluid milk pasteurization. This interest is partly due to microbiological concerns, such as reports that the current pasteurization temperatures may not completely inactivate *Mycobacterium paratuberculosis*. *Mycobacterium paratuberculosis* has been linked to Crohn's disease in humans, although there have been conflicting reports and research efforts are ongoing (McDonald et al., 2005; Stabel et al., 1997). While the dairy industry has a low incidence of foodborne illnesses (Stabel et al., 1997), the industry needs to maintain consumer confidence that appropriate measures are in place to adequately safeguard the food supply (Boor, 2001). The interest in higher pasteurization temperatures also comes from a desire in the industry to increase the shelf-life of fluid milk so that the product may be more competitive in the beverage industry (Chapman et al., 2001).

While it has been reported (Erba et al., 1997; White, 2005) that many processing plants already operate at temperatures well above the minimum standards, little work has been published to date describing the changes in sensory attributes or consumer acceptance of fluid milk that is pasteurized at higher temperatures. The shelf-life of the bottled fluid milk in the United States is reported to average between 10 to 21 d when stored at 4-8°C (Allen and Joseph, 1985; Chapman, et al., 2001). The shelf-life varies depending on raw milk quality, processing conditions, microbial growth, packaging materials, temperature abuse, and exposure to light (Handbook of Dairy Processing, 2000; Simon and Hansen, 2001). In research performed on chocolate milk, Douglas and others (2000) found that the use of extended pasteurization conditions (78°C for 16-30 s), coupled with decreased postpastuerization contamination, enabled commercial fluid milk processors to achieve 15 to 25 d refrigerated shelf-life (6°C). The dairy industry is able to achieve 45 d shelf-life through the use of ultra-pasteurization (UP) processes; however, this method of heat treatment imparts a strong cooked flavor in the milk which consumers, especially children, may find undesirable (Chapman and Boor, 2001). Due to milk's naturally mild, slightly sweet flavor, the development of any off-flavors is particularly noticeable in the product. There are many causes of off-flavors, including feed sources, post-pasteurization contamination, artificial or natural light exposure, storage temperature, and packaging materials. Researchers have concluded that the development of objectionable flavors in pasteurized milk is generally a result of bacterial growth (Simon and Hansen, 2001), and unpleasant aromas in fluid milk are characteristic of milk spoilage (Hayes et al., 2002). Little work was encountered in our literature search that describes the effect that changing HTST pasteurization temperature has on sensory attributes of the product as perceived by consumers. There is an apparent need in the industry for studies on HTST pasteurized fluid milk similar to the work performed by Chapman and others (2001). These researchers characterized the sensory attributes of ultrapasteurized milk through quantitative descriptive analysis and principal components analysis.

Researchers have utilized multiple extraction techniques, including static headspace sampling, dynamic headspace, purge and trap, and solvent-assisted flavor evaporation (Christensen and Reineccius, 1992; Contarini et al., 1997; Bendall, 2001; Simon and Hansen, 2001; Toso et al., 2002) in pursuit of identification and quantitative measurement of volatile compounds responsible for off-flavors in bovine milk. Christensen and Reineccius (1992) reported that it is generally recognized that the heated milk off-flavor is due to an increase in the concentration of sulfur compounds. However, in their analysis of heated milk using static headspace sampling methods, Christensen and Reineccius (1992) found that sulfur volatile compounds reached maximum levels after moderate heat treatments and then remained the same or decreased following more severe heat treatments. Contarini and Povolo (2002) studied the effect of heat treatments on volatile compounds in commercially processed milk samples using HS-SPME and gas chromatography (GC). These researchers identified 11 compounds, most of which belonged to the ketone family. Compounds with a higher molecular weight, such as 2-pentanone and 2-undecanone, increased in direct correlation with more severe heat treatments. Among the volatile compounds identified, 2-heptanone was determined by Contarini and Povolo (2002) to have the most potential as a marker for severity of heat treatment.

Preference mapping refers to a group of methods that are used to relate descriptive sensory and consumer data (Carpenter et al., 2000). Preference mapping is frequently used to understand the underlying sensory attributes revealed by descriptive analysis that push consumer preferences (Thompson et al., 2004). Preference mapping may help explain the relationship between consumer acceptability and sensory descriptors of 2 % fluid milk pasteurized at varying temperatures.

The primary objective of this research was to determine the effect of pasteurization temperature on the consumer acceptability and shelf-life of fluid milk. The second objective of this research was to utilize descriptive analysis to characterize differences in fluid milk due to increased pasteurization temperatures over storage time. The third objective of this research was to perform instrumental analysis to identify the volatile compounds present in milk pasteurized at different treatment temperatures and variable storage times. The final objective of this research was to relate consumer acceptability data to sensory descriptors, volatile compounds, and shelf-life as determined by dairy judges using principal components analysis and preference mapping.

CHAPTER II

LITERATURE REVIEW

History of Fluid Milk

Milk production may have begun over 6000 years ago, but it was French chemist Louis Pasteur's discovery in 1857 that heating wine postponed spoilage which revolutionized the milk industry (Handbook of Dairy Processing, 2000). Commercial milk processing and packing operations quickly followed the invention of heating methods that are now referred to as pasteurization (Erba et al., 1997). New mechanical innovations in the late 1880's – early 1900's improved the safety and efficiency of the industry. Automatic filling and capping equipment was introduced in 1886 and was refined for large scale use by 1911. Significant increases in efficiency were again experienced during the period between 1930 and 1950 as high temperature-short time (HTST) continuous flow pasteurization replaced batch pasteurization as the primary heating method used in fluid milk treatment and bottling. Since that time, further gains in productivity have been realized with the development of new packaging methods and materials, clean-in-place (CIP) systems, automated stackers, conveyors and palletizers (Erba et al., 1997).

Goff and Griffiths (2006) stated that efforts by dairy researchers and technologists to further improve mechanization, automation, quality, safety and new product development were at an “all-time high”. These authors attributed part of the success of the milk industry in recent years to consolidation. In 1977, there were 1,924 plants in the United States, producing an average of 13.2 million liters of fluid milk. By 1997 there were only 612 plants. However, during this 20 year span, fluid milk production increased to an average of 43.8 million liters per year. Milk processing plants are now capable of producing up to 200,000 L/h (Goff and Griffiths, 2006).

The consumption of fluid milk has declined somewhat in the United States, following a trend that can also be seen in countries such as Australia and Canada. Per capita consumption of fluid milk decreased by 20L during the last 25 years, to a rate of 90 L in 2003. During the same time period, within this division of the dairy industry, there have been significant changes in product purchases based on fat content. Today, 65% of fluid milk consumed is reduced-fat or lowfat milk, which is a notable change from only 14% low-fat milk consumption 25 years ago (Goff and Griffiths, 2006).

Fluid Milk Processing

The finished product of fluid milk processing will only be as good as the raw material used and the attention paid to proper sanitation and handling during the entire process of bottling and transportation of the product. Fresh milk from healthy cows is generally considered free from bacteria. However, milk is a desirable medium for many microorganisms and the risk of contamination is present at every step in the process (Handbook of Dairy Processing, 2000).

Milk is usually transported from dairy farms to processing plants via bulk trucks or tractor-trailers. Raw milk can not be held for more than 72 hours without processing, and it must be held at or below 7°C. During the HTST process, milk is passed through a series of heat exchange plates, which heats the milk to a minimum temperature of 72°C for 15 s (21 CFR 113). It is not uncommon for the product to be heated to 79-81°C for 15-18 s (Erba et al., 1997; White, 2005). A temperature transmitter and flow diversion valve are used in the line to return milk that has not been sufficiently heated to the balance tank. During or prior to pasteurization, a separator removes milkfat from the skim milk portion. The cream portion may be added back to the skim milk portion or used in the manufacture of other products, such as butter or ice cream. The purpose of this standardization is to allow for a defined fat content. The regulations for fat content vary by country (Handbook of Dairy Processing, 2000). In the United States, the fat contents are set at greater than or equal to 3.25% for whole milk, 1.5 or 2% for reduced fat, 0.5 or 1% for low fat, and less than 0.5% for skim milk (Pasteurized Milk Ordinance, 2001). Once the in-line standardization step is complete, a homogenizer is utilized to break down milkfat particles, which prevent the separation of cream from the skim portion of milk in the finished product. Pasteurized milk storage tanks hold the product at or below 7°C until it is pumped to the filling and packaging equipment (Handbook of Dairy Processing, 2000; Erba et al., 1997).

The shelf-life of milk is considered the length of time from packaging until the product becomes unacceptable to the consumer. The shelf-life of the bottled fluid milk in the United States is reported to average between 10 to 21 d when stored at 4-8°C (Allen and Joseph, 1985; Chapman et al., 2001). The shelf-life varies depending on the quality of the raw milk, processing and packaging conditions, microbial growth, packaging materials, and temperature or light abuse (Handbook of Dairy Processing, 2000; Simon and Hansen, 2001).

Descriptive Sensory Analysis

Descriptive sensory analysis is the evaluation of a product's perceived attributes by a panel of trained evaluators. Descriptive analysis is used to describe both the qualitative (attributes of product) and quantitative (intensity of attributes) characteristics of the evaluated product. The use of descriptive analysis has rapidly expanded in recent years and is expected to continue to grow in coming years (Murray et al., 2001). There are several defined methods of descriptive analysis, as discussed by Meilgaard et al., (1991), Murray et al., (2001) and others. Perhaps the most frequently discussed methods include the Flavor Profile Method, Quantitative Descriptive Analysis (QDA)®, and the Spectrum Method™. Each method has benefits and limitations for use by sensory scientists (Meilgaard et al., 1991). In addition to the established methods, sensory analysts may utilize generic descriptive analysis techniques, which allow the researcher to combine components of different descriptive analysis methods to best fit the practical applications (Murray et al., 2001). One common thread among all descriptive analysis methods is the importance of selecting appropriate panelists and providing adequate training for the panelists. Descriptive panelists should be familiar with the product and its origins, able to discriminately judge differences in product(s), motivated to serve on the panel and fit the group climate (Meilgaard et al., 1991). The required hours of training for panelists may differ based upon product complexity, quantity of products for analysis, selected descriptive method, and panelists' prior training. A useful tool for training panelists is the use of references or standards. References allow panelists to establish scales of intensity for appearance, odors, tastes, mouthfeel and textural properties (Meilgaard et al., 1991; Stampanoni, 1997). Civille and Lawless (1997) recommend the use of several reference samples when possible to demonstrate the range of product intensities for each attribute or concept.

Score sheets or evaluation forms are usually customized for each descriptive evaluation panel. The descriptive panel generally develops their own sensory language to describe all the attributes of the product. However, an existing language or lexicon may also be adopted by the panel, as long as care is taken to ensure that panelists understand and identify all terms and standards (Murray et al., 2001). Three types of scales are commonly used in descriptive analysis. The first type is known as category scales, which are limited sets or words with equal intervals between categories, for example a scale from 0 to 9. The second type is line scales. Line scales are constructed along a 15 cm long line upon which the panelist places a mark. The third type is magnitude estimation (ME) scales, which are mostly used for academic research purposes. Using this scale, a panelist assigns a number to the first sample tested, and then all subsequent numbers are assigned in proportion to the first sample (Meilgaard et al., 1991).

