Abstract

CAUDLE, ALISSA DAWN. Flavor Formation in Skim Milk Powder and Flavor Carry-Through into Ingredient Applications. (Under the direction of Dr. MaryAnne Drake.)

Skim milk powder (SMP) is commonly used as a food ingredient. Both processing procedures and storage conditions have been noted as important contributors to SMP flavor. Research has not addressed how or if SMP flavor variability impacts consumer acceptance of ingredient applications. Further, many studies have addressed the importance and impact of SMP storage on flavor variability, but the impact of SMP processing on flavor formation has not been extensively researched. In this study, we examined both the impact of SMP flavor variability on consumer acceptability in SMP ingredient applications as well as the flavor formation of SMP throughout production, with emphasis on steps where heat was applied.

For the first study, twenty reconstituted low heat SMP were screened using a previously established defined sensory language. Two SMP free of off-flavors (flavor similar to fresh fluid skim milk) and two SMP exhibiting off-flavors (exhibiting flavors not found in fluid skim milk) were selected for further study. Each SMP was directly reconstituted or incorporated into standard formulas for vanilla ice cream, strawberry yogurt, hot cocoa mix, and white chocolate bars followed by consumer acceptance testing. Descriptive panelists documented sweet aromatic and cooked flavors in the fresh-flavored SMP and these flavors as well as fatty/fryer oil and animal-like flavors in the off-flavored SMP. Consumer acceptance scores were lower for the off-flavored SMP than for the fresh-flavored SMP when evaluated directly reconstituted ($p<0.05$) and for ingredient applications made using off-flavored SMP ($p<0.05$). Off-flavors in SMP

documented by descriptive analysis and trained panelists can carry-through into ingredient applications with SMP and negatively affect consumer acceptability.

For the second study, samples were collected throughout the production process (raw milk to finished powder) across a 40 hour production run. Descriptive sensory analysis and instrumental volatile analysis were conducted. The sensory attributes of the milk changed significantly throughout the SMP production process ($p<0.05$). Cooked and sweet aromatic flavors increased throughout the process. Important flavors identified by gas chromotography-olfactometry were heat generated compounds, including methional, 2-acetyl-1-pyrroline, and homofuraneol. More compounds were identified in samples from the end of the run versus beginning and middle run samples. There was not a collection time effect for any processed (heat treated) ($p>0.05$) suggesting that equipment burn-on or soiling throughout 40 h of production does not impact sensory properties of the finished product.

Flavor Formation in Skim Milk Powder and Flavor Carry-Through into Ingredient Applications

By

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Biography

Alissa Dawn Caudle was born on February 8, 1981 to Roger and Cindy Caudle. She grew up in Hays, a small town in the foothills of North Carolina, with her parents and younger brother, Drew. Alissa attended North Wilkes High School, where she was active in many activities, including cheerleading, softball, and Beta Club. After graduating as valedictorian of her high school class in 1999, Alissa continued her education at North Carolina State University where she received a Bachelor's degree in Microbiology with a minor in Genetics in May, 2003. During her senior year at NCSU, she worked as a lab assistant in a food microbiology lab, where she discovered her interest in food science. Alissa chose to stay at NC State to pursue her Master's degree in Food Science with a minor in Food Safety. During her two years in the department, Alissa was an active member of the Food Science Club, serving as the Activities Co-Chair her last year. After completing her Master's degree, Alissa will be moving to Winston-Salem, NC to begin her career as a Food Technologist at Golding Farms Foods, Inc.

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Table of Contents

List of Tables vi
List of Figures viii
Chapter 1: Introduction 1
Chapter 2: Review of Literature 4
2.1 Introduction 5
2.2 Processing and Production 6
2.3 Sensory Analysis 14
2.4 Instrumental Analysis of Flavor 18
2.5 Sensory and Instrumental Analysis of SMP 22
2.6 Storage of SMP 26
2.7 Product Applications 29
2.8 Conclusions 31
2.9 References 32
Chapter 3: Influence of Flavor Variability in Skim Milk Powder on Consumer Acceptability of Ingredient Applications 49
3.1 Abstract 51
3.2 Introduction 52
3.3 Materials and Methods 53
3.3.1 SMP selection 53
3.3.2 Proximate Analysis 54
3.3.3 Instrumental Volatile Analysis 54
3.3.4 Preparation of Ingredient Applications 55
3.3.5 Consumer Acceptance Testing 58
3.3.6 Statistical Analysis 58
3.4 Results and Discussion 58
3.4.1 Descriptive Sensory Analysis and Proximate Analysis 58
3.4.2 Volatile Analysis 59
3.4.3 Consumer Acceptance 60
3.5 Conclusions 63
3.6 Acknowledgements 64
3.7 References 65
Chapter 4: Flavor Formation in Skim Milk Powder Throughout Production 79
4.1 Abstract 81
4.2 Introduction 82
4.3 Materials and Methods 85
4.3.1 Samples 85
4.3.2 Proximate Analysis 86
4.3.3 Descriptive Analysis 86
4.3.4 Direct Solvent Extraction 87
4.3.5 Solvent Assisted Flavor Evaporation (SAFE) 88
4.3.6 Gas Chromotography – Olfactometry/ AEDA 89
4.3.7 Gas Chromotography Mass Spectometry 90

4.3.8 Compound Identification **90**
4.3.9 Compound Quantification **91**
4.3.10 Solid Phase Microextraction (SPME) **91**
4.3.11 Statistical Analysis **92**
4.4 Results and Discussion **93**
4.4.1 Proximate Analysis **93**
4.4.2 Sensory Analysis **93**
4.4.3 GC-O post peak intensity **94**
4.4.4 AEDA **95**
4.4.5 Quantification **97**
4.4.6 SPME **98**
4.5 Conclusions **98**
4.6 Acknowledgements **99**
4.7 References **100**

List of Tables

2.1 Average Composition of Cow's Milk **37**

2.2 Utilization of low-, medium, and high-heat milk powder **37**

2.3 Preparation of Reference Materials for Descriptive Sensory Evaluation of Nonfat Dried Milk **38**

2.4 Descriptive Lexicon for Cheddar Cheese **40**

2.5 Neutral/basic aroma-active compounds of goat cheese during GC/O **47**

3.1 Descriptive sensory language for rehydrated skim milk powders **68**

3.2 Vanilla Ice Cream Formula **69**

3.3 White Chocolate Formula **70**

3.4 Hot Cocoa Formula **71**

3.5 Descriptive sensory profiles of selected SMP **72**

3.6 Relative abundance of volatile compounds in SMP **73**

3.7 Consumer acceptability of reconstituted SMP and fluid skim milk **74**

3.8 Consumer acceptability of vanilla ice creams made with different SMP **75**

3.9 Consumer acceptability of strawberry yogurts made with different SMP **76**

3.10 Consumer acceptability of hot cocoa made with different SMP **77**

3.11 Consumer acceptability of white chocolate made with different SMP **78**

4.1 Descriptive sensory language for fluid milk and rehydrated SMP **105**

4.2 Proximate analysis for samples across SMP Production **106**

4.3 Microbiological analysis for samples across SMP production **107**

4.4 Sensory attributes of SMP throughout the production process **108**

4.5 Sensory attributes of each sample across SMP production **109**

4.6 Summary of aroma-active compounds identified in SMP throughout processing (beginning of run) **110**

4.7 Summary of aroma-active compounds identified in SMP throughout processing (middle of run) **112**

4.8 Summary of aroma-active compounds identified in SMP throughout processing (end of run) **114**

4.9 Quantification of selected compounds across SMP production **116**

4.10 Quantification of selected compounds across SMP production **117**

4.11 Quantification of selected compounds throughout SMP production **118**

4.12 Relative Abundance of compounds identified by SPME **119**

List of Figures

2.1 Skim Milk Powder Processing Flow Chart **42**

2.2 Diagram of a two-phase Spray Dryer **43**

2.3 Example of Dairy Grading Scorecard for Dried Milk **44**

4.1 Diagram of a two-phase Spray Dryer **103**

4.2 Product sampling points **104**

Chapter 1

Introduction

Introduction

Skim milk powder (SMP) is one of the most commonly produced dry milk products in the United States (Caric, 2004)). It is often consumed directly, but it is also a commonly used ingredient in many other foods to improve the body, texture, and flavor of the product (Karagul-Yuceer et al., 2002). Very large quantities of dried milk are produced, and many of them can be directed towards specific end product use through the drying process. The process used to dry milk products is usually spray drying. The drying process, in its simplest form, is the addition of heat above ambient temperature (150ºC to 220ºC) to small droplets of fluid milk (<300μm) to remove the moisture. The skim milk powder production process begins with skimming, standardizing, and heat treating/pasteurization of fluid milk. After these initial steps to the fluid milk, it is concentrated by evaporation to 45%-55% total solids before entering a spray drier.

The processing of SMP is important to the flavor production of the product. Sensory defects in dry milk products, including dry skim milk can be due to fluid milk quality, processing, handling, drying method, and inappropriate storage (Bodyfelt et al., 1988). Drake and others (2003) developed a defined sensory language for SMP and documented wide flavor variability (including non-fluid milk or off-flavors) among fresh (< 3 mo) reconstituted low heat SMP. Although grading and judging was previously used to determine the "goodness" and "badness" of dairy products, the data produced is not continuous and cannot be analyzed statistically for this same reason (Drake and Civille, 2003). However, descriptive sensory analysis cannot be translated into consumer like or dislike. For this reason, it is important to conduct affective tests by which quantitative data is generated by panelists that are not trained and are generally normal consumers of a product. These tests measure intensity of a flavor attribute, degree of liking of a flavor attribute, and overall liking of a product. The overall preference between products can also be determined using this method (Lawless and Heymann, 1999).

Fluid skim milk is mild and bland in flavor, and SMP should ideally exhibit a similar flavor (Shiratsuchi et al., 1994a,b). Because SMP is used so widely, flavor, flavor stability and off-flavor carry-through in ingredient applications is crucial information to determine usage and storage conditions (Ulberth and Roubicek, 1994). The use of sensory and instrumental analysis techniques throughout SMP production to document where flavor occurs in the drying process will enhance our understanding of SMP flavor development, a crucial property as this product becomes more widely used in ingredient applications, nationally and globally. The objectives of this study were to determine how flavor variability in SMP impacts consumer perception of SMP ingredient applications and to document flavor formation in SMP throughout the production process.

Chapter 2

Literature Review

Introduction:

Skim milk powder is the most commonly produced dry milk product in the United States (Caric,1993; USDEC, 2000). It is often consumed directly, but it is also a commonly used ingredient in many other foods to improve the body, texture, and flavor of the product (Karagul-Yuceer et al., 2002). Because perception of food depends greatly on taste, it is important that individual ingredients have an acceptable flavor. The quality of the raw milk is a determining factor in the flavor attributes of SMP, but the processing steps that produce milk powder can alter those characteristics and affect the taste (Shiratsuchi et al., 1994b).

The Food and Drug Administration has two definitions for nonfat dry milk. The first definition is:

> Nonfat dry milk is the product obtained by removal of water only from pasteurized skim milk. It contains not more than 5% by weight of moisture and not more that 1.5% by weight of milkfat and it conforms to the applicable provisions of 21 CFR part 131 "milk and cream" as issued by the Food and Drug Administration. Nonfat dry milk covered by these standards shall not contain nor be derived from dry buttermilk, dry whey, or products other than skim milk, and shall not contain any added preservative, neutralizing agent, or other chemical. (USDA effective 1996).

The second definition allows for addition of Vitamins A and D.

The definition for Dry Whole Milk is:

> Dry whole milk is the product obtained by removal of water only from pasteurized milk, as defined in 131.110(a), which may have been homogenized. Alternatively, dry whole milk may be obtained by blending fluid, condensed, or dried nonfat milk with fluid, condensed, or dried milks as appropriate, provided the resulting dry whole milk is equivalent in composition to that obtained by the method described in the first sentence of this paragraph. It contains the lactose, milk proteins, milkfat and milk minerals in the same relative proportions as the milk from which it was made. It contains not less than 26% but less than 40% by weight of milkfat on an as is basis. It contains not more than 5% by weight of moisture on a milk-solids-not-fat basis.

Other dry milk products used less frequently than milk powders include dry buttermilk, dry whey, dry cream, edible dry casein, and others. Each product is distinct in its characteristics. Fresh dry whole milk has a clean, rich, sweet flavor ideally similar to whole milk, while dry skim milk ideally has the characteristics of fluid skim milk with slightly more cooked flavors (Drake et al., 2003). Sensory defects in dry milk products, including dry skim milk and dry whole milk, can be due to fluid milk quality, processing, handling, drying method, and inappropriate storage (Bodyfelt et al., 1988).

Processing and Production:

Milk is a complex fluid primarily composed of lipids, proteins, and carbohydrates. The composition varies between species of origin, but cows' milk is consumed more than any other type of milk and all references generally refer to cows' milk. The average composition of cows' milk can be found in Table 1. Carbohydrates are found in milk in the form of lactose, which is the most abundant solid component in milk. Lactose is a disaccharide of α-D-glucose and β-D-galactose existing in 3 forms, α-lactose monohydride and α- and β- lactose anhydrous forms. The primary role of lactose in milk is in the colligative properties, but it also provides a source of dietary energy and can aid in calcium absorption.

Proteins are another key constituent in milk. There are two types of proteins found in milk, whey proteins and caseins. Caseins make up most (>80%) of the total protein composition of milk. They are globular proteins that have 5 subdivisions, $\alpha_{s1,}$ α_{s2}, β-, γ- , κ-caseins. Milk caseins form a micelle structure with each other and with calcium phosphate. During processing many of the changes in milk involve changes and interactions of the casein micelles. Gelation at reduced pH in cheese production is an example of a desirable reaction. Whey proteins are made up of β-lactoglobulins and α-lactalbumins, protease-peptones, blood-derived proteins, serum albumin, and immunoglobulins. They do not strongly aggregate and do not interact with other proteins readily. Beta-lactoglobulin is highly susceptible to denaturation at high temperatures, but α-lactalbumin is more heat stable (Varnum and Sutherland, 1994).

Milk fat is a complex part of milk composition. Triacylglycerols make up about 98% of milk fat, and di- and monoacylglycerols account for another 1%. Phospholipids, cholesterol, and cholesterol esters can also be found at very small concentrations. The fat content of milk highly affects the aqueous phase of the milk. Density differences enable milk fat to be separated from the fluid phase during processing. The triacylglycerols of milk are an energy source for humans and provide vitamin A precursor, vitamin D, and βcarotene in the diet. Small concentrations of minerals, enzymes, and vitamins can be found in milk also (Varnum and Sutherland, 1994).

Raw milk is transported and stored in bulk at refrigeration temperatures, sometimes for as long as 4 days. Because even proper cold storage provides a chance for microbial contamination and spoilage, a heating step is part of the subsequent processing of milk to help ensure the safety of the product and to prolong shelf life. Pasteurization is the most common heat treatment method for milk. There are three different time/temperature combinations that have been approved for pasteurization of milk in the United States. Low temperature-long time (LTLT) pasteurization, also called vat pasteurization, is done at 62.8°C for 30 minutes. High temperature-short time pasteurization (HTST) is 71.1°C for 15 seconds. Both of these are equivalent processes for the elimination of pathogenic bacteria in milk. Pasteurized milk is not sterile and requires refrigeration to further prolong shelf life. A high demand for a shelf stable milk product was met in many countries by Ultra-heat-temperature-treated (UHT) milk. These processes vary between countries from 130-150°C for 1-4 seconds. UHT milk is microbiologically stable at room temperature if packaged aseptically, but is more likely to have chemical deterioration due to the high temperature of processing (Varnum and Sutherland, 1994).

Pasteurization has a history in the United States. In 1924, the Public Health Service developed the Standard Milk Ordinance to be voluntarily adopted by State and Local Milk Control Agencies. The goal of the Standard Milk Ordinance was to provide guidelines for the safe processing and handling of milk to decrease the incidence of foodborne disease associated with dairy products. After 24 revisions, the ordinance is now called the Grade “A” Pasteurized Milk Ordinance (PMO). This ordinance regulates the production, transportation, processing, handling, sampling, examination, labeling, and sale of Grade “A” milk and milk products, as well as the inspection of dairy farms, plants, and transfer stations. If adopted by state or local agencies, people who violate the PMO are subject to legal actions, which are recommended in the revised ordinance (Grade “A” Pasteurized Milk Ordinance, U.S. Dept. of Health and Human Services and U.S. Food and Drug Administration, 2001 Revision).

Very large quantities of dried milk are produced, and many of them can be directed towards specific end product uses through the drying process. The process used to dry milk products is usually spray drying. Three different types of skim milk powder can be produced; low heat, medium heat, or high heat powders (Varnum and Sutherland, 1994). The heat treatment of the milk before it is spray-dried is the criteria used to determine which category the skim milk powder will be in. The treatments are 72°C/ 15s, 82°C/ 3min, and 82°C/ 30 min for low, medium, and high heat, respectively (Rehman et al., 2003). Table 2 shows some of the different applications using each type of skim milk powder.

The drying process, in its simplest form, is the addition of heat above ambient temperature (150ºC to 220ºC) to small droplets of fluid milk (<300µm) to remove the moisture. The skim milk powder production process begins with skimming, standardizing, and heat treating/pasteurization of fluid milk. After these initial steps to the fluid milk, it is concentrated by evaporation to 45%-55% total solids. The evaporator is the most important part of the system in respect to quality of milk concentrate and operating efficiency. There are several types of evaporators, including batch-pan, circulation evaporators, and falling film evaporators. The latter of these methods is used more frequently than the others (Varnum and Sutherland, 1994). In this type of evaporator, the milk is passed over heated surfaces and the milk flows down tubes by gravity at high velocities. The evaporator is linked to a separator and condenser, which separate the concentrated milk from vapor and recondenses the vapor to use as an energy source within the system. Then, the concentrated milk passes into the spray dryer (see Figure 2) where it is dried and cooled inside the chamber. Figure 1 shows the flow of milk through the drying process.

Whole milk powder has a minimum fat content of 26%. The method of drying is mostly the same as that for skim milk powder, but whole milk must be standardized to 3.6% milk fat before the first heat treatment, and then to 45%-50% total solids during concentration. To minimize free fat in the whole milk powder, the temperature used to spray dry whole milk is at the lower end of the temperature range used to dry skim milk. The reduced heat treatment can reduce cooked flavors in the powder and slow deterioration, making it more acceptable to consumers over longer periods of time (Varnum and Sutherland, 1994). However, the high fat content of WMP makes it very susceptible to lipid oxidation and associated off-flavors during storage. The quality of dried milk is associated with the dehydration process, as well as further handling and storage of the product and the physical and chemical changes that can occur (Roos, 2002). Spray drying can increase the shelf life of milk from several days up to two years (Kockel et al., 2002).

Some milk powders, mainly whole milk powders, are used as coffee whiteners. The drying process can have a huge effect on the ability of these powders to act as whitening agents. High evaporator preheat temperature, long holding times, and high total solids concentration negatively affect the coffee whitening potential of the milk powder. From a sensory aspect, poor stability in coffee is characterized by a greater sediment formation in milk powders (Oldfield et al., 2000).

Milk powder provides nutritional benefits to many countries, including the U.S. and is commonly consumed in many products in the human diet. Therefore, it is important to maintain the nutrient value of milk during the drying process. A relatively insignificant loss of Vitamin D3 occurred in the spray drying of whole milk (Indyk et al., 1996). Spray drying transforms milk droplets into solid particles, which each are individual powder surfaces. The compositions of powder surfaces are very different than the bulk powder, and they are very important to the functional properties of the powder. Solids are likely to segregate during the drying process, causing the outermost surface of milk powders to be covered in unprotected fat particles that are susceptible to oxidation (Esther et al. 2003).

To optimize the spray drying process, the phase transitions of milk must be understood, especially those concerning lactose (Vuataz, 2002). The method of manufacturing of whole milk powders can have a significant effect on its impact in the making of milk chocolate. It is possible that using high lactose milk powders could be a substitute for glucose in such products. It is important to understand how the addition of lactose to dry milk products would affect the properties of the powder. Chemical composition and x-ray diffraction analysis show that lactose concentration did not affect the moisture content of the powder and that composition of the powder remained steady through conventional spray drying and post process crystallization. The amount of free fat and total fat decreased as lactose concentration increased. Exposure of milk powders to high humidity caused lactose to crystallize, which resulted in increased free fat and particle density, as well as a decreased surface area (Aguilar et al, 1993).

Lactose hydrolyzed milk powders are highly susceptible to the Maillard reaction, which can lead to protein loss during storage if the product is stored at high temperature or humidity. At high humidity and temperature, freeze dried milk powders have less loss of reactive and total lysine. However, both freeze- and spray dried milks had essentially the same lysine content and protein nutritional content when stored under optimal conditions. Deterioration of protein quality was greater at water activity above 0.11 (Rawson and Mahoney, 1983). Optimal heating and cooling systems are required to ensure that spray dried milk has an acceptable flavor. Processing temperatures that are too high or low cooling temperatures, combined with long or short holding times, can ruin the flavor, appearance, or solubility of milk powders. Understanding the thermal conductivity of milk powders is important in order to find the correct holding time and temperature for processing. The thermal conductivity of milk powders was increased with increasing temperature and with bulk density. (MacCarthy 1985).

The Maillard reaction is the chemical reaction of reducing sugars, usually D-glucose, and a free amino acid or amino acid group that is part of a protein chain. The reaction can also be called non-enzymatic browning (NEB) to differentiate it from the enzyme-catalyzed browning commonly found in fresh cut fruits and vegetables. Storage can produce Maillard reaction products, which can be either desirable or undesirable flavors, aromas, or colors. These products occur when aldoses or ketoses are heated in solution with amines and both reactants disappear slowly. In such reactions, the reducing sugar reacts reversibly with the amine to produce a glycosylamine, which then undergoes Amadori rearrangement to give a 1-amino-1-deoxy-D-fructose derivative. At pH values greater than 5, the reactive cyclic compounds polymerize quickly to dark-colored, insoluble materials (BeMiller and Whistler, 1999). Maillard reactions occur faster in liquid systems than in dried foods (Baltes, 1982). In the production of some products, such as milk chocolate, caramels, toffees, and fudges, the reducing sugars react with milk proteins in desirable Maillard and caramelization reactions. Heating carbohydrates and reducing sugars, as is done in milk processing, causes a caramelization reaction. Negative aspects of Maillard reactions decrease protein quality and cause off-flavors (BeMiller and Whistler, 1996). Because milk products contain large amounts of lactose and casein, they are highly sucseptable to Maillard reactions. Results of these reactions in milk powder are loss of solubility, decreased nutritional quality, and undesirable changes in color and flavor (Renner, 1988).

Cohesion is an important property of milk powders during the spray drying process and in ingredient applications. A powder that is extremely cohesive will have reduced flowability and may stick to equipment. However, if a powder is not cohesive enough and is used in a product, it may separate from the other ingredients, causing quality or consumer acceptance problems. It is important to standardize the cohesive properties of milk powders to avoid these complications. Temperature, moisture content, particle size, and powder composition can influence the cohesion of milk powder, which can impact efficiency of the spray drying process. Contributors to cohesion other than temperature are lactose and fat content. Whole milk powder is twice as cohesive as skim milk powder because of the fat content. This difference can increase to three fold with the addition of lactose or moisture. The cohesion of both skim and whole milk powders increases as particle size decreases (Rennie et al., 1999). Whole milk powder is much more cohesive than skim milk powder, but SMP has a greater wall friction. The cohesiveness of SMP and WMP increases with temperature from 5-25ºC, and decreases again at 30-40ºC. Above 60ºC, the powder can become extremely sticky. Free fat content did not significantly affect the powder cohesion (Fitzpatrick et al., 2004).

The physical properties of milk powder due to production and processing procedures have a direct impact on the sensory quality of skim milk powders, including taste, aroma, and mouthfeel/feeling factor. Taste and aroma are strongly linked in the physiology of flavor. The basic tastes (sweet, salty, sour, bitter, and umami) are perceived by the taste buds, but volatile flavors in food are sensed by the olfactory receptors. Trigeminal feeling factors are responsible for other sensations, such as burn or prickle. All together, these factors make up the flavor of foods, including skim milk powders. Inappropriate or inaccurate heat treatments or holding times at any stage of the drying process can cause undesirable flavors and often cause inconsistencies in SMP flavor. Therefore, sensory and instrumental analysis is needed after production to optimize the skim milk powder production process.

Sensory Analysis:

There are three main types of sensory analysis for dairy products and ingredients: grading/judging, analytical tests, and affective tests. Grading/judging is used to measure the quality of a product as good or bad. Analytical tests are used to quantify the amount of a certain attribute, and affective tests are used to assess the acceptability or preference of an attribute or a product (Lawless and Heymann, 1999). The use of sensory techniques to measure the quality of dairy products dates back hundreds of years, to the early use of these products. However, dairy grading and judging did not appear until the late 1800's. In 1916, the first Students' National contest in Judging of Dairy Products was held, where butter was judged. In the following years, other dairy products, such as ice cream and cheese, were also included in the competition. Thus analysis of dairy products was born. In the early twentieth century trade names and brand names were developed for the first time, and each was given a quality grade by the USDA. The original purpose of dairy grading and judging was to determine if a product had good "eating quality", versus the product passing chemical and physical evaluation standards. In judging and grading, a judge has a score card and rates a product based on the score card (Bodyfelt et al., 1988). An example of a dairy judging score card can be seen in Figure 3. The long history of these analyses emphasizes the importance of sensory quality.

Grading and judging are based on the characteristic "goodness" of a food. An overall flavor and/or texture quality score is generated based on the presence/absence of specific pre-determined defects. Grading/judging are not accurate tools in assessing the full range of flavors associated with foods for several reasons. The tests are subjective and defect oriented, which means that rather than assessing a flavor attribute for intensity, the attribute is rated as good or bad, and these types of ratings can vary between judges. Further, what is good or bad to a trained judge does not necessarily translate to consumer like or dislike. Judging and grading data is not continuous and cannot be analyzed statistically for this same reason (Drake and Civille, 2003).

More recently, descriptive sensory analysis has begun to take the place of grading and judging. These methods seek to profile a food on all of its characteristics, which is different than judging. These types of analyses are used to evaluate the quality of a product, compare products to each other, and to understand consumer responses to different products (Murray et al., 2001). Murray et al. (2001) described several different descriptive sensory analysis techniques, including the Flavor Profile Method, Texture Profile Method, Quantitative Descriptive Analysis, Quantitative Flavor Profiling Technique, and the Spectrum Method. For each method, there is a descriptive panel which must be screened for test performance skills, and then trained at some level. The new panel either develops a sensory language for the product they are testing or adopts a language that has already been developed for such products. Training occurs in reference to the language, with attributes being described by a reference solution or common taste (Table 3, Table 4). No judgement is made to "good" or "bad"; instead, all flavors or textures are identified and their intensities are quantified.

To accurately describe off-flavors in dairy products, a full lexicon needs to be developed that identifies a descriptor for a flavor, explains the descriptor, and provides a reference for that descriptor. An example of a full lexicon can be found in Table 4: Descriptive lexicon for cheddar cheese. A developed lexicon allows sensory research to be more objective, and terms to be standardized. A lexicon is produced by first generating a rough language by round table discussion among experienced tasters. Other panelists are trained on the existing descriptors, and encouraged to volunteer new descriptive terms. The panel is presented with possible references and collectively choose the best reference for each descriptor (Drake et. al, 2003).

Flavor profiling was the first method of descriptive sensory analysis and was developed at Arthur D. Little and Co. The panel consists of four to six judges who are trained specifically for the product they are evaluating. Texture profiling is based on flavor profiling, but involves descriptive analysis of texture. Trained panelists evaluate texture from first bite until the food is completely chewed. Quantitative descriptive analysis (QDA) is a method that was developed based on Flavor profiling, but also addressed problems or issues of flavor profiling. QDA was developed by Stone and Sidel in the 1970's at Tragon Co. In QDA the language is "everyday" and non-technical. References are only used if a problem with a term is encountered. The panel leader is not an active member of the panel, but is trained in leading these types of panels and acts as a facilitator. QDA uses a panel of 8-12 people who are trained on the descriptors. The data from these panels is both quantitative and qualitative. Quantitative flavor profiling (QFP) is a modified version of QDA that uses a standardized, technical language. Also, in QFP, references are used to demonstrate the language and concepts. The Spectrum method is a widely used technique, developed by Gail Vance Civille in the 1970's, that relies on references, specialized training, and multi-product evaluation. Eight to 12 panelists are highly trained on attributes that are non-product specific. References are provided for each term, and do not change if the panel evaluates another product, though some may be added as new attributes are defined. Table 3 shows an example of descriptors and references for Spectrum testing of Skim Milk Powder. These are the methods that are widely used today, and descriptive analysis is the most useful tool in providing information on sensory attributes of food (Murray et al, 2001).