Sensory Descriptors for Milk

Sensory Defects in Milk

Consumer acceptance and preference of milk is influenced primarily by its flavor, according to Thomas (1981), as cited by McSweeney et al. (1997). Due to milk's naturally mild, slightly sweet flavor, the development of any off-flavors is particularly noticeable in the product. There are many causes of off-flavors, including feed sources, post-pasteurization contamination, artificial or natural light exposure, storage temperature and packaging materials. However, researchers have concluded that the development of objectionable flavors in pasteurized milk is generally a result of bacterial growth (Simon and Hansen, 2001), and unpleasant aromas in fluid milk are characteristic of milk spoilage (Hayes et al., 2002). Gruetzmacher and Bradley (1999) reported that filling machines and improperly sanitized pasteurizers are the two most common sources of post-pasteurization contamination.

The sensory defects in milk are greatly influenced by the source of off-flavor or aroma development. A slight cooked flavor is normal for the majority of milk available in today's marketplace, since milk is heated. The time and temperature of pasteurization conditions determine the degree of cooked flavor. The flavor of high quality raw milk pasteurized at minimum standards (72°C for 15 s) will be minimally affected. As heat treatment increases, so do the changes in flavor (McSweeney et al., 1997). Severe heat treatments result in aromas characterized as scorched, caramelized, and sulfurous (Shipe et al., 1978). Shipe et al. (1978) categorized the cooked flavors into four types: cooked or sulfurous, heated, caramelized, and scorched. Compounds including diacetyl, lactones, methyl ketones, vanillin, benzaldehyde, and hydrogen sulfide are responsible for the cooked flavor and aroma (Shipe et al., 1978). Feed or weedy flavors may appear in fluid milk depending on the type of feed consumed, time consumed prior to milking, and the geographical region of the animals (Bodyfelt et al., 1988). In addition to influencing the aroma and taste, different types of feed obviously result in different volatile compounds within the product. For example, wild onion and garlic will yield unclean flavors caused by benzyl compounds, while grass or corn silage impart methyl sulfide, aldehydes, alcohols, ketones, and simple esters (Marsili, 2007). The hydrolysis of short chain triglycerides to free fatty acids by lipases, which naturally occur in milk and are also produced by psychotropic bacteria, results in a rancid flavor in milk (Bodyfelt et al., 1988). The sensory defect of oxidation may be the result of more than one factor, yet it is often characterized as cardboardy, oily or painty. Exposure to ultraviolet light may result in flavors described as light-activated flavors, burnt, or sunlight flavor. Marsili (2007) has proposed that lipid oxidation forms aldehydes such as pentanal, hexanal, ketones, and alcohols. Marsili (2007) also reports that metals such as copper, iron, and nickel that are sometimes present in processed dairy products, may accelerate the rate of oxidation. "Microbial flavors" is a term used to describe off-flavors and aromas in milk that result from undesirable organisms and enzymes, such as the sour flavor characterized by acetic and propionic acids (Shipe et al., 1978).

ADSA Scorecard

The American Dairy Science Association (ADSA) Milk Scorecard (Bodyfelt et al., 1988) has traditionally been used by expert judges to score defects in milk. The dairy industry often uses grading techniques, such as the ADSA scorecard method, to evaluate the sensory quality of the product for quality control purposes. Unlike descriptive evaluation, which uses simple terms to identify all attributes of a product, dairy judging involves the use of defect-oriented terms that may be complex and often refer to the root cause of the observed sensory experience (Claassen and Lawless, 1992). Thus, Claasen and Lawless reported that the traditional dairy judging system has been criticized by some members of the sensory evaluation field and food industry. However, the traditional dairy judging method has served as a cost efficient tool for the industry for many years (Claassen and Lawless, 1992). Using the ADSA scorecard method, dairy judges quickly evaluate the flavor, appearance and body of milk samples, noting observed attributes on a scale of 0-10. A score of 5 or lower on the ADSA scorecard (Figure A3, Appendix) indicates that the evaluated sample has an objectionable off-flavor, while a 10 would be assigned to a product free of defects. A few of the 21 flavor criticisms on the scorecard include: acid, astringent, bitter, cooked, cowy, feed, fermented/fruity, flat, malty, rancid, and unclean.

Volatile Flavor Compounds in Fluid Milk

The development of off-flavors in milk, which consumers expect to have a mild, delicate flavor, leads to rejection of the product. Researchers have utilized multiple extraction techniques, including static headspace sampling, dynamic headspace, purge and trap, and solvent-assisted flavor evaporation (Christensen and Reineccius, 1992; Contarini et al., 1997; Bendall, 2001; Simon and Hansen, 2001; Toso et al., 2002) in pursuit of identification and quantitative measurement of volatile compounds responsible for off-flavors in bovine milk. The usefulness of the traditional extraction techniques is often limited, however, by the extensive analysis time or sample size required (Vazquez-Landaverde, et al., 2005). Christensen and Reineccius (1992) pointed out that in order for an instrumental method to be useful, it must be rapid, reproducible, well correlated with sensory scores, and applicable to the conventionally encountered off-flavors in milk. Therefore, there has been increasing interest in recent years in the relatively new extraction method known as solid-phase microextraction (SPME).

Pawliszyn and co-workers (1997) first developed solid-phase microextraction to address the need for more rapid sample preparation. The introduction of a commercial SPME device by Supleco in 1993 further facilitated advancement of the technique. Currently, there are greater than 10 different fiber types commercially available for specific applications. Marsili (1999) stated that Carboxen-polydimethylsiloxane was able to "detect parts per billion levels of pentanal, hexanal and heptanal produced in light irritated milk." Due to its solvent-free nature, SPME can be used with various analytical instruments, although the standard gas chromatograph (GC) is most frequently employed (Pawliszyn, 1997).

Sample preparation is simplified with the SPME technique. Salt concentration and sample pH control may be added to the sample in order to enhance extraction (Pawliszyn, 1997). Pawliszyn (1997) stated that the SPME process may be thought of as a two-step process: 1) separation of analytes between the coating and the sample matrix and 2) extract desorption into an analytical instrument. The coated fiber is exposed to the sample or its headspace in the first step, at which point the target analytes partition from the sample matrix into the coating. In the second step, the fiber is injected in an instrument for desorption, and segregation and quantitation of compounds occur (Pawliszyn, 1997).

Contarini and others (1997) coupled dynamic headspace capillary gas chromatography with multivariate statistical techniques to distinguish milk samples of three different heat treatments (pasteurization, direct ultrahigh-temperature method, and "in-bottle" sterilization). The researchers also studied the influence of storage time and temperature on whole and partially skimmed UHT milk. The most abundant class of volatile compounds identified by the researchers was ketones, such as acetone, 2-butanone, 2-pentanone, and 2-heptanone. The aldehydes detected included pentanal, hexanal, and heptanal (formed by autooxidation of unsaturated fatty acids) and 3-methylbutanal, which the authors attributed to the nonenzymatic browning reaction of leucine. Pentanal, hexanal, and heptanal concentration showed an increase in partially skimmed UHT milk during storage time, although this was not true of whole UHT milk. Dimethyl disulfide was the only sulfur compound detected, and its presence may be the result of oxidation of methanethiol in heat-treated milk. Contarini and co-workers (1997) also identified toluene and limonene in the study, although they were unable to find references about the influence of heat treatment on the concentration of these compounds.

Christensen and Reineccius (1992) reported that it was generally recognized that the heated milk off-flavor was due to an increase in sulfur compounds such as dimethyl sulfide and hydrogen sulfide. In their analysis of heated milk using static headspace sampling methods, they found that sulfur volatile compounds reached maximum levels after moderate heat treatments and then remained the same or decreased following more severe heat treatments. Therefore, the authors concluded that these sulfur compounds have limited usefulness in a quality control setting.

Contarini and Povolo (2002) studied the effect of heat treatments on volatile compounds in commercially processed milk samples using HS-SPME and gas chromatography (GC). They identified 11 compounds, most of which belonged to the ketone family. Compounds with a higher molecular weight, such as 2-pentanone and 2-undecanone, increased in direct correlation with more severe heat treatments. Among the volatile compounds identified, 2-heptanone was determined by Contarini and Povolo to have the most potential as a marker for severity of heat treatment.

Vazquez-Landaverde et al. (2005) developed a headspace solid-phase microextraction (HS-SPME)/gas chromatographic method for quantitative analysis of thermally derived off-flavor compounds. They concluded that their technique demonstrated significant potential for rapid quantitative analysis of milk volatiles “due to its accurate determination of the compounds of interest, the simple steps, and short time required for extraction and analysis.” In their studies, Vazquez-Landaverde et al. (2005) determined time to be the most significant factor affecting sensitivity. They determined that increasing the extraction time up to 3 h improved milk volatile extraction, although they used 1 h extraction times, citing productivity limitations. Vazquez-Landaverde et al. (2005) found no significant effect on volatile compound extraction from milk based on sample size. The researchers identified 35°C as the optimal temperature for extraction, stating that extraction temperatures of 45-75°C used in other studies (Contarini and Povolo, 2002; Toso et al., 2002, Simon and Hansen, 2001) may result in artifact formation. Vazquez-Landaverde et al. (2005) quantified 20 volatile compounds in raw, pasteurized, and UHT milk samples with various fat contents. Ketone concentrations were not significantly different in raw and pasteurized milk, but their concentrations were substantially higher in UHT milk. Based on their results, 2-heptanone and 2-nonanone were not important aroma contributors in raw and pasteurized milk samples. Total amount of aldehydes, including nonanal, decanal, and hexenal, were less affected by heat treatment than ketones. However, aldehydes were determined to contribute to the aroma of heated milk. Ethyl acetate was the only ester and 3-methylbutanal was the only alcohol quantified (Vazquez-Landaverde et al., 2005).

Marsili (1999) developed a technique for utilizing solid-phase microextraction, mass spectrometry, and multivariate analysis to study off-flavors in reduced-fat milk abused by light, heat, copper and microbial contamination. He reported that this method was rapid and advantageous over other electronic nose instruments used in quality control settings.

The primary advantages of the SPME technique include the fact that it is solvent free, it enables rapid sample preparation, it serves a wide range of applications, and the sensitivity has been reported to be as great as or greater than other techniques with longer preparation time (Contarini et al., 1997; Pawliszyn, 1997).

Preference Mapping

Preference mapping refers to a group of methods that are used to relate sensory and consumer data (Carpenter et al., 2000). Preference mapping is frequently used to understand the underlying sensory attributes revealed by descriptive analysis that drive consumer preferences (Thompson et al., 2004). Preference mapping can aid new product development by ensuring optimization of formula and process prior to product launch and discovering key market segments (Helgesen et al., 1997). Preference mapping also enables market researchers to identify different consumption patterns (Helgesen et al., 1997). Preference mapping may be of use to the dairy industry in helping explain the relationship between consumer acceptability and sensory descriptors of 2 % fluid milk pasteurized at varying temperatures.

There are two main categories of preference mapping: internal preference mapping and external preference mapping. Internal preference mapping is the simpler method (Carpenter et al., 2000) and only uses consumer data. Therefore, internal preference mapping is often used when only consumer data are available (Helgesen et al., 1997). However, results from internal preference mapping may be related to other types of data, such as descriptive profiling attributes (Helgesen et al., 1997; Carpenter et al., 2000). In external preference mapping, sensory profiling data are first analyzed, often through principal components analysis (PCA), and then consumer responses are overlaid on the external map (Helgesen et al., 1997).

Both types of preference mapping techniques have been used with a wide variety of products, including commercial chocolate milks (Thompson et al., 1997), dry fermented lamb sausages (Helgesen et al., 1997), Cheddar cheese (Casapia et al., 2006; Drake et al., 2001; Murray and Delahunty, 2000), pear fruit leather (Huang and Hsiesh, 2005), and white corn tortialla chips (Meullenet et al., 2002).

Cluster Analysis

Preference mapping enables researchers to identify groups of consumers who share similar responses and differ from other groups in variables such as responses to product attributes, consumption patterns, viewpoints, and demographic information (Westad et al., 2004). Cluster analysis is one technique available to identify such segments. In other words, "cluster analysis is a method for clustering observations (products) into different groups (Carpenter et al., 2000; Schilling and Coggins, 2007)."

Cluster analysis allows researchers to explore data sets and potentially summarize the data in small groups of consumers with similar preferences, attitudes, habits, demographics, etc., to each other and dissimilarity to other groups (Everitt et al., 2001).

There are a variety of cluster analysis methods, including agglomerative hierarchical clustering. Schilling and Coggins (2007) point out that agglomerative hierarchical clustering is seldom employed apart from preference mapping in published sensory evaluation research. However, the authors conclude that clustering is a viable technique "that often improves the interpretation of hedonic scaled consumer data".

The benefits of classification include easier understanding of large data sets, more efficient information retrieval and identification of patterns of similarity and dissimilarity (Everitt et al., 2001). As an example of the usefulness of cluster analysis for the food industry, market research may reveal a niche market for a new product. Westad et al. (2004) point out that "the identification of possible consumer segments is an important area for strategic product development."

While in most instances cluster analysis is used to partition data so that each object belongs to only one cluster, it is possible for overlapping clusters to occur, as well as no justifiable grouping (Everitt et al., 2001). Jones (1997) cautions that although there are many "potential applications", cluster analysis "is not universally applicable and requires some skill both in application and interpretation."

Principal Components Analysis (PCA)

Principal Components Analysis (PCA) is commonly used in the field of sensory evaluation. PCA is a multivariate statistical analysis technique that allows researchers to simplify complex data sets, such as the results of descriptive analysis, and simplifies the dependent variables into a new, smaller set of underlying variables. (Lawless and Heyman, 1998; Jones, 1997). As explained by Lawless and Heyman (1998), “The first principal component accounts for the maximum amount of variance among the samples” and each “subsequent principal component accounts for successively smaller amounts of total variance in the data set.” Essentially, PCA accounts for correlation among multiple attributes (or variables) in complex data sets and reduces them to smaller data sets more suitable for further analysis (Popper et al., 1997; Jones, 1997).

Results of Principal Components Analysis are frequently represented graphically in the form of biplots that are a combination of the first three principal components, such as can be seen in the work of Drake et al. (2001).

CHAPTER III

MATERIALS AND METHODS

Fluid Milk Processing

Fluid milk used in this study was reduced fat (2% fat), homogenized, pasteurized milk (Mueller Accu-therm Plate Exchanger, Model A120BF, Springfield, MO) from the Mississippi State University dairy processing plant (Starkville, MS). In order to minimize variation in milk quality among samples, one filler head was used to fill the plastic half-gallon containers. Four pasteurization temperatures were utilized in this study (77°C, 79°C, 82°C and 85°C), and each treatment temperature was held for 15 s. Milk samples were stored in a $7^{\circ}C \pm 1^{\circ}C$ cooler. The storage temperature was monitored and recorded daily.

Shelf-Life/Expert Judges

Expert judges skilled in the use of the American Dairy Science Association (ADSA) milk scorecard (Figure A3, Appendix) were utilized for shelf-life determination throughout the course of this study. The judges began evaluations of the milk 7 d after pasteurization and continued tasting each sample daily until the end of shelf-life was reached for all samples. The shelf-life of each sample was considered to be one day prior to when judges scored it unacceptable from a sensory standpoint.

Unacceptable samples were ones that would ordinarily be given a value of 5 or lower on the ADSA scorecard. Therefore, if a sample was judged unacceptable on 15 d, the shelf-life for that sample was recorded as 14 d. For the first three replications, expert judges tasted three samples for each treatment from previously unopened jugs. For the second phase of the research (reps 4-6), judges opened two containers of fluid milk per treatment temperature (77°C, 79°C, 82°C and 85°C). A third plastic jug was opened in the event that one sample of a treatment temperature was scored as objectionable (a score of 5 or below) while the other sample of the treatment temperature was still acceptable, which is a practice commonly used in the dairy industry (White, 2005). Once two or more samples of each treatment were scored at 5 or below, the treatment was considered to have passed its shelf-life.

Consumer Acceptability

Consumer panels were conducted on 0 d (day of pasteurization) and 6 d for all six replications in this study. All consumer evaluations were performed at Garrison Sensory Evaluation Laboratory, Mississippi State University. The primary investigators were certified by the Institutional Review Board of Regulatory Compliance and all test procedures were in compliance with human subject testing regulations. Participants were recruited from the department, University and surrounding community. Panelists evaluated the four temperature treatments plus a control (79°C) for a total of 5 milk samples. All samples for consumer tests, as well as descriptive and instrumental analysis, were poured in dim lighting. Consumers received 1 oz. of milk sample in hot/cold insulated cups with snap on lids (Dart Container Corporation, Mason, MI). Samples were labeled with random 3-digit numbers and the order of samples was randomized on the score sheets. Panelists were asked to expectorate and rinse their mouths with water (Mountain Spring Water, Blue Ridge, GA) between each sample. The score sheets (Figure A1) directed panelists to evaluate the milk samples on the attributes of "flavor" and "overall liking" using a 9-point hedonic scale (Meilgaard et al., 1991). A minimum of 50 panelists performed the evaluations each test day.

Descriptive Sensory Analysis

Eight panelists were trained in descriptive evaluation of fluid milk attributes over a 2 month period (~35 hrs). The panelists ranged in age from mid-20's to early-40's, and the gender ratio was balanced. All panelists were recruited from the Department of Food Science, Nutrition and Health Promotion, Mississippi State University, and were selected based on availability, willingness to participate and prior experience on trained panels.

Evaluations were performed via round table in a temperature controlled descriptive analysis room separated from the preparation area. Commercially available fluid milk as well as food and chemical references were used for panelists' training. Each training session lasted approximately one hour. Panelists evaluated samples and discussed their sensory descriptors. During training sessions, panelists generated four aroma and seven flavor attributes (Table 1) to be included on the score sheet. The attributes were scored on a 15-point numerical intensity scale

(Meilgaard et al., 1991) where 0="none" and 15="extreme" (Figure A2, Appendix). In addition to the established attributes, unlabeled intensity scales allowed assessors to write in other perceived aromas or flavors as detected. In order to minimize variance, samples for descriptive analysis, consumer tests and instrumental analysis were poured from the sample container for each treatment temperature. Fluid milk for descriptive analysis was served in the same 6 oz insulated cups with snap on lids (Dart Container Corporation, Mason, MI) as were used for consumer tests. The samples were coded with random 3-digit numbers and were served in randomized order. Panelists were provided unsalted crackers (Unsalted Tops Premium Saltine Crackers, Nabisco) and spring water (Mountain Spring Water, Blue Ridge, GA) as well as expectorant cups (Dart Container Corporation) for rinsing their palate between each sample. Evaluations were performed on 0 and 6 d for all three replications, as well as days throughout the shelf-life of each replication (d 10, 13, 19).

Table 1. Food and Chemical Standards Used for Descriptive Analysis.

Attribute	Description	Reference
Aroma		
Cooked	Aromatic associated with cooked milk	Evaporated milk (Nestlé Carnation)
Free Fatty Acid	Aromatic associated with short chain free fatty acids	Crumbled feta cheese (Athenos) Butyric acid
Fruity	Aromas associated with fruits such as pineapple	Dole pineapple chunks in 100% pineapple juice (Dole Packaged Foods Corp.)
Sulfur/Eggy	Aromatics associated with sulfurous compounds	Boiled mashed egg
Flavor		
Cooked	Taste associated with cooked milk	Evaporated milk (Nestlé Carnation)
Sour	Taste stimulated by acids	Citric acid solution (0.08%)
Free Fatty Acid	Taste associated with short chain free fatty acids	Dannon Lowfat Plain Yogurt (The Dannon Company, Inc.) Crumbled feta cheese (Athenos)
Fruity	Taste associated with fruits such as pineapple	Dole pineapple chunks in 100% pineapple juice (Dole Packaged Foods Corp.)
Lactone	Taste associated with milkfat	Coconut milk (A Taste of Thai)
Rancid	Taste associated with oxidized oils	Old coconut milk (A Taste of Thai)
Oxidized	Taste resulting from lipid oxidation	Milk exposed to natural light

(Sources: Drake et al., 2001; Meilgaard et al., 1991)

Volatile flavor compounds

Sample Preparation

Solid Phase Microextraction (SPME) was utilized to extract the volatile flavor compounds from the milk samples. An aliquot (10 ml) of each treatment was placed in a pre –cleaned 40 ml amber vial (40 mL; O.D. 28 mm x height 98 mm; Supelco) with a screw cap, a Teflon silica septum (Tan PTFE/white silicone; O.D. 22 mm x thickness 31.75 mm; Supelco), and a magnetic stirring bar (diameter 8 mm x length 13 mm, magnetic octagonal bar; Fisher, Pittsburgh, PA) for agitation of samples and tightly closed. The internal standard solution (1,3-dichlorobenzene; Sigma-Aldrich Chemical Co., Milwaukee, WI), which was prepared (1 ppm v/v) using high resolution gas chromatography grade methanol (EMD Chemicals Inc., Gibbstown, NJ), was added (1 μl) into the vial for quantification of the relative abundance (ppm) of detected volatile odor chemicals. The sample was stored at room temperature (21°C) for 30 min to allow for equilibration between the sample and the headspace within the vial. Volatile odor compounds were extracted from the headspace of the sample using a StableFlex 1cm-50/30 μm three phase (DVB/CAR/PDMS) SPME fiber that was stabilized at 50°C along with the sample using a heating block (Reacti-Therm^{TM}, Pierce Biotechnology, Inc., Rockford IL) for 30 min. The SPME fiber was injected into a splitless injection port of a 5890 Series II gas chromatograph (Hewlett-Packard Co., Palo Alto, CA). Volatile compounds were analyzed in triplicate for each sample.