Another type of sensory testing by which quantitative data is generated is affective testing, in which the panelists are not trained and are generally normal consumers of a product. These tests are conducted in a manner that uses terms representative of the way consumers would describe a food rather than a developed lexicon used for descriptive sensory analysis. Affective tests measure intensity of a flavor attribute, degree of liking of a flavor attribute, and overall liking of a product. The overall preference between products can also be determined using this method (Lawless and Heymann, 1999).

Instrumental Analysis of Flavor:

The human nose is the most sensitive instrument used for flavor evaluation and cannot be replaced by instrumentation. However, to fully understand flavor, descriptive sensory analysis needs to be accompanied by instrumentation to identify and characterize the compounds responsible for the flavor and aroma in foods. There are several ways to analyze flavors in food. The first step is to identify the compounds present in the food matrix, so those compounds must be extracted for analysis. Methods for extraction of volatiles from foods include direct solvent extraction, distillation, and headspace analysis techniques including purge-and-trap, static, and solid-phase microextraction (SPME). For solid matrices, such as milk powders, direct solvent extraction is the simplest method for extracting volatiles. A highly volatile organic solvent, such as diethyl ether, is combined with the food matrix and, after several mixing cycles, the solvent along with the extracted volatile compounds are collected from the solid or aqueous phase by density separation. The volatile compounds can then be distilled. High vacuum distillation is a common method for further concentration of volatile compounds, as it allows a gentle heat treatment and prevents formation of heat generated artifacts. The concentrated solvent extract is then injected onto a gas chromatograph (GC) for separation and identification.

Solid-phase microextraction (SPME) is a technique used for the rapid, solventless extraction or preconcentration of volatile and semi-volatile organic compounds from headspace. SPME utilizes the principle of partitioning the organic component of a sample between a bulk aqueous or vapor phase and the thin polymeric films coated onto fused silica fibers in an apparatus specifically for this technique. This technique is different from other extraction methods because the method remains the same for liquid, solid, and gaseous samples. The device is a modified syringe that allows the plunger to be held at a specific length for extended periods of time while inside the injector port of the GC. The barrel of the syringe is a stainless steel port containing the silica fiber, which is coated at the bottom with the thin polymeric film. The introduction of this fiber directly into the headspace of the sample collects the volatile components which are then subsequently injected into the injection port of a gas chromatograph for separation and identification of the headspace volatiles of the sample (Harmon, 1997).

Gas chromatography/mass spectrometry (GC/MS) is the most common method used to separate and identify volatile compounds. GC/MS combines chromatographic separation with a multichannel detector to produce qualitative and quantitative information. After a small sample is injected onto the GC, the volatiles travel through a coiled column where individual compounds are separated based on their specific chemical properties. The separated compounds then enter the MS where they are bombarded by electrons to produce ion fragments. Different compounds will produce different characteristic ion fragments. The fragmentation patterns can be compared to existing databases to identify which compounds are present in the food (Huston, 1997). There are two basic types of MS: quadrupole and ion trap. The quadrupole MS utilizes a quadrupole electric field to affect the motion of ions. An ion trap MS stores and concentrates ions prior to mass analysis, and therefore is more sensitive than a quadrupole apparatus (Huston, 1997). Another, less expensive type of detector is a flame ionization detector or FID. Using an FID, ions are passed through a flame, which pushes the ions through a detector. Because the ions are not differentiated through a magnetic pole as they are in MS, the flame ionization detector does not infer what the compound is, only that a compound is present, making it much less sensitive than MS (Huston, 1997).

Though instrumental analysis and descriptive sensory analysis are powerful tools on their own, they are even more powerful when used in combination. By relating instrumental data to descriptive sensory data, researchers are able to determine which compounds are responsible for flavor in food (Drake and Civille, 2003). This goal is challenging because instruments may find many compounds that are present at levels below the human threshold and therefore do not contribute to the flavor of the food. Differentiating the small number of compounds that are present in foods above the human threshold from those that are not becomes challenging when there are so many compounds in the product (Drake and Civille, 2003). One such method that assists in linking instrumental data to the sensory perception of flavor is gas chromatography-olfactometry (GC-O). This method of evaluating odor compounds involves splitting the

GC effluent between a traditional detector and a human nose. The main reasons for using such techniques are to provide additional confirmation of compound identity, and to determine aroma quality of individual compounds and their significance to the flavor of a food. The procedure for GC-O combines gas chromatography with a human sniffer to determine what compounds are released from the column, aroma quality, and at what intensity the odor of the compound is found. The human sniffer records the aroma and intensity as compounds elute and a chromatogram is established by the traditional detector, usually an FID (Mistry et al., 1997). A table can then be generated identifying the retention time (RT) or retention index (RI) of a compound and its aroma quality and intensity. Table 5 shows an example of this type of table.

GC-O has been applied to several dairy products (Karagul-Yuceer et al., 2002, 2003, 2004; Carunchia Whetstine et al., 2003). Friedrich and Acree (1988) reported that seven common odor-active compounds could be identified in raw milk. Those were dimethylsulphone, ethyl butanoate, ethyl hexanoate, heptanal, indole, nonanal, and 1-octen-3-ol. Fifteen compounds were detected in milk after it was heated, and four of those were unique to the heated milk only. Those four compounds were hexanal, 2-nonanone, benxothiazole and δ-decalactone. Other odor-active volatile compounds were found to be common in other dairy products, such as cheese and fermented products (Friedrich and Acree, 1988). Several aroma active compounds were found in skim milk powder by GC-O (Karagul- Yuceer et al., 2001,2002). Compounds identified by GC-O included p-cresol, skatole, octanoic acid, nonanoic acid, decanoic acid, and dodecanoic acid. All of these acid compounds gave soapy/waxy/rubbery odors. Butanoic and hexanoic acids smelled cheesy and fecal (Karagul-Yuceer et al., 2002)

Sensory and Instrumental Analysis of SMP:

Fluid skim milk is mild and bland in flavor, and skim milk powder should ideally exhibit a similar flavor (Shiratsuchi et al., 1994a,b). Because skim milk powder is used so widely, flavor, flavor stability and off-flavor carry-through in ingredient applications is crucial information to determine usage and storage regimes (Ulberth and Roubicek, 1994). The flavors that are the most intense are those that are associated with high heat treated powders, such as cooked/ sulfurous and caramelized flavors (Karagul-Yuceer et al., 2001).

Lang et al. (1976) studied the influence of composition on the sensory perception of fluid skim milk. Researchers found that panelists could not detect significant differences in samples with different fat percentages, but could detect differences between samples with different solids-not-fat (SNF) content. The differences were not detected after pasteurization at 73.8°C but were easily detected in those products pasteurized at 85°C and homogenized. The samples differing in SNF content were most frequently described as watery followed by heated. The same researchers studied the addition of stabilizers and emulsifiers on the sensory quality of milks. The addition of stabilizers was detected at 200 and 300 ppm in samples pasteurized at 85°C, and flavors were described as watery and heated. Emulsifiers had an impact on sensory quality at 400 ppm in milks containing 0.1% fat, but not those containing 0.5% fat. The different samples were most frequently described as watery and oxidized (Lang et al., 1976).

Flavor consistency has been a problem in many dried dairy products, but no descriptive sensory research on dried milk had been performed prior to Drake et al.

(2003), who developed a standardized language for skim milk powder and dried dairy ingredients (Table 3). Low-, medium-, and high-heat powders were used to develop the dried dairy ingredients to get a broad language that encompassed all of the flavors in the powders. Panelists were able to differentiate flavors such as caramelized/butterscotch and sweet aromatic/cake mix, which were originally grouped together. They also differentiated animal-type flavors into two groups – animal/barny and animal/wet dog, and they differentiated fried fatty/painty from fishy. Lexicon development is a powerful research tool for the differentiation of off-flavors in milk powders, allowing for subsequent identification and quantification of off-flavor causing compounds by instrumental analysis.

Different volatile chemical compound classes were found in different amounts in fresh flavored skim milk powder versus an off-flavored skim milk powder. Fatty acids and lactones were found in higher concentrations in the fresh skim milk powder compared to stored skim milk powder. Chemicals that were found in higher amounts in the off flavored milk powder included aldehydes, alcohols, esters, and nitrogen-containing compounds. Specific aldehydes found in off-flavored skim milk powders were octanal, (E)-2-octenal, (E,Z)-2,6,nonadienal, salicylaldehyde, dodecanal, a tridecenal isomer, pentadecanal, and (Z)-9-octadecenal. These were thought to greatly contribute to off-flavor because the off-flavored skim milk had the odor of aldehydes and (X)-9-octadecenal was ten times more abundant in the off-flavored powder. Some alcohols, 2-methyl-1-propanol, 1-methyl-4-(methylethy)-(E)-2-cyclohexanol, 1-tetradecanol, and 1-docosanol were only detected in the off-flavored powder. The difference in N-containing compounds was quite significant. There were not abundant amounts in the fresh powder, but levels of 1H-pyrrole, indole, and 2-pentadecanone O-methyloxime increased three fold in the off-flavored powder. Through gas chromatography-olfactometry, researchers concluded that tetradecanal, β-ionone, and benxothiazole were responsible for the off flavors in the skim milk powder. They had sickening/aldyhydic, fragrant/haylike, and sulfuric/quinoline-like odors, respectively (Shiratsuchi et al., 1994a). Descriptive sensory analysis was not used to further clarify instrumental results.

Karagul-Yuceer et al (2001) studied the aroma-active components of fresh nonfat dry milk using both descriptive sensory and instrumental analysis. Quantitative descriptive analysis of skim milk powders showed that all aroma descriptors ranged between 0 and 4.25 in intensity on a 10 point scale. These low intensities were expected due to the mild nature of skim milk flavor. High heat treated milk powders exhibited more intense cooked/sulfurous and caramelized flavors than did low or medium heat treated powders. Free fatty acids caused by fat hydrolysis, such as butanoic, pentanoic, and hexanoic acids, were found as major contributors to skim milk powder flavor, as well as thermally induced compounds such as 2-acetyl-1-pyroline, 2-acetyl-2-thiolozine, and 2-acetylthiazole. The latter three compounds exhibit popcorn-like notes. Free fatty acids commonly contribute to cheesy flavor in dairy products. Lactones were concluded to be contributors to sweet and fatty flavors. These included δ-decalactone and γ-dodecalactone. Other aroma-active compounds that were detected in the fresh SMP were furaneol, maltol, sotolon, and vanillin. All of these compounds were detected at higher levels in the high-heat treated powders and these compounds are heat-generated compounds.

A subsequent study was performed on skim milk powders that exhibited off-flavors, ranging from 3 months to 25 years in age. Aldehydes, ketones, free fatty acids, lactones, and alcohols were found to contribute to undesirable flavors over storage time. *o*-Aminoacetophenone was identified in all samples as a foxy note. Animal-like, cowy, or fecal flavors were indicated by sensory evaluation in all samples. Instrumental analysis concluded that *p*-cresol and skatole were possible contributors to these flavors. Researchers concluded that these and a wide variety of other compounds were associated with off-flavors of SMP (Karagul-Yuceer, 2002). These same researchers conducted a model system sensory study to confirm the key volatile compounds that contributed to the flavor of fresh SMP (Karagul-Yuceer et al., 2004). In the study, 12 odor-active compounds from SMP and one from fluid skim milk were selected based of flavor dilution factors and GCO. Thresholds for the 13 compounds were found using ascending forced choice analysis, and these thresholds were used to spike the compounds into a model system, which was rehydrated milk retentate (RMR). RMR was chosen because it is odorless and has characteristics similar to milk. There were two parts to the model systems study. First, model mixtures were prepared and compared for overall similarity to rehydrated fresh SMP. Then, compounds selected as primary contributors to the aroma of fresh SMP were added to the RMR in different combinations and evaluated by a trained panel. Based on the characteristics described by the panelists, the amount of some of the compounds was adjusted until the matrix was overall very similar to fresh SMP (9.0 $\pm$ 0.1 out of 10). Results showed that there was a matrix effect on the threshold of compounds between water and skim milk, but the effect varied between compounds (Karagul-Yuceer et al., 2004)

In general, off-flavors are caused in milk powders due to lipid oxidation as well as Maillard reactions (Shiratsuchi et al., 1994b, Karagul-Yuceer et al., 2002, Ulberth and Roubicek, 1994, Tamsma et al., 1974). Measurement of deterioration in milk powders is done through analysis of certain volatile compounds. The compounds generated by lipid oxidation in milk powders have been analyzed through headspace gas-chromatography. Specifically, research has made it possible to detect and quantify straight-aldehydes that can cause off-flavors (Ulberth and Roubicek, 1994). Previous studies have shown that the flavor of milk powders containing 1-2% fat score as well as those with as much as 3% fat. However, milk powders with considerable lower fat content (.1%) do not score as well. These lowfat dried milk products, with fat contents lower than whole milk powder, have less oxidative flavor when reconstituted after storage (Tamsma et al., 1974). It is, therefore, important to document the storage conditions which are optimal for keeping SMP free of off-flavors for the longest period of time.

Storage of SMP:

It is generally accepted that stored milk powder will not be exactly the same, chemically and physically, as fresh milk powder, but it is also expected that the differences will not be large enough to render the milk powder unacceptable to consumers after storage (Hough, 2002). Milk powders are free flowing, but have been found to lose this free flowing quality during handling and storage, mainly due to cake formation of the powders. Temperature, pressure, and moisture content of skim milk powders was responsible for faster caking, and therefore, faster loss of the free flowing nature (Ozkan et al., 2003). Some of the changes that occurred in whole milk powders were loss of free fat content, and increase in insolubility index and peroxide value over the time of storage (Celestino et al., 1997).

During the storage of ultra high temperature processed fluid milk, off-flavors developed during storage at higher temperatures and were due to enzymatic and oxidative reactions. Also, the physio-chemical properties were affected. Longer storage of powders resulted in greater sediment and lower pH in reconstituted UHT milks as well as increased rates of enzymatic and oxidative reactions (Celestino et al., 1997). Milk powders contain high concentrations of lactose, which makes them highly susceptible to the Maillard reaction (De Block et al., 2003). Lipid oxidation reaction products, specifically hexanal, are found in higher concentrations in stored powders than in fresh ones (Ulberth and Roubicek, 1995). The storage time of fluid milk before the drying process can affect the sensory quality of the resulting powder. Free fatty acid content has been found to be greater in powder made from stored fluid milk compared to that made from a fresh raw milk control (Celestino et al., 1997).