Gas Chromatography–Mass Spectrometry

Samples were screened using two different gas chromatograph-mass selective detectors (GC-MSD) to obtain a general understanding of the volatile flavor compounds that were present in the milk pasteurization treatments at different days during their shelf-life. The first GC-MS consisted of an HP 5890 Series II GC/HP 5972 mass selective detector (MSD, Hewlett-Packard Co., Palo Alto, CA) equipped with a Rtx®-5 (Crossbond® 5% diphenyl-95% dimethyl polysiloxane) capillary column (RESTEK; Bellefonte, PA) with the following dimensions: 30 m length x 0.25 mm i.d. x 0.25 μm film thickness (d_f). GC conditions were as follows: injection port, 225ºC; the oven temperature was programmed at 40ºC for 1 min with a 13ºC/min of ramp rate until it reached 250ºC where it was held for 1 min (total running time of 18.15 min). The conditions of the MSD were as follows: interface temperature, 250ºC; ionization energy, 70 eV; mass range, 33-350 a.m.u.; scan rate, 2.2 scans/s. Ultra high purity helium was used as the carrier gas at a constant flow rate of 0.96 mL/min for the first GC-MS. The conditions of the second GC-MS were identical to the first instrument and were performed on a Varian 3900 GC/ Saturn 2100 Turbo Ion trap MSD (Palo Alto, CA). Volatile compounds were identified using either the Wiley 138K Mass Spectral Database (John Wiley and Sons, Inc., New York, NY) for HP-GC-MSD or the NIST 02 library (National Institute of Standards and Technology, Gaithersburg, MD).

Statistical Analysis

A randomized complete block design with six replications was utilized to analyze the effects ($p<0.05$) of fluid milk pasteurization treatment on shelf-life and consumer acceptability. The Least Significant Difference (LSD) test was utilized to separate means when differences occurred. Agglomerative hierarchical clustering (XLStat, 2006) was performed to cluster consumers together based on their liking and preference of milk pasteurization treatments. Descriptive sensory attributes data were analyzed using Principal Component Analysis (PCA) (SAS 9.1 Cary, NC) to differentiate between milk pasteurization treatments. External preference mapping (XLStat, 2006) was conducted on the descriptive attribute data and the consumer acceptability scores to determine the relationship between sensory attributes and consumer preference.

CHAPTER IV

RESULTS AND DISCUSSION

Shelf-Life

There was no difference ($p>0.05$) in shelf-life among the four pasteurization treatments. The shelf-life for the four fluid milk samples ranged from 13.2 to 14.9 days (Table 2). From these results, it appears that shelf-life at these pasteurization temperatures may be more dependent on other factors such as bacterial load, post-pasteurization contamination, and storage conditions. The shelf-life of the bottled fluid milk in the United States is reported to average between 10 to 21 d when stored at 4-8°C (Allen and Joseph, 1985; Chapman et al., 2001). The milk samples in the present study were held at 7°C ± 1°C throughout the course of the study, and Barnard (1972) states that for every 3°C increase in holding temperature, the shelf-life of milk is shortened by one half. The temperature used in this study was chosen as it is more customary in dairy research, reflecting typical temperatures of dairy cases in the supermarket and home refrigerators (White, 1993).

Consumer Acceptability

Differences ($p<0.05$) were found in consumer acceptability of the four fluid milk treatment temperatures (Table 2). On day 0 (day of pasteurization), consumers (n=298) preferred ($p<0.05$) the milk sample pasteurized at 79°C over all other treatments with a mean score of 6.7, which would be categorized between “like slightly” and “like moderately” on the 9-point hedonic scale. The other three treatment temperatures (77°C, 82° C and 85°C) received mean ratings corresponding to “like slightly” on the hedonic scale. Similar results were noted for consumer scores of the attribute “flavor” (Table 2). Differences ($p<0.05$) were also observed for consumer panels conducted 6 days post-pasteurization. The panelists (n=300) preferred ($p<0.05$) samples pasteurized at 79°C and 82°C over 77°F, based on the attribute of “overall acceptability”. However, all 6 d milk samples received mean scores categorized between “like slightly” and “like moderately” on the 9-point hedonic scale. Based on the attribute of “flavor”, consumers preferred the 79°C sample over the other three treatments. Results demonstrate that consumer acceptability differences between the 79°C treatment and other pasteurization treatments were more distinctive at Day 0 than Day 6.

Table 2. Mean Overall Consumer Acceptability, Flavor Acceptability and Shelf-Life of 2 % Fluid Milk Pasteurized at Varying Temperatures.

Treatment	Day 0 Overall Acceptability	Day 0 Flavor Acceptability	Day 6 Overall Acceptability	Day 6 Flavor Acceptability	Shelf-Life
77°C	6.1[b]	6.2[b]	6.1[b]	6.2[b]	13.2[a]
79°C	6.7[a]	6.6[a]	6.5[a]	6.6[a]	14.9[a]
82°C	6.1[b]	6.2[b]	6.6[a]	6.2[b]	14.7[a]
85°C	5.9[b]	6.0[b]	6.4[ab]	6.0[b]	13.7[a]
Std. Error	0.09	0.11	0.11	0.13	1.1

[abc] Means within each column with unlike superscripts are different ($p<0.05$).

[1] Consumer overall acceptability and flavor scores were evaluated using a nine point hedonic scale where 1 represents dislike extremely, 5 represents neither like nor dislike, and 9 represents like extremely.

[2] All treatment temperatures were held for 15 s.

Cluster Analysis

Cluster analysis was utilized to further understand the consumer acceptability of fluid milk. Consumers were grouped into eight clusters (Tables 3-4) based on treatment preference for both 0 and 6 d taste tests. Each cluster represents consumers with similar milk preferences. The analysis of acceptability by consumer segments serves as a functional indicator for the dairy industry of potential implications for changes to the pasteurization process upon purchaser preference.

Day 0 Cluster Analysis

Consumers in clusters 4-8 like milk in general, and all clusters find 79°C highly acceptable (Table 3). This may be positive for the industry since the majority of processing plants pasteurize their fluid milk products at 79-81°C for 15-18 s (White, 2007). In cluster 4, panelists preferred ($p<0.05$) 79°C and 82°C, which they "liked moderately", while they "liked slightly" 85°C and "disliked slightly" 77°C. Consumers in cluster 5 preferred ($p<0.05$) 79°C and 85°C treatments. The 6th cluster, which contains 8% of consumers, "neither liked nor disliked" 85°C, while other samples were in the "like moderately" to "like very much" range. Perhaps this cluster did not like the cooked flavor associated with the higher pasteurization temperature (Figure 1). The largest number of consumers was in cluster 7. These consumers "like moderately" to "like very much" all samples of milk. The final group of consumers on 0 d prefers ($p<0.05$) 77°C and 79°C over the other samples, followed by 82°C and then 85°C, revealing that the consumers did not prefer milk with cooked flavor.

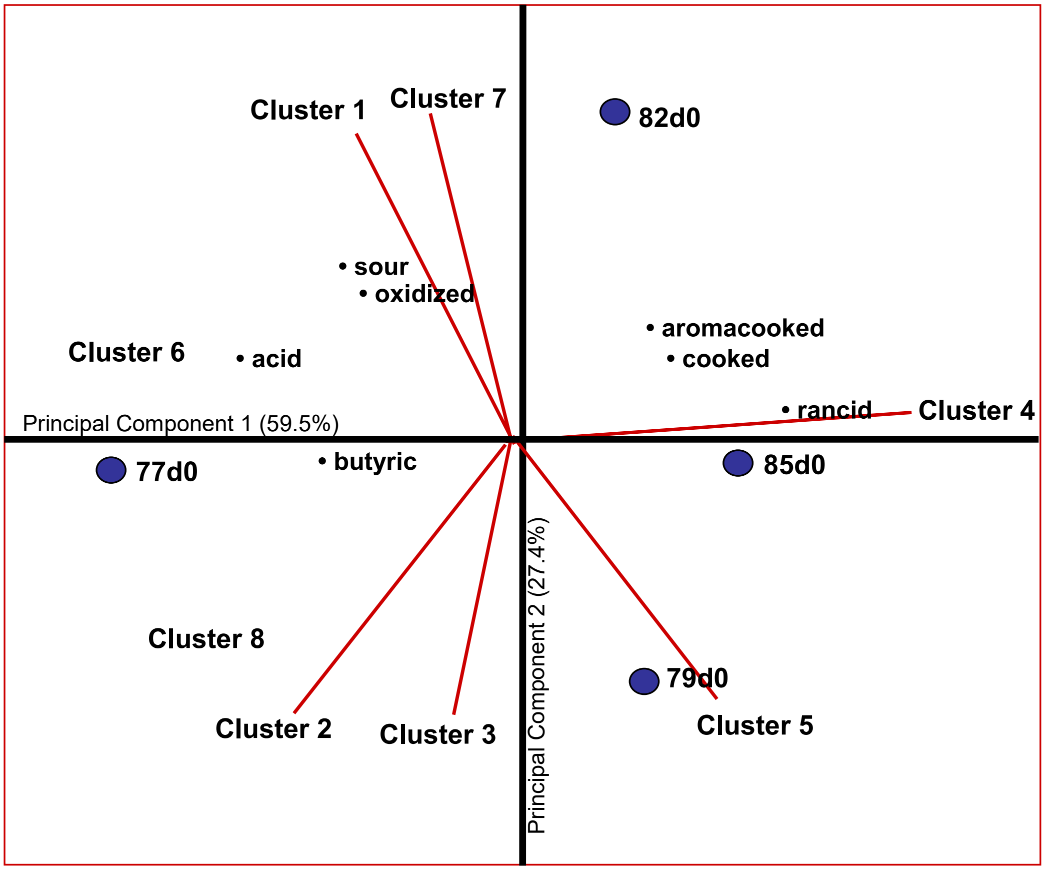

Figure 1. External Preference Map of Day 0 Consumer Data for Milk Pasteurized at Four Treatment Temperatures.

Table 3. Consumer Acceptability, Based on Cluster Analysis, of 2 % Fluid Milk Pasteurized at Varying Temperatures Immediately Post-Pasteurization (Day 0).

Cluster	Number of Consumers	Percent of Consumers	77°C	79°C	82°C	85°C
1	45	15.1	5.6^{a}	4.5^{b}	5.8^{a}	5.4^{a}
2	33	11.1	5.4^{b}	6.1^{a}	3.5^{c}	3.0^{c}
3	11	3.7	4.6^{ab}	5.2^{a}	3.0^{b}	4.3^{ab}
4	49	16.4	4.3^{c}	7.4^{a}	7.2^{a}	6.2^{b}
5	24	8.1	5.1^{b}	7.2^{a}	4.1^{b}	7.6^{a}
6	17	5.7	7.7^{a}	7.1^{a}	7.4^{a}	5.1^{b}
7	68	22.8	7.5^{ab}	7.5^{ab}	7.8^{a}	7.3^{b}
8	51	17.1	7.2^{a}	7.2^{a}	6.1^{b}	5.5^{c}

[abc] Means within each cluster with unlike superscripts are different ($p<0.05$)
[1] Consumer acceptability was evaluated using a nine point hedonic scale where 1 represents dislike extremely, 5 represents neither like nor dislike, and 9 represents like extremely.