Dry milk products are supposed to have long storage potential, so the storage conditions are very important. Not only do storage conditions include temperature and moisture content, but the type of packaging is also important. For example, it is important that the packaging is a good barrier to outside moisture (Rehman et al., 2003). Research has shown that there is not a significant difference in physical properties of skim milk powders stored in plastic bags versus paper bags, but that other properties were in fact affected by the packaging. Plastic bags are better in terms of maintaining water activity, flowability, and solubility of the product, while paper bags are better for preserving titratable acidity (Rehman et al., 2003). Lactose crystallization may occur if milk powders are stored at high humidity or if they are submitted to heat treatments after a period of storage (Vuataz, 2002). Another type of reaction that can occur in milk powders is light-induced oxidation. Two reactions are responsible for light-induced deterioration; lipid oxidation and amino acid degradation. Sulfur compounds such as thiols, sulfides and disulfides are contributing factors to light-induced reactions (Reineccius, 1999). Thus, milk powders, like fluid milk, should be protected from light exposure.

Temperature and humidity during processing and storage can also change the monosaccharides in milk powders. Prolonged storage considerably altered the galactose content of dried milk. Maillard reactions occurred during storage of milk powders, but did not explain the increase in glucose concentration. Both glucose and galactose could be products of lactose hydrolysis over storage time. Tagatose also increased over storage time, and is due to isomeration of galactose. It is thought that the tagatose is a byproduct of the Maillard reaction. Another compound, 3-deoxypentulose, was also detected after storage, but is produced from lactose and can be produced during milk sterilization. Both compounds are deterioration indicators (Troyano et al., 1994)

Studies have also characterized the effects of storage on sensory quality. Driscoll et al. (1985) reported an overall decrease in milk powder acceptability during storage as a direct reflection of the development of an oxidized/stale flavor. The research compared the flavor of fresh skim milk powders (regular and instant) and the effects of the type of packaging, the atmosphere in the container, and storage temperature on the development of off-flavors over storage time. No differences were reported between the regular SMP and the instant SMP. However, the samples stored in Polybags were less acceptable than those stored in cans, and samples held at the highest temperature, 32°C, scored lower than those stored at other temperatures. The Polybags were more permeable, so oxygen was present inside, allowing for oxidation reactions inside the bags. Though there were differences detected, they were only found after 42 months, and only in the containers stored at the highest temperature use in the study (32°C). A temperature study showed the greatest amount of difference between stored and fresh samples. After only 6 months of storage, samples stored at 32°C showed significant differences when compared to samples stored at 10°C or 21°C. After 12 months, there were substantial acceptability differences in the samples (Driscoll et al., 1985).

Product Applications:

Skim milk powder is one of the most widely used dry ingredients in a number of food products (Caric., 1993; USDEC, 2000). Functionality attributes of SMP include water absorption and binding, foaming, emulsification, solubility, viscosity, gelation, colloidal properties, and heat stability. The quality of SMP has a direct impact on the quality of the finished product, as well as its protein content (Varnum and Sutherland, 1994). Table 2 shows the types of milk powders used for various products. In chocolate production, the amount of free fat available in the powder has the strongest impact on the physical and rheological properties of the chocolate (Liang and Hartel, 2004). Milk fat concentrations that are too high can make chocolate unacceptably hard. However, up to 30% of the cocoa butter can be replaced with milk fat (Attaie et al., 2003). Dried milk ingredients also affect the consumer acceptability scores of chocolate. In consumer tests, the chocolates that scored the highest acceptability scores were those made from milk powders containing high amounts of free fat, such as skim milk powder with anhydrous milk fat (Bolenz et al., 2002). Milk fat also makes a difference in ice creams made with vanilla flavor. Consumer liking increased as the total milk fat concentration increases in such products (Li et al.. 1997). In another study, where researchers tried to optimize the ingredients for soft ice cream, consumers also preferred those products in which the cream and skim milk powder concentrations were increased (Sharma et al., 2003).

The viscosity of a product greatly affects the texture and mouthfeel, which in turn, can affect the sensory perception. Viscosity is caused by the interaction of the milk components in the milk powder, so optimizing those components for food applications is very important in adjusting the viscosity to an acceptable level. This can be done through concentration methods and the spray or roller drying process (Guzman-Gonzalez et al., 2000). In yogurt products, milk powders can serve to increase the percent solids in the product, making the texture more appealing (Drake et al., 2000). Milk powder is also often used as a coffee whitener (Oldfield et al., 2000). Skim milk powder was used in the production of fruit preserves and the sensory changes were observed. They were found to alter the preserves to a degree that sensory acceptability was decreased. The flavor and spreadability was acceptable to panelists, but the color and texture were not (O'Beirne and Kelly, 1985).

Milk powders have been used in meat products, such as sausages, in an attempt to use non-meat proteins to increase yield and decrease ingredient costs. The milk proteins act as emulsifiers, and bind water and fat. Samples of SMP with 1.5% milk protein were the most similar to controls, but differences between samples with increased milk protein concentration were significant. With increased milk protein concentration, panelists described higher off flavor and stickiness, and lower meat flavor, firmness and elasticity. There was a higher cooking loss in sausages with skim milk powder added. A 1:1 mixture of whey protein and skim milk powder was the most similar to the control sausages in sensory attributes and cooking loss (Ellekjaer et al., 1996)

Conclusions:

Sensory and instrumental analyses are crucial to understanding SMP flavor. The raw product, processing procedures, and storage conditions are all factors effecting SMP quality. Many previous studies have been focused on the flavor of SMP, specifically documenting differences in fresh SMP and stored SMP. SMP is widely used as an ingredient in many products. However, no research has addressed the carry-through of SMP flavor into ingredient applications. The first objective addressed by the following research is to determine the effect of SMP flavor on consumer acceptability of products using SMP as an ingredient. After determining the importance of flavor, it is also crucial to understand what processing steps play a role in forming SMP flavor. The second objective is to determine where flavors are formed during SMP processing. Both studies will help understand the importance of SMP flavor in the food industry.

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Table 1: Average Composition of Cow's Milk

Component	Percentage	Percentage of Solids
Lactose	4.8	37.5
Fat	3.7	28.9
Protein	3.4	26.6
Non-protein Nitrogen	0.19	1.5
Ash	0.7	5.5
Total Solids	12.79	

Varnum and Sutherland (1994)

Table 2: Utilization of low-, medium, and high-heat milk powder

Product	Powder type	Attributes
Reconstituted Milk and Milk Drinks	Low/medium heat	High solubility, minimal cooked flavor
Recombined evaporated milk	High heat	Heat stability, high viscosity
Cheese	Low heat	Rennetability
Ice Cream	Medium heat	Emulsification, foaming, water absorption
Confectionery	High heat	Texture modification, water absorption
Comminuted meat	High heat	Emulsification, gelation, water absorption
Baked goods	High heat	Water binding, texture modification

Varnum and Sutherland (1994)

Table 3: Preparation of Reference Materials for Descriptive Sensory Evaluation of Nonfat Dried Milk

Descriptor	Reference	Preparation
Cooked/sulfurous	-Heated milk	-Heat pateurized skim milk to 85ºC for 45 min
Caramelized/butterscotch	-Autoclaved milk -Caramel syrup	- Autoclave whole milk at 121º for 30 min - Dilute a tablespoon of caramel syrup in 400mL ski milk
Sweet aromatic/ cake mix	-Pillsbury-White cake mix -vanillin	-dilute 5mg of vanillin in skim milk
Cereal/grass-like	-breakfast cereals (corn flakes, oat and wheaties)	-soak one cup cereal into three cups milk for 30 min and filter to remove cereals
Barny	-*p*-cresol	-20 ppm in skim milk
Brothy/potato-like	-Kroger-Canned white potato slices -methional	-remove the sliced potatoes from the broth -few drops of 20 ppm methional in methanol in sniffing jars
Animal/gelatin-like/wet dog	-Knox-unflavored gelatin	-dissolve one bag of gelatin (28g) in two cups of distilled water
Milkfat/lactone	-Heavy cream -delta dodecalactone	40 ppm on filter paper
Fried fatty/ painty	-(E,E)-2,4-decadienal	-2 ppb in skim milk
Fishy	-Fresh fish with skin -Canned tuna juice	
Mushroom/metallic	-fresh mushroom	-slice fresh mushroom in skim milk for 30 min and filter to remove mushroom slices
Papery/cardboard	-cardboard paper	-oak pieces of cardboard paper in skim milk overnight
Burnt/charcoal	-over toasted bread slice	
Vitamin/rubber	- Enfamil- liquid Polyvisol vitamins	
Diacetyl	-diacetyl	-Diacetyl, 20 ppm on filter paper
Earthy/musty	-potting soil, odor reminiscent of damp basement	
Sweet taste	-sucrose	-5% sucrose solution

Table 3 continued:

Salty	-NaCl	-2% NaCl solution
Sour	-citric acid	-1% citric acid solution
Bitter	-caffeine	-0.5% caffeine solution
Umami	-monosodium glutamate	-1% MSG in water
Astringent	-tea	-soak 6 tea bags in water for 10 min

Karaguul-Yuceer, 2003

Table 4: Descriptive Lexicon for Cheddar Cheese

Term	Definition	Reference
Cooked	Aromatics associated with cooked milk	Skim milk heated to 85ºC for 30 min
Whey	Aromatics associated with Cheddar whey	Fresh Cheddar whey
Diacetyl	Aromatic associated with diacetyl	Diacetyl, 20ppm
Milkfat/Lactone	Aromatics associated with milkfat	Freash coconut meat, heavy cream, δ dodecalactone, 40ppm
Fruity	Aromatics associated with different fruits	Fresh pineapple
Sulfur	Aromatics associated with sulfurous compounds	Boiled mashed egg
Free fatty acid	Aromatica associated with short chain fatty acids	Butyric acid, 20ppm
Brothy	Aromatics associated with boiled meat or vegetable stock	Canned potatoes, qylers low sodium beef broth cubes, methional, 20ppm
Nutty	The nut-like aromatic associated with different nuts	Lightly toasted unsalted nuts, wheat germ, unsalted wheat thins, roasted peanut oil extract
Catty	Aroma associated with tom-cat urine	2 mercapto-2 methyl-pentan-4-one, 20ppm
Cowy/phenolic	Aromas associated with barns and stock trailer, indicative of animal sweat and waste	p-cresol, 160ppm, bandaids
Age**	Flavors indicated age in Cheddar cheese	Aged Cheeddar cheese (1 y or longer)
Yeasty*	Aromatics associated with fermenting yeast	Raw yeast dough, yeast in 3% warm sucrose water
Moldy/Musty*	Aromas associated with molds and/or freshly turned soil	2-ethyl-1-hexnol, potting soil
Methyl Ketone/ bleu*	Aroma associated with blue-veined cheeses	2-octanone, 40ppm
Oxidized*	Aroma associated with oxidized fat	2,4 decadienal, 20ppm
Waxy/Crayon*	Aromatics associated with medium chain fatty acids	Capric acid lauric acid or decanoic acid, 100mg/mL
Fecal*	Aroma associated with complex protein decomposition	Indol, skatole, 20ppm

Table 4 continued:

Bell pepper*	Aroma associated with freshly cut green vegetables	Methoxy pyrazines, 5ppb
Rosy/Floral	Aroma associated with flowers	2-phenethylamine, 20ppm
Scorched*	Aroma associated with extreme heat treament of milk proteins	Milk heated to 121°C for 25 min
Bitter	Fundamental taste sensation elicited by caffeine, quinine	Caffeine (0.08% in water)
Salty	Fundamental taste sensation elicited by salts	Sodium chloride (0.5% in water)
Sweet	Fundamental taste sensation elicited by sugars	Sucrose (5% in water)
Sour	Fundamental taste sensation elicited by acids	Citric acid (0.08% in water)
Umami	Chemical feeling factor elicited by certain peptides and nucleotides	MSG (1% in water)
Prickle/bite*	Chemical feeling factor of which the sensation of carbonation on the tongue is typical	Soda water

*Indicates term was not frequently encountered in Cheddar cheese
**Data analysis indicated term is redundant and is a combination of several terms.
Chemical references prepared in 95% ethanol
(Drake et al., 2001)

Figure 1: Skim Milk Powder Processing Flow Chart

Raw milk

Skimming/Separating

Standardizing

Heat treatment 1 (at least pasteurization)

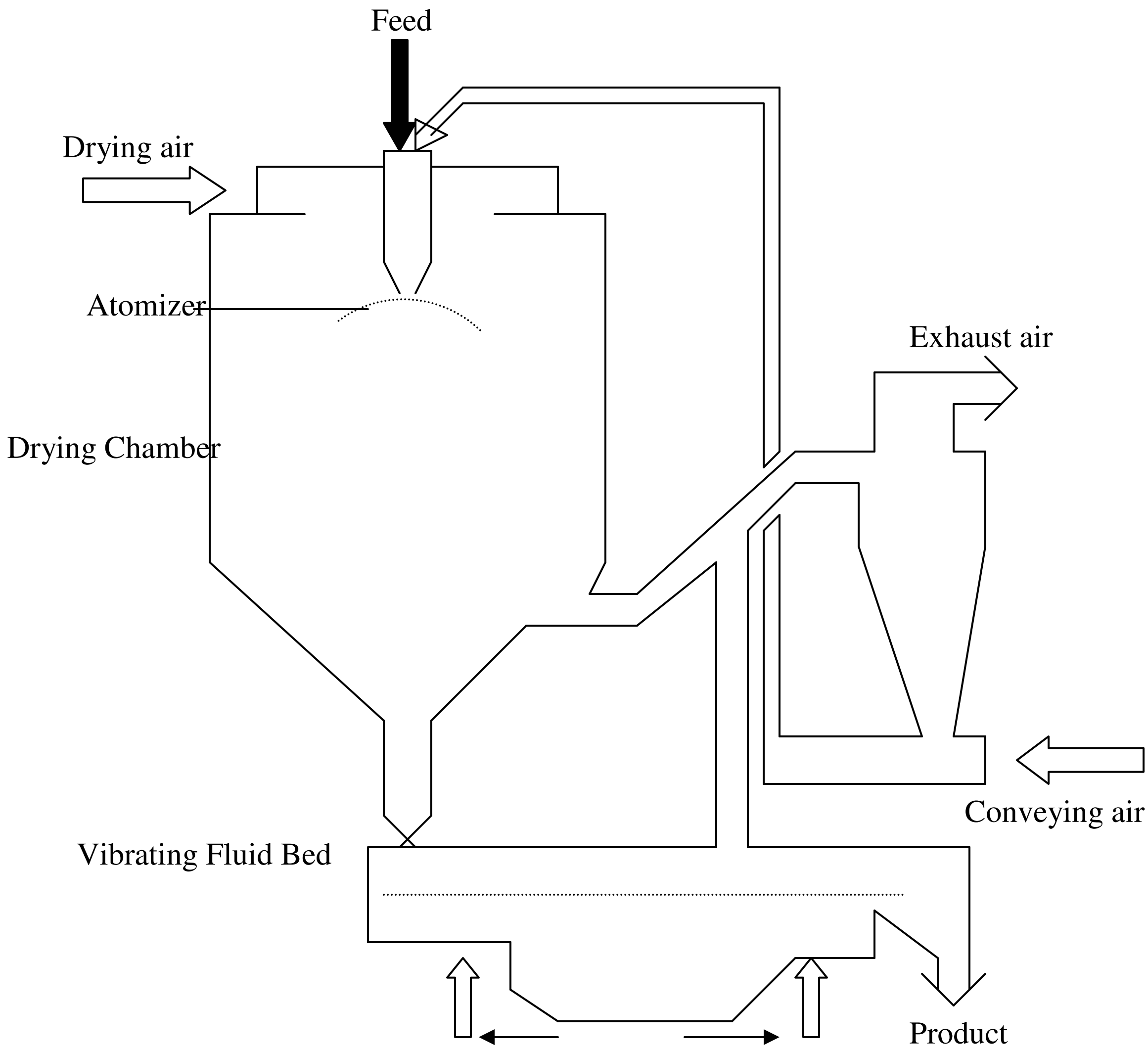
Concentration

Spray Drying and cool

Packaging

Distribution

Figure 2: Diagram of a two-phase Spray Dryer



Varnum and Sutherland, 1994

Figure 3: Example of Dairy Grading Scorecard for Dried Milk

Product: _______________

SAMPLE NO. Date: ____________

Flavor 10	Criticism Score								
No Criticism 10 Unsalable 0 Normal Range 1-5	Acid								
	Astringent								
	Bitter								
	Chalky								
	Cooked								
	Feed								
	Fermented								
	Flat								
	Foreign								
	Gluey								
	Metallic								
	Neutralizer								
	Oxidized/Tallowy								
	Rancid (Lipolysis)								
	Salty								
	Scorched								
	Stale								
	Storage								
	Unclean/Utensil								
	Weedy								
Physical Appearance 5	Score								
No Criticism 5 Unsalable 0 Normal Range 1-5	Dry Product:								
	Caked								
	Dark Particles								
	Lumpy								
	Unnatural Color								
	Reconstituted								
	Product:								
	Churned Particles								
	Dark Particles								
	Grainy								
	Undispersed Lumps								

Figure 3 continued:

Package 5	Score →								
No Criticism	Ruptured Vapor								
5	Barrier								
Unsalable 0	Soiled								
Normal Range	Unsealed								
1-5									
Laboratory Tests 5	Score →								
	Fat (%)								
	Moisture (%)								
	Titratable Acidity								
	(% Lactic Acid)								
No Criticism	Solubility Index (ML)								
5	Bacterial Estimate								
	(Per Gram)								
	Coliform (Per Gram)								
	Direct Microscopic								
	Clump Count (Per G)								
Unsalable 0	Scorched Particles (MG)								
	Dispersibility (Modified								
	Moats-Dabbah Method,%)								
	Phospahatase test								
	Micrograms Phenol/ML								
	Undenatured Whey Protein								
	Nitrogen (MG/G)								
	Oxygen Content (%)								
	Copper (PPM)								
	Iron (PPM)								
	Vitamin A (I.U.)								
	Vitamin D (I.U.)								
	Alkalinity of Ash								
	(ML/ 100 G)								
	Protein Content (%)								
	Mesh (Screen %)								
	Ash, Phosphorus Fixed (%)								
	Lead (PPM)								
	Yeast and Mold (Per 0.1 G)								

	Thermophiles (Per G)								
	Reducing Sugars (As								
	Lactose %)								
	Staphylococcus								
	(Coagulase Positive)								
	Salmonella (In 100 G)								

SIGNATURES: ________________ __________________ __________________

Bodyfelt et al., 1988

Table 5: Neutral/basic aroma-active compounds of goat cheese during GC/O

		Mean intensities[a]			RI[c]		
Nr.	Compound	Cheese 1	Cheese 2	Odor[b]	DB-Wax	DB-5	Method of Identification[d]
1	Diacetyl	3.00(4/4)	3.65(4/4)	Buttery	937	623	RI,odor,MS
2	Acetoin	1.50(3/4)	1.40(3/4)	Buttery		730	RI,odor
3	Methyl 2-methybutanoate	ND	2.25(4/4)	Butterscotch	1006	787	RI,odor,MS
4	Hexanal	1.50(2/4)	2.25(4/4)	Green grassy	1020	787	RI,odor
5	3-methyl thiophene	3.30(3/4)	4.30(4/4)	Sweet/ plastic	1026		RI,odor
6	Unknown	ND	1.90(3/4)	Ammonia	1099		
7	2-pentanol	ND	2.60(4/4)	Minty	1120	959	RI,odor,MS
8	Unknown	ND	2.00(3/4)	Geranium	1140	831	
9	1-hexen-3-one	1.70(3/4)	3.25(3/4)	Cooked/vegatable	1153		RI,odor
10	Octanal	1.25(3/4)	ND	Sweet/citrus		1023	RI,odor,MS
11	Heptanal	3.70(3/4)	3.55(4/4)	Fatty	1181	916	RI,odor,MS
12	1-hepten-3-one	ND	1.50(3/4)	Mushroom	1218	883	RI,odor
13	1-octen-3-one	4.00(4/4)	3.90(4/4)	Mushroom	1249	991	RI,odor
14	2-heptanol	1.50(4/4)	2.00(4/4)	Mushroom	1254	926	RI,odor,MS
15	2-acetyl-1-pyrroline	3.00(4/4)	2.05(4/4)	Popcorn	1285	939	RI,odor
16	(Z)-1,5-octadien-3-one	3.50(3/4)	3.60(4/4)	Geranium	1312	997	RI,odor
17	Unknown	2.00(2/4)	1.30(3/4)	Earthy/ Chocolate	1074		
18	Nonanal	2.50(4/4)	3.75(4/4)	Hay/sweet	1378	1107	RI,odor,MS
19	Unknown	ND	2.25(4/4)	Horsehair	1125		
20	Methional	5.25(4/4)	4.75(4/4)	Potato	1392	925	RI,odor,MS
21	2,5-dimethyl-3-ethylpyrazine	ND	2.50(4/4)	Potato	1441	1084	RI,odor
22	2-nonanone	2.75(3/4)	2.00(1/4)	Vitamin/ sour	1450	1096	RI,odor,MS
23	(Z,Z)-3,6-nonadienal	3.20(3/4)	ND	Fatty		1116	RI,odor
24	Unknown	4.60(4/4)	1.00(2/4)	Floral	1470	1150	
25	(E)-2-nonenal	3.30(4/4)	3.65(4/4)	Cucumber	1525	1170	RI,odor,MS
26	(E,Z)-2,6-nonadienal	4.25(2/4)	ND	Hay/fatty	1565	1186	RI,odor
27	Unknown	2.75(4/4)	2.20(3/4)	Vitamin/ minty	1199		
28	2-undecanone	2.50(2/4)	3.20(2/4)	Floral		1285	RI,odor,MS
29	(E)-2-decenal	2.65(3/4)	2.50(4/4)	Hay/fatty	1585	1267	RI,odor
30	(Z)-2-decenal	3.55(4/4)	2.85(4/4)	Fatty	1596	1246	RI,odor
31	Benzothiazole	4.00(2/4)	ND	Plastic/	1572		RI,odor,MS

Table 5 continued:

				Rubber			
32	Unknown	ND	3.35(4/4)	Coconut	1260		
33	(E,E)-2,4-nonadienal	1.50(3/4)	2.75(2/4)	Fatty	1609	1217	RI,odor
34	Unknown	5.50(4/4)	3.30(4/4)	Fatty/fried	1687	1354	
35	(E,E)-2,4-decadienal	2.50(4/4)	2.45(4/4)	Fried	1700	1304	RI,odor
36	Unknown	4.00(2/4)	3.35(3/4)	Fatty/waxy	1738		
37	2-acetyl-2-thiazoline	2.85(4/4)	2.00(1/4)	Popcorn	1763	1106	RI,odor
38	Dodecanal	2.20(3/4)	2.61(4/4)	Floral	1765	1387	RI,odor,MS
39	Unknown	3.35(2/4)	2.15(4/4)	Coconut/ hay	1434		
40	Decanal	2.15(3/4)	4.25(2/4)	Fatty/hay	1771	1267	RI,odor
41	Indole	3.50(2/4)	1.75(2/4)	Musty	1796	1254	RI,odor
42	γ -Butyrolactone	2.00(1/4)	2.35(4/4)	Coconut		1313	RI,odor,MS
43	γ –Dodecalactone	ND	ND	Coconut		1684	RI,odor,MS
44	Unknown	2.50(4/4)	3.35(4/4)	Goaty/waxy		1371	
45	3-Methyl indole (skatole)	3.45(4/4)	4.10(4/4)	Fecal/ mothball		1440	RI,odor
46	Geraniol	2.35(3/4)	1.75(2/4)	Grassy floral	1863	1278	RI,odor,MS
47	γ –Octalactone	3.90(4/4)	4.40(4/4)	Coconut		1547	RI,odor,MS
48	Vanillin	ND	2.50(4/4)	Vanilla	1899	1412	RI,odor
49	δ -decalactone	2.85(3/4)	1.85(4/4)	Peach	1972	1518	RI,odor,MS
50	γ -decalactone	2.00(1/4)	1.00(1/4)	Peach	2103	1508	RI,odor
51	Unknown	3.20(3/4)	3.70(4/4)	Peach	1725		
52	o – Aminoacetophenone	2.50(4/4)	3.95(4/4)	Grape	2281	1346	RI,odor
53	δ -Dodecalactone	2.95(4/4)	5.00(1/4)	Coconut		1733	RI,odor,MS

Carunchia Whetstine et al., 2003

Chaper 3

Influence of Flavor Variability in Skim Milk Powder on Consumer Acceptability of Ingredient Applications

Influence of Flavor Variability in Skim Milk Powder on Consumer Acceptability of Ingredient Applications

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Short title: Flavor variability and acceptance

Journal section: Sensory and nutritive qualities of food

Abstract

Flavor variability in SMP has been documented by descriptive sensory analysis and instrumental analysis. However, research has not addressed how or if SMP flavor variability impacts consumer acceptance of ingredient applications. Twenty reconstituted low heat SMP were screened using a previously established defined sensory language. Two SMP free of off-flavors (flavor similar to fresh fluid skim milk) and two SMP exhibiting off-flavors (exhibiting flavors not found in fluid skim milk) were selected for further study. Volatile compounds were determined using solid phase microextraction (SPME) with gas chromatography-mass spectrometry (GC-MS). Each SMP was subsequently directly reconstituted or incorporated into standard formulas for vanilla ice cream, strawberry yogurt, hot cocoa mix, and white chocolate bars. Consumer acceptance testing was conducted. Descriptive panelists documented sweet aromatic and cooked flavors in the fresh-flavored SMP and these flavors as well as fatty/fryer oil and animal-like flavors in the off-flavored SMP. Concurrently, instrumental volatile analysis revealed higher relative abundances of lipid oxidation compounds in the off-flavored SMP. Consumer acceptance scores were lower for the off-flavored SMP than for the fresh-flavored SMP when evaluated directly reconstituted ($p<0.05$) and for ingredient applications made using off-flavored SMP ($p<0.05$). Off-flavors in SMP documented by descriptive analysis and trained panelists can carry-through into ingredient applications with SMP and negatively affect consumer acceptability.

Introduction

Low heat skim milk powder (SMP) is a commonly used ingredient in many foods. Fresh SMP should ideally exhibit a mild and bland flavor reminiscent of fluid skim milk (Bodyfelt and others 1988). The proposed shelf life of SMP varies. A shelf life of anywhere between 6-36 months for non-instantized unfortified SMP under optimal storage conditions has been proposed by various sources (Varnum and Sutherland 1994; USDEC 2001; ADPI 2002). Because SMP is used so widely, flavor, flavor stability, and off-flavor carry-through in ingredient applications is crucial information to determine usage and storage regimes (Ulberth and Roubicek 1995). However, since SMP is low in fat, less attention is given to its flavor variability and how this may impact consumer perception of quality when directly rehydrated or when used in ingredient applications.

Dairy grading/judging standards include a flavor defect descriptor for ice cream and yogurt called “old ingredient” flavor. This flavor defect refers to ice cream and yogurt made with mishandled dairy or other ingredients which may have developed off-flavors which carry through into finished product (Bodyfelt and others 1988). Clearly, traditional dairy products judging suggests that ingredient quality can impact sensory quality of finished products. Flavor variability in SMP has been documented by trained panelists. Driscoll and others (1985) evaluated sensory quality of rehydrated SMP under various storage conditions. They reported an overall decrease in milk powder quality during storage as a direct reflection of the development of an oxidized/stale flavor. However, minimal storage time of SMP does not guarantee a product with a flavor profile reminiscent of fluid skim milk (free of off-flavors). Drake and others (2003) developed a defined sensory language for SMP and documented wide flavor variability (including non-fluid milk or off-flavors) among fresh (< 3 mo) reconstituted low heat SMP. Flavor variability in SMP with minimal storage time emphasizes the need to demonstrate how or if flavor variability documented by trained panelists may impact consumer perception. Flavor of reconstituted SMP and consumer acceptance, and the potential for off-flavors in SMP to carry-through in ingredient applications and impact consumer acceptability have not been quantitatively addressed. In this study we examined the effect of flavor variability in SMP on consumer acceptability of four different products that commonly contain SMP -- vanilla ice cream, strawberry yogurt, white chocolate bars, and hot cocoa mix- as well as directly reconstituted SMP.