Table 4. Consumer Acceptability, Based on Cluster Analysis, of 2 % Fluid Milk Pasteurized at Varying Temperatures Six Days Following Pasteurization (Day 6).

Cluster	Number of Consumers	Percent of Consumers	77°C	79°C	82°C	85°C
1	28	9.3	4.2^{b}	6.0^{a}	6.7^{a}	4.4^{b}
2	18	6.0	5.2^{b}	3.9^{c}	5.7^{b}	6.8^{a}
3	17	5.7	6.1^{a}	3.6^{c}	5.9^{a}	4.6^{b}
4	16	5.3	6.2^{a}	6.4^{a}	3.9^{b}	3.4^{b}
5	25	8.3	4.4^{b}	7.0^{a}	7.3^{a}	7.5^{a}
6	59	19.7	5.4^{c}	6.2^{b}	5.3^{c}	7.1^{a}
7	46	15.3	6.8^{b}	6.7^{b}	7.5^{a}	5.2^{c}
8	91	30.3	7.3^{a}	7.6^{a}	7.6^{a}	7.6^{a}

[abc] Means within each cluster with unlike superscripts are different ($p<0.05$)
[1] Consumer acceptability was evaluated using a nine point hedonic scale where 1 represents dislike extremely, 5 represents neither like nor dislike, and 9 represents like extremely.

Day 6 Cluster Analysis

Consumers from the 6 d taste panel were also grouped into eight clusters based on milk preferences. In cluster 2, 85°C was the most preferred treatment with a mean overall acceptability score corresponding to “like moderately” on the hedonic scale. This group, which comprises 15% of consumers, must like the cooked flavor (Figure 2) of this sample while in general they do not find milk very acceptable. This is an interesting consideration, since perhaps there are small segments of consumers who do not typically consume fluid milk but might if the product had a more cooked flavor brought on by higher pasteurization temperatures. On the other hand, consumers in cluster 4 “disliked slightly” to “disliked moderately” the higher treatment temperatures (82 and 85°C), likely due to the cooked flavor. Cluster 5 preferred ($p<0.05$) 79, 82, and 85^{o} C over the lowest pasteurization temperature (77°C). Cluster 6 contains the second largest segment of consumers, and this group preferred treatment 85°C, followed by 79°C, and then 77°C and 82°C. Cluster 7 demonstrates an interesting contrast in milk preferences. While cluster 6 preferred 85°C, cluster 7 rated that treatment temperature the lowest. Furthermore, while cluster 6 rated the 82°C a mean score of 5.3, cluster 7 preferred this treatment temperature above the others and rated it 7.5, which falls between “like moderately” and “like very much” on the hedonic scale. Cluster 8 had the largest number of consumers (30%), and this group considers all treatment temperatures very acceptable.

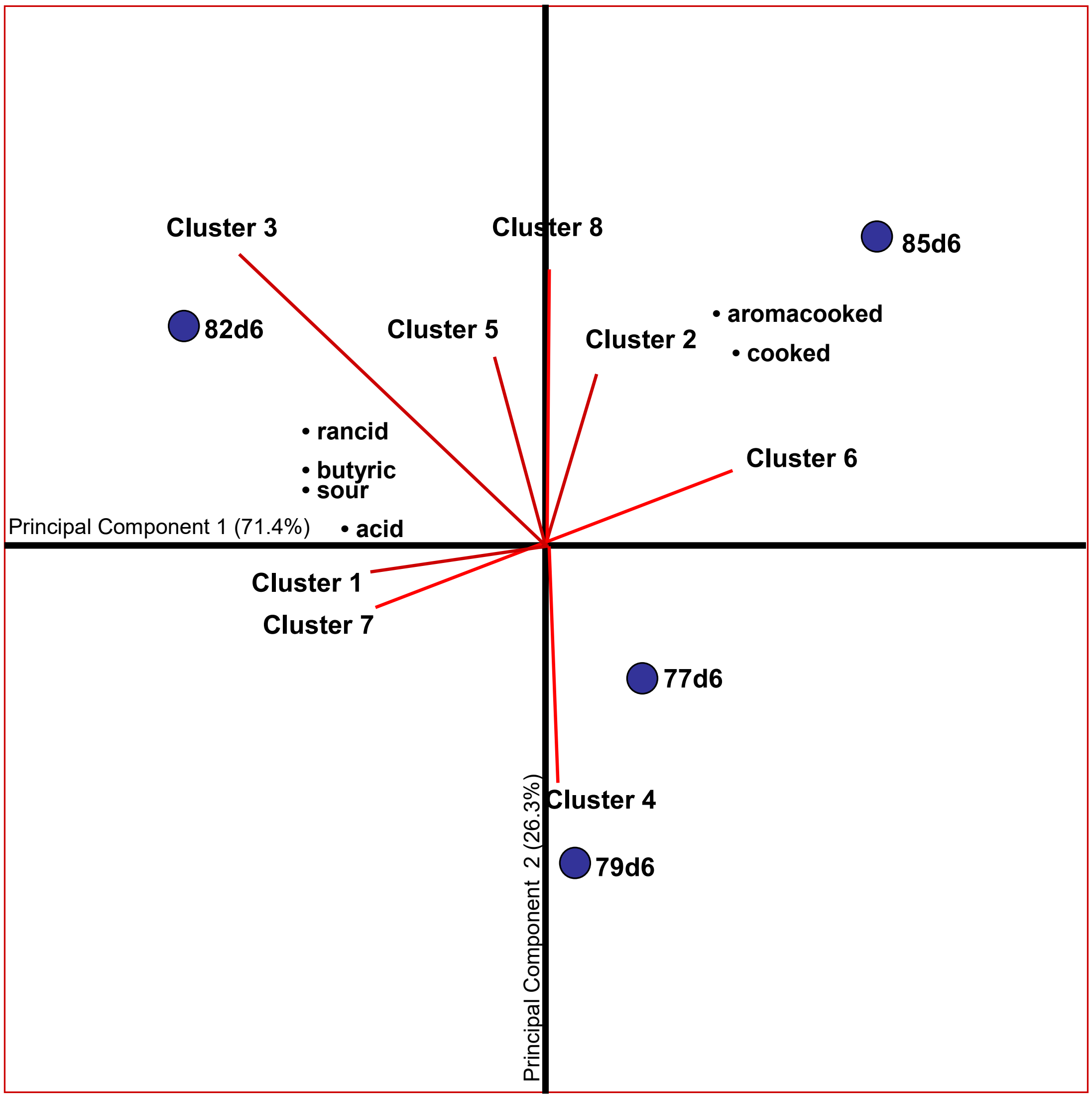

Figure 2. External Preference Map of Day 6 Consumer Data for Milk Pasteurized at Four Treatment Temperatures.

Descriptive Analysis

Milks that were pasteurized at four different temperatures could be differentiated by aroma and flavor attributes using principal components analysis (Figure 3). The principal components analysis biplot is useful for understanding the differences observed. According to the eigenvalues, the majority (65.2%) of variation is explained by dimension 1, which means attributes closer to the horizontal axis. Dimension 2 explains 14.2% of variation in milk samples. Thus, samples spaced farther apart on the biplot are perceptually more different than samples found closer together (Dijksterhuis, 1997).

As would be expected, the two higher treatment temperatures (82°C and 85°C) were highly characterized by a cooked aroma and flavor at 0 d (day of pasteurization). Meanwhile, treatments 77°C and 79°C appear to have a milder taste on 0 d. Treatment 85°C was still distinguishable by the cooked flavor and aroma on 6 d. When the descriptive analysis results are applied to the cluster analysis results, the idea is reinforced that some consumers object to a strong cooked flavor, such as can be seen in 6 d cluster 4 (Figure 2), while others seem to prefer a cooked flavor (6 d cluster 6). By 13d in product shelf-life, treatments 77°C, 79°C and 85°C were more closely associated with the flavor term “oxidized”. The sensory defect of oxidation may be the result of more than one factor, yet it is often characterized as cardboardy, oily or painty. Light exposure may result in flavors described as light-activated flavors (LAF), burnt, or sunlight flavor (Shipe et al., 1978; Marsili, 2007). Throughout the course of milk shelf-life, the product changes as off-flavors develop. The descriptive panelists were able to discern those changes, and by 17 d and 19 d the product was characterized by the flavor terms “acid,” “sour,” “butyric,” and “rancid.” Results reveal that milk at day 0 can be differentiated by cooked aroma, cooked flavor, and oxidized descriptors, but different temperature treatments cannot be differentiated at 10 days or more post-pasteurization and rancid, acid, sour, and butyric descriptors define the end of shelf-life for all treatments. Rancid flavor in milk is a result of the hydrolysis of short chain triglycerides to free fatty acids by lipases, which naturally occur in milk and are also produced by psychrotrophic bacteria (Bodyfelt et al., 1988). “Microbial flavors” is a term used to describe off-flavors and aromas in milk that result from undesirable organisms and enzymes, such as the sour flavor characterized by acetic and propionic acids (Shipe et al., 1978).