Materials and Methods

SMP Selection

Twenty domestic commercially-packaged non-instantized low heat SMP (25 kg 2-ply bags) of varying ages (3 mo-5 years) were rehydrated on a 10% (w/w) basis in deodorized water and screened by 3 individuals, each with more than 500 h of experience in the descriptive sensory analysis of dairy products, including SMP. Four SMP were selected. SMP 1 and 2 were 4 months old. SMP 3 was 18 months, and SMP 4 was 4 years old. The four SMP were selected to represent samples free of off-flavors (flavor similar to fresh fluid skim milk) and samples with off-flavors (exhibiting flavors not found in fluid skim milk) (Drake and others 2003).

Descriptive sensory analysis was subsequently conducted on the selected reconstituted SMP using the descriptive language and methods described by Drake and others (2003) (Table 1). SMP (30 ml) were dispensed into 2 oz soufflé cups with lids and 3 digit codes. Sensory analysis was conducted at 12°C. Seven trained panelists (5 female, 2 male) evaluated each SMP in duplicate. Each panelist had more than 50 h of training on the descriptive sensory analysis of dried dairy ingredients with a previously identified descriptive language and the Spectrum™ descriptive analysis method (Drake and others 2003; Meilgaard and others 1999).

Proximate Analysis

Vacuum oven moisture content and whey protein nitrogen (WPN) analysis were conducted in duplicate on the four selected SMP using standard methods (Bradley and others 1993).

Instrumental Volatile Analysis

Solid phase microextraction (SPME) was used to analyze volatile compounds in SMP. Ten grams of SMP was transferred into pre-washed Quorpak clear standard wide mouth jars (250mL, Fisher; Pittsburgh, PA). Deodorized water (100 mL) was added to the bottle and the bottle was gently shaken to dissolve the SMP followed by the addition of 2.5 µL of internal standard (0.185 ppm 2-methyl-3-heptanone in methanol; Aldrich Chem. Co.; Milwaukee, WI). The rehydrated SMP (20 g) was transferred into pre-cleaned 40 mL amber vials with screw caps and Teflon silica septa (Supelco; Bellefonte, PA). A magnetic octagonal stirring bar (8mm OD x 13mm L, Fisher; Pittsburgh, PA) and commercial sodium chloride (1 g) were added. Each sample was allowed to equilibrate in the vial at 47°C for 30 minutes using a heating/stirring module (Reacti-Therm™, Pierce Biotechnology, Inc.; Rockford, IL).

Volatile odor compounds from rehydrated SMP were extracted by exposing a 2 cm-50/30 µm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) StableFlex SPME fiber (Supelco; Bellefonte, PA) to the headspace of each sample at 47°C for 30 minutes. Following extraction of the volatile odor compounds, the SPME fiber was inserted into a splitless injection port (250°C) of a 5890 Series II Plus gas chromatograph (Hewlett Packard, Palo Alto, CA) with a fused nonpolar silica capillary column (DB-5, 30 m, 0.25µm i.d., J&W Scientific, Albany, NY). Helium gas was used as a carrier at a constant flow of 1mL/min. The initial temperature of the oven was 40°C with a hold time of 5 min., increasing 8°C per minute to a final temperature of 250°C, which was held for 5 min. The detector was a 5972 Series Mass Selective Dectector (Hewlett Packard, Palo Alto, CA). MSD conditions were as follows: capillary direct interface temperature 280°C, ionization energy 70eV, mass range 33-330 a.m.u., EM voltage (Atune + 200V), scan rate, 5 scans/sec. Each sample was analyzed in quadruplicate. The volatile compounds were identified using the NIST02 mass spectra library, and retention indices of authentic standard compounds analyzed under identical conditions.

Preparation of Ingredient Applications

<u>Direct reconstitution</u>. Each SMP was reconstituted 10% w/v in deodorized water. Milks were stored at 5°C overnight to fully rehydrate and then dispensed (30 mL) into 118 mL sample cups with lids (Sweetheart Cup Co., Owings Mills, MD) and three digit random codes. Pasteurized (HTST) fluid skim milk was obtained from the North Carolina State University creamery. Products were served to consumers at 5˚C.

Vanilla ice cream. Vanilla ice cream mix was prepared in 2 L batches using a standard formula (Table 2). Skim milk, cream (40% fat), SMP, sugar, and stabilizer consisting of mono and diglycerides, guar gum, polysorbate 80, locust bean gum, carageenan, calcium sulfate, and silicon dioxide (AO565-DR1-1, Danisco, New Century, KS) were mixed together using a hand held mixer and pasteurized in a double boiler at 68˚C for 30 minutes. The mixture was immediately cooled in an ice bath to approximately 5˚C and held at this temperature overnight. Single fold pure bourbon vanilla extract (VA01, Virginia Dare, Brooklyn, NY) was added and the mixture was blended using a hand held mixer directly prior to freezing. The ice cream mix was placed into a 4 quart ice cream freezer (Rival, El Paso, TX), and rock salt and ice were used in a 1:10 ratio to rapidly freeze the mixture (ca. 70 % overrun). Ice cream was packed into 11.4-L paperboard containers (Huhtamaki, Fulton , NY) with lids and hardened at -20C for 24 h. Ice creams were tempered at -15 º C for 24 hours and samples (20 g) were dispensed into 118-ml plastic cups fitted with lids (Sweetheart Cup Co., Owings Mills, MD) and held at -15 ± 2 º C until served.

Strawberry yogurt. Strawberry yogurt (4L) was made on a 20% total solids basis with non-fat fluid milk (10% w/w), skim milk powder (5% w/w), sucrose (5% w/w), and stabilizer (1.6% w/w; Danisco Grindsted® Yogurt 5996, Copenhagen, Denmark) using the procedure described by Drake and others (2000). The ingredients were mixed in a stainless steel container with a hand held mixer. The mixture was pasteurized in a double boiler at 68˚C for 30 minutes, and immediately cooled to 43˚C in an ice bath. The mixture was inoculated with a mixed strain culture (YoFast 17, Chr. Hansen, Milwaukee, WI) and subsequently incubated at 43˚C to a pH of 4.2-4.3 (ca 6 h). Yogurts were stored at 5˚C overnight. A strawberry fruit base (Strawberry Base #7924, Fruitcrown, Farmingdale, NY) was added at 15% (w/w) and strawberry flavoring (Natural Strawberry Flavor WONF #060-01059, Degussa, Cincinnati, OH) was added at 0.35% (w/w). The finished yogurt (10 g) was scooped into 118 mL sample cups with lids (Sweetheart Cup Co., Owings Mills, MD) and stored at 5˚C until served to consumers.

White chocolate bars. White chocolate bars (Table 3) were prepared according to the following procedure. Sucrose, SMP, cocoa butter, anhydrous milkfat and vanillin were combined in a mixing bowl and heated to 40˚C. The particle size of the slurry was reduced to a d_{90} of 35-45 µm using a 3-roll mill. Chocolate was conched at 55˚C for 3 hours. Lecithin and polyglycerol polyricinoleate (PGPR) were added directly to the chocolate 30 minutes prior to completion of conching. Chocolate was tempered and molded into 40 g bars. Finished chocolate bars were stored in sealed foil pouches at 18˚C for 12 weeks prior to sensory evaluation. Chocolate (approx. 10g) was dispensed into 118 mL sample cups with lids (Sweetheart Cup Co., Owings Mills, MD) for consumer testing.

Hot cocoa. Hot cocoa mix was made using the formula shown in Table 4. All dry ingredients were mixed and sifted twice to reduce powder lumps. Two hundred fifty grams of the powder was mixed with 1L of deodorized water heated to boiling and blended with a hand held mixer. Vanilla extract was added in the amount of 1.875mL/1L of mixed cocoa and the mixture was blended again. Cocoas were served to consumers at 70˚C in 8 oz. styrofoam cups.

Consumer Acceptance Testing

Consumer testing was conducted in compliance with NCSU Institutional Review Board guidelines for human subjects. Consumer acceptance testing was performed on each type of product on different days. Consumers (n = 100 to 120) were recruited from university staff and students by fliers and email. Demographic information (age, gender, product usage) was collected from each consumer prior to tasting. Consumers were given a paper ballot, deionized water and unsalted crackers for palate cleansing. Samples were labeled with 3-digit codes and presented in randomized balanced order. The ballot featured questions regarding product specific flavor attribute intensities, product specific flavor attribute likings, texture liking, sweet taste intensity and liking, freshness intensity, and overall liking, and these were evaluated using a 9-point hedonic scale anchored on the left with "dislike extremely" and on the right with "like extremely".

Statistical Analysis

Analysis of variance with means separation was conducted to determine differences between treatments. Statistical analysis was performed using SAS, version 8.2 (Cary, NC).

Results and Discussion

Descriptive sensory analysis and proximate analysis

Descriptive sensory panelists documented fresh fluid-milk types of flavors in SMP 1 and 2 and storage or off-flavors in SMP 3 and 4 (Table 5) (Drake and others 2003). SMP 3 exhibited a distinct fatty/fryer oil flavor while SMP 4 was characterized by an animal-like flavor. Concurrent with the development of off-flavors, SMP3 and SMP4 also displayed lower intensities of fresh fluid milk flavors such as sweet aromatic and cooked flavors. These profiles are also consistent with previous studies (Karagul-Yuceer and others 2002; Drake and others 2003) and may indicate that loss of "fresh flavors" is just as important in off-flavor development as the evolution of the off-flavors themselves or that low intensities of these fresh flavors alone are indicative of off-flavors. Additional studies with a wider sampling of SMP would be necessary to confirm this hypothesis. Moisture content of SMP 1 and 2 was lower than SMP 3 and 4 ($p<0.05$) (3.69 ± 0.02, 4.10 ± 0.04 compared to 4.64 ± 0.02, 4.66 ± 0.02 %, respectively). Higher moisture content of SMP has been associated with lower quality (Driscoll and others 1985; Karagul-Yuceer and others 2002). WPN content was similar between the four SMP (7.3 + 0.4 %), values consistent with low heat SMP.

Volatile analysis

The mean relative abundance of selected volatile compounds found in each of the SMP is given in Table 6. Some consistent differences were observed among the fresh and off-flavored SMP. Both dimethyl sulfide and dimethyl disulfide were found in both off-flavored SMP, but not in SMP 1 or 2 suggesting that these compounds contribute to off-flavors. Maltol was found in SMP 1 and 2, but not in SMP 3 or 4. Maltol, a sugar caramelization product, is characterized by a sweet/burnt sugary/cotton candy type of aroma and may have contributed to the higher sweet aromatic intensities observed in SMP 1 and 2. Karagul-Yuceer and others (2004) determined that maltol was one of the key contributing volatile compounds to the flavor of fresh SMP. Sweet aromatic is a key descriptor for fresh flavored SMP. Loss of this flavor is consistent with loss of fresh flavors in SMP and the development of off-flavors (Drake and others 2003). SMP 3, which was characterized by a fatty off-flavor by sensory analysis, had the highest concentrations and types of aldehydes. Aldedydes are lipid oxidation products and can be characterized by fatty/oily types of flavors and have been associated with flavor degradation in SMP (Karagul-Yuceer and others 2002). A definitive volatile compound(s) has not been clearly identified for the descriptive flavor “animal” in SMP. Karagul-Yuceer and others (2003) investigated the volatile components responsible for the intense “animal” flavor in rennet casein. The defined sensory language of Drake and others (2003) was used such that the attribute should be consistent between rennet casein and SMP. Hexanoic acid, indole, guaiacol, and p-cresol were determined by model system testing to be sources of this flavor in rennet casein. These compounds were not detected in SMP 4 in the current study. However, those compounds may not have been recovered using the particular volatile compound extraction technique used in this study (SPME) and does not preclude that they might contribute to this off-flavor in SMP.

Consumer Acceptance

Consumer gender and age demographics were similar across all five days of testing. Eighty-nine percent of consumers polled were between the ages of 18 and 55 years old. The other eleven percent were between the ages of 56 and 65 years of age.

Sixty percent of consumers were female, forty percent were male. For the days that ingredient applications were tested, all participants were at least occasional consumers of the particular product. For directly reconstituted SMP, all participants consumed fluid milk at least once per week. Sixty percent regularly consumed fluid skim milk. None of the consumers regularly consumed reconstituted SMP.

In the U.S., SMP is generally used as an ingredient, and not for direct consumption. Our experimental objective in the test with directly reconstituted SMP was a first step to determine if differences perceived by trained panelists (specifically off-flavors) resulted in differences in acceptability by consumers. Fluid skim milk was included in this test as a benchmark. For directly reconstituted product, none of the SMP scored as high as fluid skim milk (Table 7). Overall acceptance, flavor liking, and perceived freshness intensity for fluid skim milk were scored 1 point or more higher on the hedonic scale than the highest scored SMP; indicating that consumers can clearly detect differences between fluid milk and dried reconstituted skim milk. SMP 1 scored the highest across all attributes for the four SMP, including perceived freshness intensity, followed by SMP 2. Both SMP 3 and 4 were scored lower than SMP 1 and 2 for overall acceptance and freshness intensity. These results indicate that while consumers can detect differences between fluid skim milk and SMP, they can also detect differences between SMP with and without off-flavors and that SMP characterized by off-flavors by trained panelists are also perceived as off-flavors (lower acceptance, lower freshness) by consumers.

In general, differences in overall acceptability among the ingredient applications were not as markedly different as the differences between fluid skim milk and the directly reconstituted SMP. These results are logical as flavor differences should be more easily detected in reconstituted SMP than in ingredient applications made from SMP. Ingredient applications made with SMP 3 and 4 scored lower overall acceptance scores compared to products made using SMP 1 and 2. For vanilla ice creams, product made using SMP 4 scored the lowest scores across all attributes except texture and sweetness liking where no differences were observed (Table 8). Scores for ice creams made using SMP3 were also lower in perceived flavor intensity compared to SMP 1 or 2. Other liking and intensity scores for ice creams made from SMP3 were lower than those made with SMP 1 and 2, but not statistically significant. For strawberry yogurts and white chocolate, trends were similar (Tables 9, 10) in that products made using SMP 4 scored lower for liking and intensity attributes compared to those made with SMP 1. Yogurts made using SMP 3 did not receive scores that were different from products made using SMP 1 or 2. In contrast, hot chocolate prepared with SMP 3 scored the lowest scores in all attributes except appearance (Table 11) while scores for cocoas made with SMP 4 were not different from cocoas made with SMP 1 or 2.