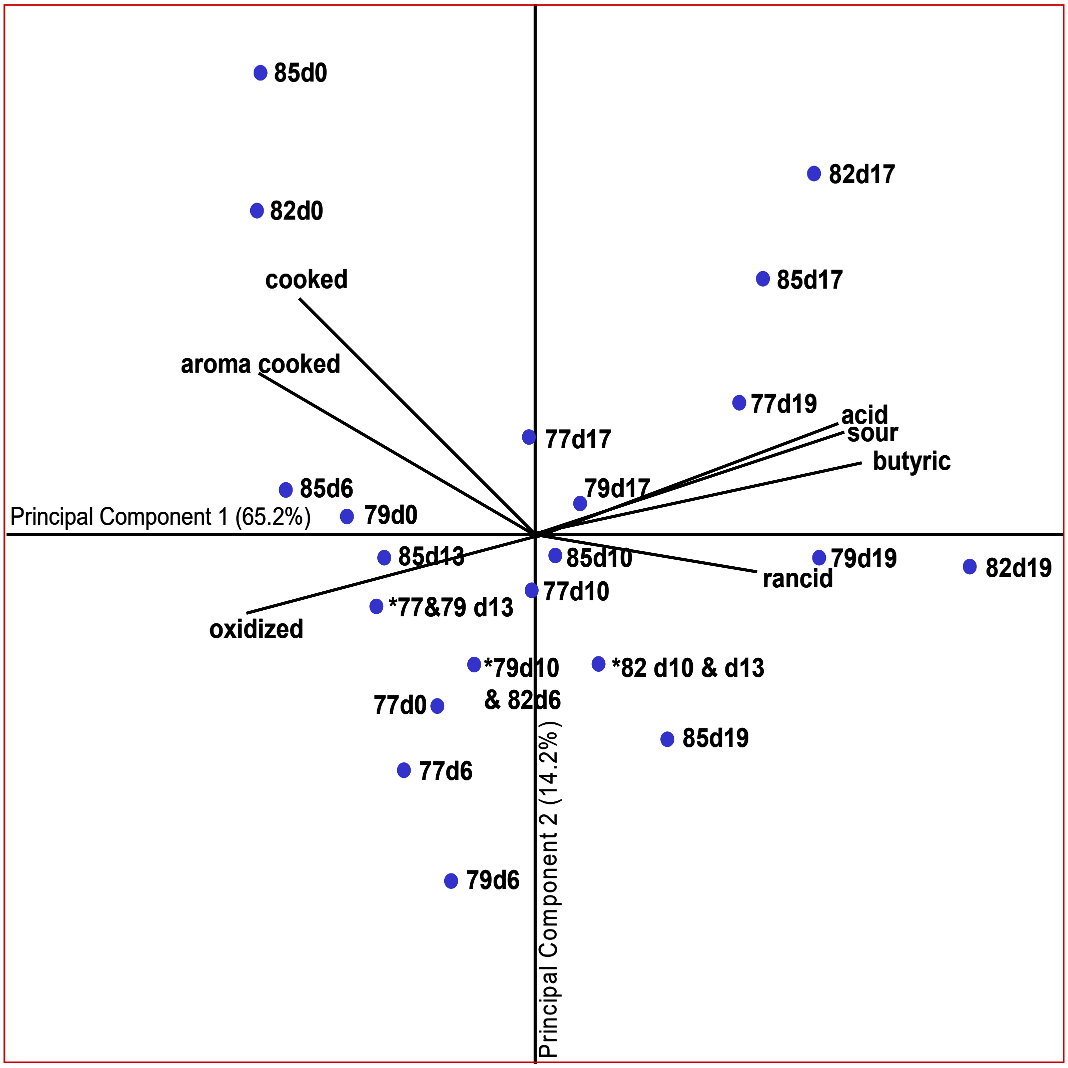

Figure 3. Principal Component Biplot of Descriptive Analysis for Milk Pasteurized at Varying Temperatures 0 to 19 Days Post-Pasteurization.

Volatile Flavor Compounds

When results of Gas Chromatography – Mass Spectrometry analysis are plotted on principal components analysis map (Figure 4), it is again made clear that there are changes in the product over the course of its shelf-life. In the present study, component 1 (horizontal) accounted for 37.5 % of the variation, while component 2 explains 18.2 % of the variation.

Starting on 0 d (day of pasteurization) treatment 77°C is highly correlated with the volatile compound hydroxylamine, as well as phenol, and butanoic acid. The 79°C treatment is associated with higher concentrations of the volatile compounds hydroxylamine, phenol, butanoic acid, hexanoic acid and hexanol. Yet, 79°C is also associated with the compounds benzoic acid, pXylene, nonanone and eugenol to a lesser extent. The 82°C treatment was also characterized by compounds such as benzoic acid, pXylene, nonanone and eugenol. Vazquez-Landaverde et al. (2005) determined that 2-nonanone was not an important aroma contributor in raw and pasteurized milk samples. On 0 d the 85°C treatment is perceptually the most different from the other treatments. This treatment is plotted in the lower right quadrant, alongside compounds such as hexenol, decane methyl, tetrahydro-furane, butanone, and hydroperoxide. By 7 d treatments 82°C and 85°C are more similar in terms of distinguishing volatile compounds present, which include butanol, methyl isobutyl ketone (MIBK), pentane, butanediol and guanidine. Milk pasteurized at 77°C is more closely characterized by the volatile compounds limonene, heptanol, and nonane 7 days post-pasteurization. Contarini et al. (1997) detected limonene in their research, yet the authors were not able to identify its contribution to milk taste and odor, nor were they able to find relevant information on this compound in their review of literature. By days 10 and 13, considerable changes occurred in the product and all treatment temperatures become more closely defined by the volatile compounds acetic acid, heptanone and hexanone. Sour or acid flavors observed by the descriptive sensory panel at the end of samples' shelf-life may be a result of enzymatic activity characterized by acetic acid (Shipe et al., 1978). It is important to note from these results that similarly to the descriptive results, milk samples could be differentiated based on volatile compound composition on Day 0 and 6 but could not on day 10 and Day 13. This indicates that each milk may be following a similar pattern of degradation and may be spoiling in similar ways.

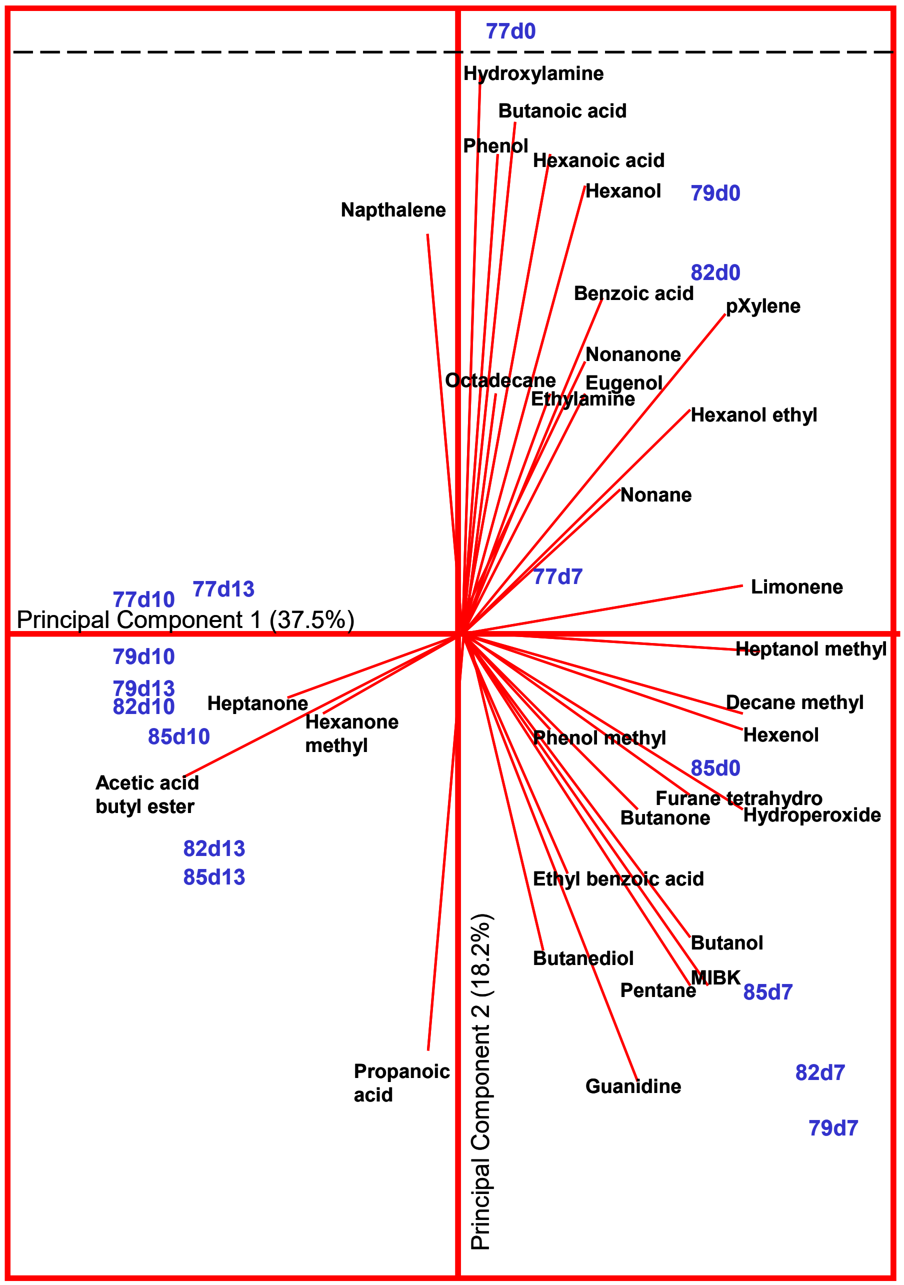

Figure 4. Principal Component Biplot of Volatile Compound Profiles of Milk Pasteurized at Varying Temperatures 0 to 13 Days Post-Pasteurization.

CHAPTER V

CONCLUSIONS

Varying pasteurization temperature had no effect on shelf-life. Consumers preferred milk that was pasteurized at 79°C over all other treatments on the day of pasteurization, yet at 6 days post-pasteurization only the 79°C treatment was preferred over the 77°C treatment. This reveals that altering pasteurization temperature between 77°C and 85°C has a greater effect on consumer acceptability early on in the shelf-life of the products. Consumers could be grouped into 8 clusters on both day 0 and 6 based on acceptability scores. All consumers that liked milk found 79°C to be highly acceptable, and the largest cluster (30% of consumers) liked all treatment temperatures. Some clusters also liked milk with a cooked flavor (82°C and 85°C treatments), and other clusters did not like milk with a cooked flavor. Therefore, altering pasteurization temperatures above 79°C may result in decreased acceptance in some but not all consumer groups on day 0 and day 6 post-pasteurization. This research also revealed that milk could not be differentiated based on pasteurization temperature by a trained sensory descriptive panel or volatile compound composition towards the end of shelf-life.

REFERENCES

Allen, J.C., and Joseph, G. 1985. Review Article: Deterioration of Pasteurized Milk on Storage. J. Dairy Res. 52: 469-486.

Barnard, S.F. 1972. Importance of Shelf Life for Consumers of Milk. J. Dairy Sci. 55(1): 134-136.

Bendall, J. G. 2001. Aroma Compounds of Fresh Milk From New Zealand Cows Fed Different Diets. J. Agric. Food Chem. 49:4825–4832.

Bodyfelt, F.W., Tobias, J., and Trout, G.M. 1988. *The Sensory Evaluation of Dairy Products.* Westport, CT: AVI.

Boor, K.J. 2001. Fluid Dairy Product Quality and Safety: Looking to the Future. J. Dairy Sci. 84:1-11.

Carpenter, R.P., Lyon, D.H., and Hasdell, T.A. 2000. *Guidelines for Sensory Analysis in Food Product Development and Quality Control* (2nd ed.). Gaithersburg, MD: Aspen Publishers.

Casapia, E.L., Coggins, P.C., Schilling, M.W., Yoon, Y., and White, C.H. 2006. The Relationship Between Consumer Acceptability and Descriptive Sensory Attributes in Cheddar Cheese. J. Sens. Studies. 21:112-127.

Chapman, K.W., and Boor, K.J. 2001. Acceptance of 2% Ultra-Pasteurized Milk by Consumers, 6-11 Years Old. J. Dairy Sci. 84:951-954.

Chapman, K.W., Lawless, H.T., and Boor, K.J. 2001. Quantitative Descriptive Analysis and Principal Component Analysis for Sensory Characterization of Ultrapasteurized Milk. J. Dairy Sci. 84:12-20.

Christensen, K.R., and Reineccius, G.A. 1992. Gas Chromatographic Analysis of Volatile Sulfur Compounds from Heated Milk Using Static Headspace Sampling. J. Dairy Sci. 75:2098-2104.