Ingredient applications tested covered a wide range of flavors, from delicate (vanilla ice cream) to stronger flavors and flavor intensities (strawberry yogurt, hot cocoa). Typical amounts of SMP used in each ingredient application also varied. Regardless, results indicated that flavor quality of the SMP did impact the flavor quality and acceptance of the finished product. For all of the ingredient applications except hot cocoa, SMP 3 characterized with a low intensity of fatty flavor by descriptive analysis, did not impact consumer acceptance in ingredient applications. By direct reconstitution, this off-flavor was also not perceived as negatively by consumers as the animal flavor in

SMP 4. However, in hot cocoa, product made from SMP 4 did not score differently from product made with SMP 1 or 2 while product made using SMP 3 scored the lowest acceptance scores. This difference might be because the animal flavor was effectively masked by chocolate flavor while the fatty flavor in SMP 3 was not masked in this ingredient application. Different flavoring systems and products are known to exhibit different masking or flavor carry-though effects. These results suggest that different SMP flavor specifications could be developed for different products. While mean consumer scores were different for products made with SMP 3 or 4 compared to those made with SMP 1 or 2, acceptance scores from some consumers were not different for products made with the four different SMP indicating that either these consumers could not detect differences or that perceived differences had no effect on acceptability. Additional experiments would be needed to determine the descriptive nature of the perceived consumer differences in these ingredient applications and how or if these differences in acceptability would impact purchase intent.

Conclusions

Taken as a whole, consumer results suggest three things. Firstly, off-flavors in SMP noted by trained panels are also detected by consumers in reconstituted product and off-flavors in SMP do carry-through into ingredient applications where they can also negatively impact consumer liking and acceptance scores. Secondly, the degree to which an off-flavor can impact an ingredient application depends on the type/intensity of the off-flavor and the nature of the ingredient application. Finally, certain off-flavors may be more easily masked in some products.

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Table 1: Descriptive sensory language for rehydrated skim milk powders.

Descriptor	Reference	Preparation
Cooked/sulfurous	-Heated milk	-Heat pateurized skim milk to 85ºC for 45 min
Sweet aromatic/ cake mix	-Pillsbury-White cake mix -vanillin	-dilute 5mg of vanillin in skim milk
Animal/gelatin-like/wet dog	-Knox-unflavored gelatin	-dissolve one bag of gelatin (28g) in two cups of distilled water
Fatty/ frier oil	-(E,E)-2,4-decadienal	-2 ppb in skim milk
Sweet taste	-sucrose	-5% sucrose solution
Salty	-NaCl	-2% NaCl solution
Sour	-citric acid	-1% citric acid solution
Bitter	-caffeine	-0.5% caffeine solution
Astringent	-tea	-soak 6 tea bags in water for 10 min

Adapted from Drake and others 2003

Table 2. Vanilla ice cream formula

Ingredient (source)	% (w/w)
Skim milk (NCSU Creamery)	50.00
Cream (40 % fat, local store)	30.00
SMP	4.00
Sucrose (local store)	15.00
Stabilizer (Danisco, New Century, KS)	0.25
Vanilla (Virginia Dare, Brooklyn, NY)	0.75
Total	100.00

Adapted from Arbuckle 1977

Table 3. White chocolate formula

Ingredient	% (w/w)
Sucrose (local store)	43.75
SMP	22.30
Cocoa butter (ADM; Decatur, IL)	27.60
Anhydrous milkfat (Fonterra; Lemoyne, PA)	5.80
Vanillin (Rhodia USA; Cranbury, NJ)	0.05
Lecithin (Degussa Texturant Systems; Atlanta, GA)	0.40
PGPR (Danisco USA; New Century, KS)	0.10
Total	100.00

Table 4. Hot cocoa formula

Ingredient	% (w/w)
Sugar (local store)	52.25
Cocoa (Hershey's® Cocoa Powder, local store)	6.5
Non-dairy Creamer (Coffeemate® Original Powder Creamer, local store)	17
SMP	21
Salt (local store)	0.4
Vanillin (Virginia Dare, Brooklyn, NY)	0.15
Guar Gum (Insta*Thick Guar, Primera Foods, Cameron, WI)	0.6
Cellulose Gum (Insta*Thick CMC 7LXF, Primera Foods, Cameron, WI)	2.1
Total	100.00

Adapted from calcium fortified cocoa formulation
http://www.doitwithdairy.com/formulations/beverages/hot_cocoa.html

Table 5: Descriptive sensory profiles of selected SMP

Attribute	SMP1	SMP2	SMP3	SMP4
Cooked	3.3a	3.1a	2.0b	3.5a
Sweet aromatic	3.0a	3.5a	1.5b	1.5b
Sweet taste	2.5ab	3.0a	2.3b	1.5c
Astringency	1.5ab	1.0b	2.0a	1.5ab
Animal	0.0b	0.0b	0.0b	2.5a
Fatty/frier oil	0.0b	0.0b	2.0a	0.0b

Means in a row followed by different letters are different ($p<0.05$)
Intensities scored on a 15-point Universal Spectrum™ intensity scale where 0=none and 15=very.

For attribute descriptions, see Table 1.

Table 6: Relative abundance of volatile compounds in SMP (ppm)

Compound	SMP1	SMP2	SMP3	SMP4
Dimethyl sulfide	ND	ND	0.096a	0.027a
Dimethyl disulfide	ND	ND	0.029a	0.034a
Hexanal	0.04a	ND	0.58a	0.06a
Benzeneacetaldehyde	ND	ND	ND	0.025
Heptanal	ND	ND	0.26	ND
Octanal	ND	ND	0.20	ND
Nonanal	0.045	ND	0.24	ND
(E)-2-hexenal	ND	8.33a	ND	3.67a
Maltol	0.54b	37a	ND	ND
Acetic acid	0.044a	ND	ND	0.044a
2-Heptanone	0.11	ND	ND	ND
Octanoic acid	0.073a	ND	0.061a	0.13a
Pentanal	ND	0.46	ND	ND
2-Nonanone	ND	0.050	ND	ND
Nonanoic acid	ND	ND	ND	0.047

Means are the results for four replications

Means in a row followed by different letter are different ($p<0.05$)

ND – not detected

Table 7: Consumer acceptability of reconstituted SMP and fluid skim milk (n= 115)

Attribute	SMP1	SMP2	SMP3	SMP4	Fluid skim
Overall acceptance	5.42b	4.51c	4.10d	3.38e	6.50a
Flavor liking	5.18b	4.22c	3.84c	3.10d	6.50a
Texture liking	5.97b	4.76d	5.15c	4.77cd	6.59a
Freshness intensity	5.75b	4.92c	4.38d	3.99d	6.75a

Means in a row followed by different letters are different ($p<0.05$)
Consumer acceptability or perceived intensity scored on a 9 point hedonic scale where 1 = dislike extremely or low intensity and 9 – like extremely or high intensity.

Table 8: Consumer acceptability of vanilla ice creams made with different SMP

Attribute	SMP1	SMP2	SMP3	SMP4
Appearance	6.90a	6.61ab	6.56bc	6.30c
Flavor intensity	6.18ab	6.19a	5.78b	5.37c
Sweetness intensity	6.29a	6.13a	6.08a	5.36b
Sweetness liking	6.11a	6.19a	6.09a	5.81a
Flavor liking	5.97ab	6.10a	5.98ab	5.60b
Texture liking	5.74a	5.87a	5.79a	5.49a
Overall acceptance	6.18ab	6.24a	5.86bc	5.65c

Means in a row followed by different letters are different ($p<0.05$)

Consumer acceptability or perceived intensity scored on a 9 point hedonic scale where 1 = dislike extremely or low intensity and 9 – like extremely or high intensity.

Table 9: Consumer acceptability of strawberry yogurts made with different SMP

Attribute	SMP1	SMP2	SMP3	SMP4
Appearance	7.16a	6.80bc	7.00ab	6.66c
Strawberry flavor intensity	5.88a	5.71ab	5.96a	5.39b
Strawberry flavor liking	6.03a	5.86a	6.18a	5.42b
Sweetness intensity	5.70a	5.88a	5.70a	5.17b
Sweetness liking	6.04a	5.92a	6.03a	5.46b
Overall flavor liking	5.97a	6.08a	6.17a	5.20b
Texture liking	6.31a	6.31a	6.14a	5.06b
Overall acceptance	6.16a	6.12a	6.30a	5.25b

Means in a row followed by different letters are different ($p<0.05$)

Consumer acceptability or perceived intensity scored on a 9 point hedonic scale where 1 = dislike extremely or low intensity and 9 – like extremely or high intensity.

Table 10: Consumer acceptability of hot cocoa made with different SMP

Attribute	SMP1	SMP2	SMP3	SMP4
Appearance	6.78a	6.78a	6.87a	6.72a
Chocolate flavor intensity	6.06a	6.05a	5.55b	6.02a
Chocolate flavor liking	5.94a	6.21a	5.17b	6.17a
Overall texture liking	6.29a	6.22ab	5.96b	6.57a
Overall flavor liking	5.81a	6.09a	5.13b	6.04a
Overall acceptance	5.82a	6.14a	5.13b	6.06a

Means in a row followed by different letters are different ($p<0.05$)

Consumer acceptability or perceived intensity scored on a 9 point hedonic scale where 1 = dislike extremely or low intensity and 9 – like extremely or high intensity.

Table 11: Consumer acceptability of white chocolate made with different SMP

Attribute	SMP1	SMP2	SMP3	SMP4
Appearance	6.92a	6.88a	6.86a	6.89a
Chocolate flavor intensity	5.14a	5.16a	4.98ab	4.75b
Chocolate flavor liking	5.65a	5.73a	5.47ab	5.14b
Milky intensity	6.03a	5.97ab	5.67b	5.65b
Milky flavor liking	6.02a	5.99a	5.87ab	5.51b
Overall texture liking	6.60a	6.29ab	6.35ab	6.16b
Overall flavor liking	6.27a	6.01a	5.98a	6.06a
Overall acceptability	6.51a	6.19ab	5.914b	6.10b

Means in a row followed by different letters are different ($p<0.05$)

Consumer acceptability or perceived intensity scored on a 9 point hedonic scale where 1 = dislike extremely or low intensity and 9 – like extremely or high intensity.

Chapter 4

Flavor Formation in Skim Milk Powder throughout Production

Flavor Formation in Skim Milk Powder throughout Production

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Abstract

Skim milk powder (SMP) is commonly used as a food ingredient. Previous studies have characterized the sensory profiles and chemical compounds responsible for the flavor in fresh and stored SMP. In this study, we examined the flavor formation of medium heat SMP throughout the production process, with emphasis on the steps where heat was applied to the product. Samples were collected throughout the production process (raw milk to finished powder) across a 40 hour production run. Descriptive sensory analysis and instrumental volatile analysis were conducted. The sensory attributes of the milk changed significantly throughout the SMP production process ($p<0.05$). Cooked and sweet aromatic flavors increased throughout the process. Important flavors identified by gas chromotography-olfactometry were heat generated compounds, including methional, 2-acetyl-1-pyrroline, and homofuraneol. More compounds were identified in samples from the end of the run versus beginning and middle run samples. There was not a collection time effect for any processed (heat treated) ($p>0.05$), suggesting that equipment burn-on or soiling throughout 40 h of production does not impact sensory properties of the finished product.

Introduction

Skim milk powder (SMP) can be rehydrated and consumed directly, but in the US it is more commonly used as an ingredient in many other foods to improve body, texture, and flavor (Karagul-Yuceer and others, 2002). Large quantities of SMP are produced in the United States, many of which are directed towards specific end product applications due to specific heat treatment throughout the drying process. The quality of the raw milk is certainly a determining factor in SMP flavor, but the processing steps that produce SMP can also affect the flavor (Karagul-Yuceer and others, 2001, 2002).

Most often, spray drying is the process used to dry milk products. Three different types of SMP can be produced; low heat, medium heat, or high heat (Varnum and Sutherland, 1994). The heat treatment of the fluid milk before it enters the spray-drier is the criteria used to determine the SMP category. The heat treatments are 72°C/ 15 s, 82°C/ 3 min, and 82°C/ 30 min for low, medium, and high heat, respectively (Rehman and others, 2003). The heat treatment affects the functionality of the SMP by denaturing whey proteins. More extensive heat treatment yields higher whey protein denaturation (Walstra and others, 1999a). The whey protein nitrogen (WPN) index is used to classify milk powder according to the intensity of the heat treatment applied during manufacture. The amount of denaturable whey protein left in the reconstituted product is reported as the quantity of denatured whey protein per gram of skim milk powder. The extent of denaturation is important to the use of the end product due to flavor and functionality differences (Walstra and others, 1999a).

Although low heat SMP is the most commonly produced type of SMP, medium heat SMP production is increasing. Medium heat SMP used in products such as milk based-drinks and ice cream, and it is also the primary SMP utilized on the global market (Varnum and Sutherland, 1994). The WPN of medium heat SMP is between 1.5 and 6 mg N per g SMP (Walstra and others 1999a). High and medium heat SMP characteristically have a noticeably higher cooked flavor compared to low heat SMP (Karagul-Yuceer and others, 2001). Inappropriate or inaccurate heat treatments or holding times at any stage of the drying process can cause undesirable flavors and/or inconsistencies in SMP flavor of any heat treatment type.

Raw milk is transported and stored in bulk at refrigeration temperatures, sometimes as long as 4 days, but more often in the US it is processed into SMP within 12 hours. The SMP production process begins with skimming, standardizing, and heat treating/pasteurization of fluid milk (Varnum and Sutherland, 1994). Skimming and standardizing mainly concern fat content but may also apply to dry matter and protein content of the product. An addition of cream, skim milk, or water may be necessary to standardize one or more of these components. At this point the milk is held at a low temperature in a bulk tank before further processing (Walstra and other, 1999b). The standardized fluid skim milk is then pasteurized using high temperature-short time pasteurization (HTST; 71.1°C for 15 seconds). The pasteurized fluid milk is transferred into a large tank (hot well) where it is heated to a high temperature (85°C) for an extended time (20 min). The pre-heating of the fluid milk before evaporation dictates low, medium, or high heat powder. After these initial steps to the fluid milk, it is concentrated by evaporation to 45%-55% total solids. Evaporation is performed under reduced pressure to allow boiling at lower temperatures (Walstra and others, 1999c) and milk solids content is increased gradually by passage through a series of evaporators (1-

6). The temperature is decreased in each stage of the evaporator from 65.5°C in the first stage to 54.4°C in the last stage. The concentrated milk passes into the spray dryer (see Figure 1) where it is atomized into small droplets that are dried in the hot air of the chamber. The concentrate is atomized by either spinning disks or pressure nozzles to small droplets that will dry quickly. The atomized liquid and hot air are mixed intensely within the drying chamber to produce a dry powder. After drying, the powder and air are separated in one or two cyclones or by filters (Walstra and others, 1999c). The total time from raw milk to SMP is less than 1 hour. Large commercial facilities may process milk continuously for 40 hours prior to a brief shut down for cleaning and sanitation. Flavor (desirable as well as undesirable) formation can occur at any stage in SMP production.