Civille, C.V., and Lawless, H.T. 1997. The Importance of Language in Describing Perceptions. Ch. 1.8 in *Descriptive Sensory Analysis in Practice*. Ed. Gacula, M.C., Jr. Food and Nutrition Press, Trumbull, Connecticut.

Claassen, M., and Lawless, H.T. 1992. Comparison of Descriptive Terminology Systems for Sensory Evaluation of Fluid Milk. J. Food Sci. 57:596-600, 621.

Contarini, G. and Povolo, M. 2002. Volatile Fraction of Milk: Comparison Between Purge and Trap and Solid Phase Microextraction Techniques. J. Agric. Food Chem. 50:7350-7355.

Contarini, G., Povolo, M., Leardi, R., and Toppino, P. M. 1997. Influence of Heat Treatment on the Volatile Compounds of Milk. J. Agric. Food Chem. 45:3171–3177.

Dijksterhuis, G.B. 1997. *Multivariate Data Analysis in Sensory and Consumer Science*. Food and Nutrition Press, Trumbull, Connecticut.

Douglas, S. A., Gray, M.J., Crandall, A.D., Boor, K.J. 2000. Characterization of Chocolate Milk Spoilage Patterns. J. Food Prot. 63:516-521.

Drake, M.A., McIngvale, S.C., Gerard, P.D., Cadwallader, K.R., and Civille, G.V. 2001. Development of a Descriptive Language for Cheddar Cheese. J. Food Sci. 66:1422-1427.

Erba, E.M., Aplin, R.D., and Stephenson, M.W. 1997. An Analysis of Processing and Distribution Productivity and Costs in 35 Fluid Milk Plants. Publication of Cornell Program on Dairy Markets and Policy. New York State College of Agriculture and Life Sciences. Dept. of Agricultural, Resource and Managerial Economics.

Everitt, B.S., Landau, S., and Leese, M. 2001. *Cluster Analysis*. Oxford University Press, New York, NY.

Goff, H.D., and Griffiths, M.W. 2006. Major Advances in Fresh Milk and Milk Products: Fluid Milk Products and Frozen Desserts. J. Dairy Sci. 89:1163-1173.

Gruetzmacher, T.J., and Bradley Jr, R.L. 1999. Identification and Control of Processing Variables that Affect the Quality and Safety of Fluid Milk. J. Food Prot. 62:625-631.

Handbook of Dairy Processing. 2000. Lund, Sweden: Tetra Pak Processing Systems AB.

Hayes, W., White, C.H., and Drake, M.A. 2002. Sensory Aroma Characteristics of Milk Spoilage by *Pseudomonas* Species. J. Food Sci. 67:861-867.

Helgesen, H., Solheim, R., and Naes, T. 1997. Consumer Preference Mapping of Dry Fermented Lamb Sausages. Food Qual. and Pref. 8(2):97-109.

Huang, X., and Hsieh, F.H. 2005. Physical Properties, Sensory Attributes, and Consumer Preference of Pear Fruit Leather. J. Food Sci. 70:E177-E186.

Jones, R.M. 1997. Statistical Techniques for Data Relationships. Ch. 4 in *Relating Consumer, Descriptive, and Laboratory Data*. Muñoz. A.M., Ed. (Manual 30). West Conshohocken, PA: ASTM.

Lawless H.T., and Heyman, H. 1998. *Sensory Evaluation of Food*. Chapman and Hall. Aspen Publications, Gaithersburg, MD.

Marsili, R.T. 1999. SPME-MS-MVA as an Electronic Nose for the Study of Off-Flavors in Milk. J. Agric. Food Chem. 47:648-654.

Marsili, R.T. 2007. *Sensory Directed Flavor Analysis.* CRC Press, Taylor and Francis Group. Boca Raton, FL.

McDonald, W.L., O'Riley, K.J., Schroen, C.J., and Condron, R.J. 2005. Heat Inactivation of *Mycobacterium Avium* Subsp. *Paratuberculosis* in Milk. Appl. and Environ. Micro. 71:1785-1789.

McSweeney, P.L.H., Nursten, H.E. and Urbach, G., 1997. Flavours and Off-Flavours in Milk and Dairy Products. In: *Advanced Dairy Chemistry*. (Vol. 3). Fox, P. F., Ed. London: Chapman & Hall.

Meilgaard, M., Civille, G. V., and Carr, B. T. 1991. *Sensory Evaluation Techniques*. (2^{nd} ed.). Boston, MA: CRC Press.

Meullenet, J.F., Xiang, R., Bellman-Homer, T., Dias, P., Zivanovic, S., Fromm, H., and Liu, Z. 2002. Preference Mapping of Commercial Toasted White Corn Tortilla Chips. J. Food Sci. 67:1950-1957.

Murray J.M., and Delahunty, C.M. 2000. Selection of standards to reference terms in a Cheddar-type cheese flavor language. J. Sens. Stud. 15:179–199.

Murray, J.M., Delahunty, C.M., and Baxter, I.A. 2001. Descriptive Sensory Analysis: Past, Present and Future. Food Res. Internat. 34:461-471.

Pasteurized Milk Ordinance. 2001. Public Health Service/Food and Drug Administration. Publication No. 229.

Pawliszyn, J. 1997. *Solid Phase Microextraction: Theory and Practice*. Wiley-VCH. USA.

Popper, R., Heymann, H., and Rossi, F. 1997. Three Multivariate Approaches to Relating Consumer to Descriptive Data. Ch. 5 in *Relating Consumer, Descriptive, and Laboratory Data*. Muñoz. A.M., Ed. (Manual 30). West Conshohocken, PA: ASTM.

SAS Institute Inc. 2002. Version 9.1. SAS Institute Inc. Cary, NC.

Schilling, M.W., and Coggins, P.C. 2007. Utilization of Agglomerative Hierarchical Clustering in the Analysis of Hedonic Scaled Consumer Acceptability Data. J. Sens. Studies. (In Press).

Shipe, W.F., Bassette R., Deane D.D., Dunkley W.L., Hammond E.G., Harper W.J., Kleyn D.H., Morgan M.E., Nelson J.H., Scanlan R.A. 1978. Off Flavors of Milk: Nomenclature, Standards and Bibliography. J. Dairy Sci. 61:855–869.

Simon, M., and Hansen, A.P. 2001. Effects of Various Dairy Packaging Materials on the Shelf Life and Flavor of Pasteurized Milk. J. Dairy Sci. 84: 767-773.

Stabel, J.R., Steadham, E.M., and Bolin, C.A. 1997. Heat Inactivation of *Mycobacterium paratuberculosis* in Raw Milk: Are Current Pasteurization Conditions Effective? Appl. and Environ. Micro. 63:4975-4977.

Stampanoni, C.R. (1997). The Use of Standardized Flavor Languages and Quantitative Flavor Profiling Techniques. Ch. 2.6 in *Descriptive Sensory Analysis in Practice*. Gacula, M.C., Jr., Ed. Food and Nutrition Press, Trumbull, Connecticut.

Thomas, E.L. 1981. Trends in milk flavors. J. Dairy Sci. 64:1023-1027.

Thompson, J.L., Drake, M.A., Lopetcharat, K., and Yates, M.D. 2004. Preference Mapping of Commercial Chocolate Milks. J. Food Sci. 69(9): S406-S413.

Toso, B., Procida, G., and Stefanon, B. 2002. Determination of volatile compounds in cows' milk using headspace GC-MS. J. Dairy Res. 69:569–577.

Vazquez-Landaverde, PA. Velazquez, G., Torres, J. A. and Qian, M. C. 2005. Quantitative Determination of Thermally Derived Off-Flavor Compounds in Milk Using Solid-Phase Microextraction and Gas Chromatography. J. Dairy Sci. 88:3764-3772.

XLStat, 2006.

Westad, F., Hersleth, M., and Lea., P. 2004. Strategies for Consumer Segmentation with Applications on Preference Data. Food Qual. and Pref. 15:681-687.

White, C.H. 1993. Rapid Methods for Estimation and Prediction of Shelf-Life of Milk and Dairy Products. J. Dairy Sci. 76:3126-3132.

White, C.H. 2005. Personal communication.

White, C.H. 2007. Personal communication.

APPENDIX

SCORE SHEETS, HISTOGRAMS AND DENDROGRAMS

Product: Milk **Date:**

Directions

Please taste each of the five (5) milk samples starting with the number on the left and continuing to the right. Please expectorate the sample and rinse your mouth with water in between samples. Rate each sample for flavor and overall acceptability (liking). Each column will need one check mark. Next, please rate the five milk samples in order of most preferred to least preferred.

184	753	377	296	808	**FLAVOR**
					Like extremely
					Like very much
					Like moderately
					Like slightly
					Neither like nor dislike
					Dislike slightly
					Dislike moderately
					Dislike very much
					Dislike extremely

184	753	377	296	808	**OVERALL LIKING**
					Like extremely
					Like very much
					Like moderately
					Like slightly
					Neither like nor dislike
					Dislike slightly
					Dislike moderately
					Dislike very much
					Dislike extremely

Please rank each sample in order of preference with the first (1st) being most preferred and the fifth (5th) being least preferred:

Samples: 184 753 377 296 808

____
1st

____
2nd

____
3rd

____
4th

____
5th

Additional comments: ____

____

____

Figure A1. Score Sheet for Consumer Acceptability Tests.

Descriptive Evaluation of Fluid Milk

Date_______________ Time___________________

Panelist Code_______ Gender: F M

0: low intensity
15: high intensity

<u>AROMA</u>

Cooked

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Free Fatty Acid

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Fruity/Fermented

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Sulfur/Eggy

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Other:___________________________

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Figure A2. Score Sheet for Descriptive Sensory Analysis.

FLAVOR

Cooked

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Sour

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Oxidized / Light Activated Flavor

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Free Fatty Acid

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Fruity/Fermented

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Lactone

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Rancid

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Other:__________________________

|____|____|____|____|____|____|____|____|____|____|____|____|____|____|____|
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Figure A2 continued. Score Sheet for Descriptive Sensory Analysis.

MILK JUDGING SCORE CARD

Name: **Date:**

Sample Code								
Overall Score								
Acid								
Astringent								
Barny								
Bitter								
Carton/Paperboard								
Coagulated								
Cooked								
Cowy								
Feed								
Fermented/Fruity								
Flat								
Foreign								
Garlic/Onion								
Lacks Freshness								
Oxidized – Light								
Oxidized – Lipid								
Rancid								
Salty								
Unclean								
Other (describe)								

Figure A3. Score Card for Expert Judging.

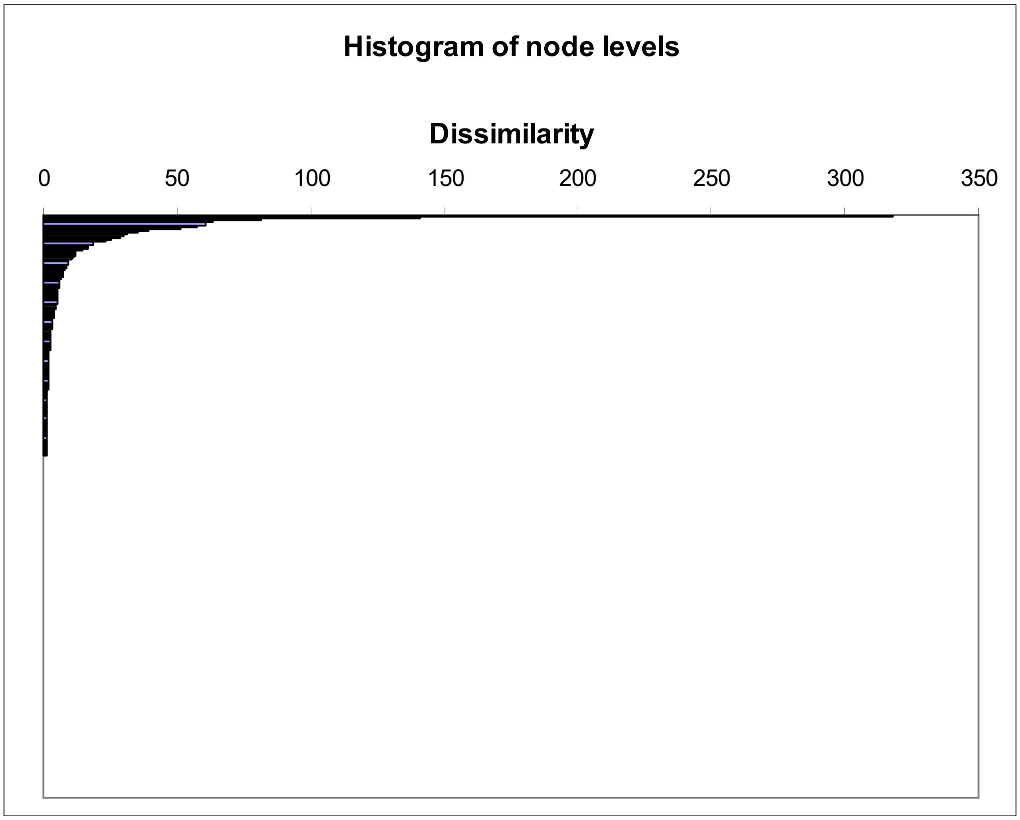

Figure A4. Histogram of Dissimilarity Utilized to Determine the Number of Clusters on Day of Pasteurization (Day 0).

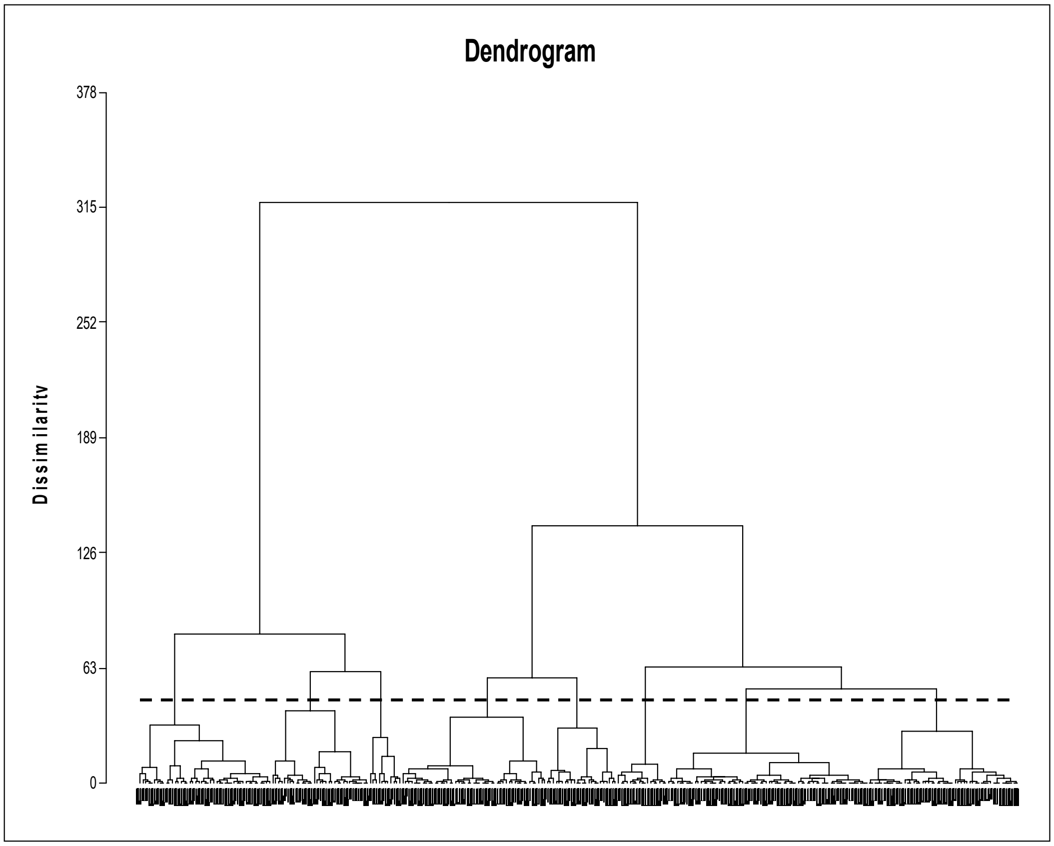

Figure A5. Dendrogram Revealing Grouping of Consumers into Clusters on Day of Pasteurization (Day 0) Based on Dissimilarity of Consumer Acceptability Scores.

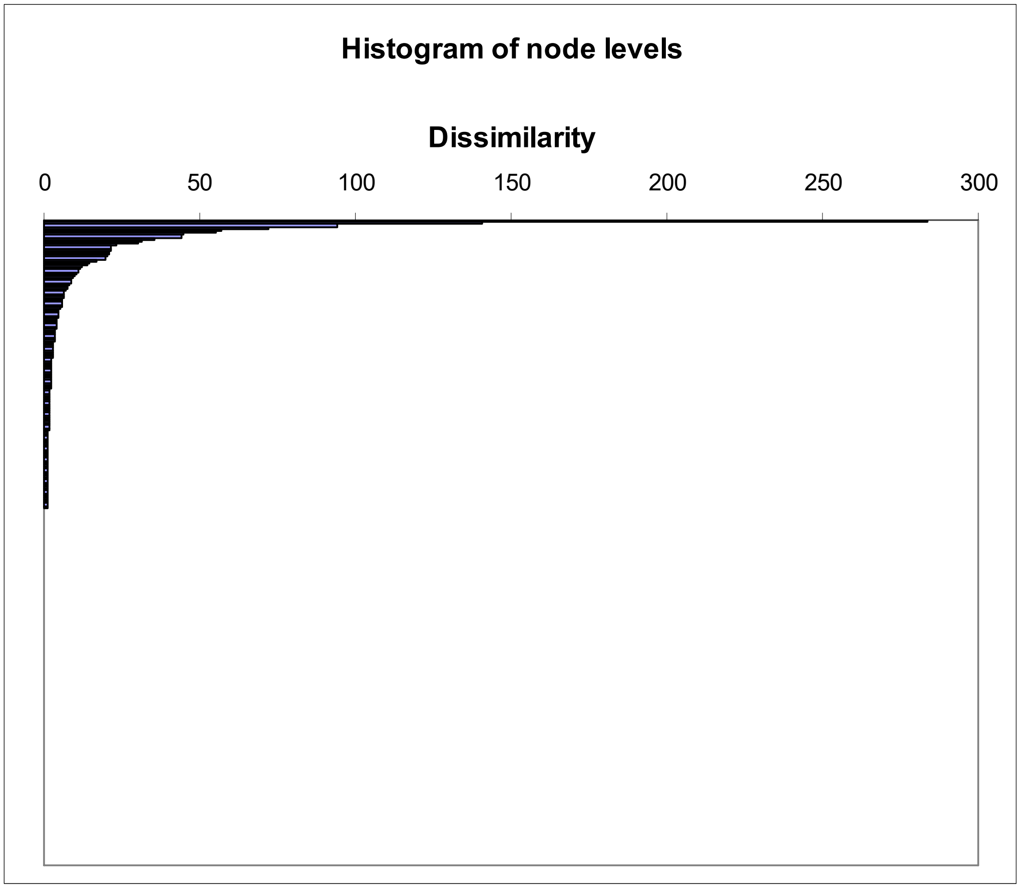

Figure A6. Histogram of Dissimilarity Utilized to Determine the Number of Clusters 6 Days Post-Pasteurization (Day 6).

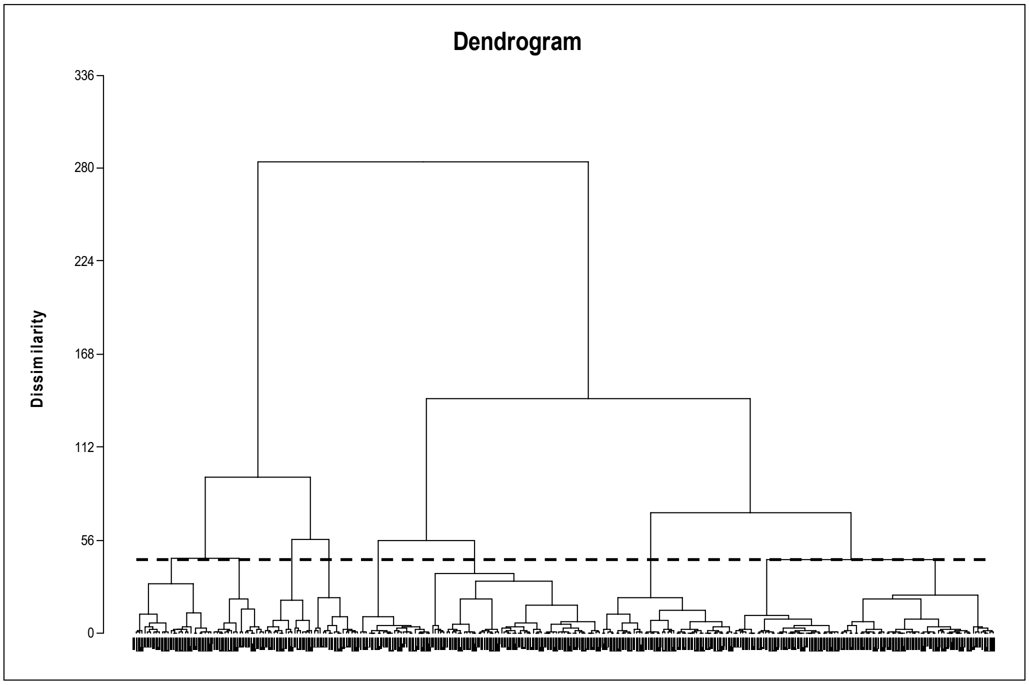

Figure A7. Dendrogram Revealing Grouping of Consumers into Clusters 6 Days Post-Pasteurization (Day 6) Based on Dissimilarity of Consumer Acceptability Scores.