One crucial source of flavor in heated products is the Maillard reaction. The Maillard reaction is the chemical reaction of reducing sugars, usually D-glucose, and a free amino acid or amino acid group that is part of a protein chain. Maillard reaction products occur when aldoses or ketoses are heated with amino groups, and these reactions occur faster in liquid systems than in dried foods (Baltes, 1982). A second source of flavor can be caramelization reactions. Because milk products contain large amounts of lactose and casein, they are highly susceptible to Maillard reactions and caramelization reactions (Renner, 1988). Karagul-Yuceer and others (2001, 2004) identified heat generated compounds including both caramelization and Maillard reaction products as key components of fresh SMP.

Descriptive sensory analysis as well as instrumental analysis have been applied to identify and characterize the compounds responsible for the flavor and aroma in fluid milk and dried dairy ingredients. Drake and others (2003) identified a descriptive sensory language to document the flavor of rehydrated dried dairy ingredients, including SMP. Karagul-Yuceer and others (2001, 2002) applied descriptive sensory and instrumental analysis to characteriaze odor-active compounds in fresh and stored SMP using a combination of sensory and instrumental analysis. The specific volatile compounds contributing to the flavor of fresh low heat SMP were later confirmed using model system analysis (Karagul Yuceer and others, 2004). Caudle and others (2005) demonstrated that off-flavors in low heat SMP carried through into a variety of ingredient applications, where they negatively impacted consumer acceptance.

Clearly, sensory quality of SMP is crucial. The use of sensory and instrumental analysis techniques throughout SMP production to document where flavor occurs in the drying process will enhance our understanding of SMP flavor development, a crucial property as it becomes more widely used in product applications, nationally and globally. In this study, we examined the flavor formation of medium heat SMP throughout the production process, with emphasis on the steps where heat is applied to the product.

Materials and Methods

Samples

Raw milk (12 % solids, 9% solids not fat (SNF)), balance tank (9% SNF), HTST pasteurized (9% SNF), evaporated milk (50% SNF), and SMP (94% SNF) samples, were collected from a large west coast production facility on three different occasions in fall 2004 (Turlock, CA) (Figure 2). Samples (4 L liquid/ 400 g powder, 5 sampling points in triplicate) were collected across processing facility run time (beginning ~2 hr, middle ~20 hr, end ~38 hr), packed in Mylar pouches (Impak; Los Angeles, CA), heat sealed, and frozen at -20°C. Frozen products were packed on dry ice and shipped by overnight carrier to North Carolina State University. Upon receipt, pouches were examined for damage and subsequently were stored at -20°C in the dark until analysis.

Proximate Analysis

Proximate analysis (ash, fat, protein, total solids, and microbial quality) was conducted in duplicate on each product. Fat, moisture, and solids content were determined by Mojonnier analysis (Mojonnier Bros. Co.; Chicago, IL). Ash was determined by muffle furnace and protein content by the Kjeldahl method. A conversion factor of 6.38 was used to convert total nitrogen to protein. Microbial analysis (coliform, total plate count, thermal tolerant spores) was conducted. Products were directly plated or serially diluted in 0.1% peptone water. Total aerobic bacteria were enumerated using plate count agar (PCA) and coliforms were enumerated using violet red bile agar followed by incubation at 32°C for 24 h (Christen and others, 1993). Thermotolerant spores were enumerated using a method adapted from Wehr and Frank, 2004. The sample was first heated to 80°C for 12 minutes to activate the spores, then plated onto milk plate count agar with 2% starch overlaid with PCA with 2% starch followed by incubation at 55°C for 48 h in humid conditions (adapted from Frank and Yousef, 2004).

Descriptive Analysis

Products were each evaluated using the descriptive sensory language and methods described by Drake and others (2003) (Table 1). The evaporated milk and SMP were reconstituted (9% SNF) using deodorized water. Evaporated product was also evaluated directly (undiluted). Fluid samples (30 mL) and undiluted evaporated milk (5g) were dispensed into 2 oz soufflé cups with lids and 3 digit codes. Sensory analysis was conducted at 12°C. Six trained panelists (5 female, 1 male) evaluated each sample in duplicate. Each panelist had more than 50 h of training on the descriptive sensory analysis of dried dairy ingredients with a previously identified descriptive language and the Spectrum™ descriptive analysis method (Drake and others 2003; Meilgaard and others 1999).

Direct Solvent Extraction

Direct solvent extraction was used to extract the volatiles in each sample. Samples were extracted in duplicate using anhydrous ethyl ether (DriSolv®, EMD chemicals; Ronstandt, Germany). The volume of total solids and the ether to volume ratio was standardized for solvent extraction. The fluid samples required more fluid volume as well as a greater volume of ether compared to rehydrated samples (evaporated milk, SMP). Evaporated milk and SMP were rehydrated (10% solids) using deodorized water (250 mL). Fluid milks (raw, balance tank, and HTST pasteurized, 522 mL) and rehydrated milks (evaporated and SMP, 250mL) were placed in clear Pyrex glass beakers (Corning; Acton, MA). Ten µL of internal standard (0.185 ppm 2-methyl-3-heptanone + 2-methyl valeric acid in methanol; Aldrich Chem. Co.; Milwaukee, WI) was added to each sample, and the sample pH was measured with an Orion 250A+ pH meter (Thermo Electron Corp.; Waltham, MA). Commercial sodium chloride (90 g) was added to each sample, and the sample pH was measured using color coded pH indicator strips (EM Science; Gibbstown, NJ). Samples (65-75 mL) were dispersed into either 4 or 6 Teflon coated centrifuge bottles (Nalgene; Rochester, NY). Ether (75 ml for fluid milk, 50 mL for rehydrated samples) was added to each of the centrifuge bottles. The bottles were shaken for thirty minutes using a motorized shaker (Thermolyne RotoMix 50800, Thermolyne; Dubuque, IA) before they were centrifuged (Du Pont Sorvall RC-5B

Refrigerated Superspeed Centrifuge, Du Pont Instruments; Wilmington, DE) at 735 x g for 15 minutes. This process was repeated twice, first with the addition of 50 mL of ether, and again with the addition of 25 mL of ether for all samples. The bottle was subsequently centrifuged without addition of ether at 735 x g for 20 minutes. After each centrifugation, the ether was collected from each bottle and pooled into a Quorpak clear wide mouth jar (Fisher; Pittsburgh, PA) and stored at -20°C. Using a Vigreux column, each sample was concentrated to 50 mL in a round bottom flask at 40°C in a water bath (Isotemp 110, Fisher; Pittsburgh, PA), transferred to small Quorpak jars (250 mL, Fisher; Pittsburgh, PA), and stored at -20°C until SAFE.

Solvent Assisted Flavor Evaporation (SAFE)

Volatile compounds were distilled from solvent extracts using SAFE (Ace Glassware; Vineland NJ), similar to the assembly described by Engel and others (1999). The SAFE apparatus was connected to a receiving tube and waste tube, which were both held in separate Dewar flasks containing liquid nitrogen at all times. The glassware assembly was connected to a rough pump/diffusion pump combination as a vacuum source. Liquid solvent samples were loaded into the top of the SAFE apparatus and released drop-wise into the vacuum. Using a circulating water bath (50 ^{o}C), the SAFE apparatus was thermostated throughout the distillation, which was carried out for 2 hours under vacuum (ca. 10^{-5} Torr).

Following SAFE, the sample was concentrated under nitrogen gas (<5 psi) to 20 mL. The concentrate was washed with 3 mL of 0.5 M Na_2CO_3 twice and shaken by hand. After each wash, the water phase was removed into another tube. The concentrate was then washed again with 2 mL saturated NaCl solution 3 times and shaken by hand.

Again, the water phase was removed using a pipette and collected into the same test tube. The ether phase in the original tube was the neutral/basic fraction of the concentrate and was complete. The pH of the aqueous phase was adjusted to ~2.0 using 18% HCl (Karagul-Yuceer and others, 2001). To this, 5 mL of ethyl ether was added three times and the tube was shaken by hand. The ether phase was removed with a pipette and placed into a clean test tube. This ether phase was the acid fraction of the concentrate. Both fractions were dried by passing through glass tubes containing anhydrous sodium sulfate and concentrated to 0.5 mL using nitrogen gas. Extracts were placed into small screw cap vials (2 mL) and stored at -20°C.

Gas Chromotography – Olfactometry(GC-O)and AEDA

Prior to sniffing, 200 µL of each extract was placed into a new screw cap vial containing an insert. The sample was concentrated inside the insert to 50 µL using a stream of nitrogen gas. GC-O was performed using an HP5890 series II gas chromatograph (Hewlett-Packard Co., Palo Alto, CA) equipped with a flame ionization detector (FID), a sniffing port, and a splitless injector. Each sample (2 µL) was injected onto a non-polar DB-5 column (DB-5, 30 m, 0.25µm i.d., J&W Scientific, Albany, NY) and a polar DB- Wax column (DB-Wax, 30 m, 0.25µm i.d., J&W Scientific, Albany, NY) with. Column effluent was split 1:1 between the FID and the sniffer port. Oven temperature was initially 40°C for 3 min., then 15°C/min to a final temperature of 200°C for 15 min. Each extract was sniffed on each column by 2 experienced sniffers, each with more than 50 h training on GC-O. Sniffers recorded aroma intensity and aroma character (Van Ruth, 2001).

Aroma extract dilution analysis (AEDA) was used to characterize crucial aroma active compounds contributing to flavor in the extracts (Grosch, 1993; Van Ruth, 2001). The extracts were diluted stepwise with diethyl ether at a ratio of 1/3 (v/v). Two experienced sniffers, each with greater than 50 h training on GC/O, were used for AEDA. The dilution procedure was followed until sniffers detected no odorants. The highest dilution was reported as the $\log_3$ flavor dilution (FD) factor (Grosch, 1993).

Gas Chromotography Mass Spectrometry

For GC/MS analysis of the solvent extracts, a HP5890 Series II GC/HP 5972 mass selective detector (MSD, Hewlett-Packard, Co.) was used. Separations were performed on a fused silica capillary column (DB-5MS 30 m length x 0.25 mm i.d. x 0.25 µm d_f, J&W Scientific). Helium gas was used as a carrier at a constant flow of 1 mL/min. Oven temperature was programmed from 40 °C to 200 °C at a rate of 5 °C/min with initial and final hold times of 5 and 45 min, respectively. MSD conditions were as follows: capillary direct interface temperature, 280 °C; ionization energy, 70 eV; mass range, 33-330 a.m.u; EM voltage (Atune+200 V); scan rate, 5 scans/s. Each extract (2 µL) was injected in the splitless mode. Duplicate analyses were performed on each sample. Relative concentrations of compounds were calculated based on recovery of the internal standard.

Compound Identification

For positive identification of each compound, retention indices (RI), mass spectra, and odor properties of unknowns were compared with those of authentic standard compounds analyzed under identical conditions. Tentative identifications were based on comparing mass spectra of unknown compounds with those in the National Institute of Standards and Technology (1992) mass spectral database or on matching the RI values and odor properties of unknowns against those of authentic standards. For the calculation of retention indices, an n-alkane series was used (Van den Dool and Kratz, 1963).

Compound Quantification

Response factors of selected compounds were calculated by direct addition of known amounts of standards to odor-free water prior to solvent extraction/SAFE. Response factors for the chosen acidic compounds were determined using a five-point standard curve ($R^2 > 0.90$) on a DB-WAX column using GC/FID. Using these response factors, the selected compounds were quantified using the response factor and the area ratio of compound to internal standards. Butanoic acid, hexanoic acid, octanoic acid, decanoic acid, and 2-methyl butanoic acid were selected for quantification as they were ubiquitous in all samples and were within linear detection range of the GC-MS. All standards were obtained from Aldrich Chemical Company (St. Louis, MO).

Solid Phase Microextraction (SPME)

Solid phase microextraction (SPME) was also used to analyze volatile compounds in each sample. Evaporated milk and SMP were re-hydrated to 10% solids using deodorized water. Each liquid sample (100 mL) was transferred into pre-washed Quorpak clear standard wide mouth jars (250 mL, Fisher; Pittsburgh, PA), followed by the addition of 2.5 µL of internal standard (0.185 ppm 2-methyl-3-heptanone in methanol; Aldrich Chem. Co.; Milwaukee, WI). The sample (20 g) was transferred into pre-cleaned 40 mL amber vials with screw caps and Teflon silica septa (Supelco;

Bellefonte, PA). A magnetic octagonal stirring bar (8mm OD x 13mm L, Fisher; Pittsburgh, PA) and commercial sodium chloride (1 g) were added. Each sample was allowed to equilibrate in the vial at 47°C for 30 minutes using a heating/stirring module (Reacti-Therm™, Pierce Biotechnology, Inc.; Rockford, IL).

Volatile odor compounds were extracted by exposing a 2 cm-50/30 µm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) StableFlex SPME fiber (Supelco; Bellefonte, PA) to the headspace of each sample at 47°C for 30 minutes. Following extraction of the volatile odor compounds, the SPME fiber was inserted into a splitless injection port (250°C) of a 5890 Series II Plus gas chromatograph (Hewlett Packard, Palo Alto, CA) with a fused nonpolar silica capillary column (DB-5, 30 m, 0.25µm i.d., J&W Scientific, Albany, NY). Helium gas was used as a carrier at a constant flow of 1mL/min. The initial temperature of the oven was 40°C with a hold time of 5 min., increasing 8°C per minute to a final temperature of 250°C, which was held for 5 min. The detector was a 5972 Series Mass Selective Dectector (Hewlett Packard, Palo Alto, CA). MSD conditions were as follows: capillary direct interface temperature 280°C, ionization energy 70eV, mass range 33-330 a.m.u., EM voltage (Atune + 200V), scan rate, 5 scans/sec. Each sample was analyzed in quadruplicate. Volatile compounds were identified using the NIST02 mass spectral library, and retention indices of authentic standard compounds analyzed under identical conditions.

Statistical Analysis

Analysis of variance with means separation (least square means) was conducted to determine differences between samples and differences between sample collection time (beginning, middle, or end of production run). Both main effects (sample, collection time) as well as interactions (sample*collection time) were addressed. Statistical analysis was performed using SAS, version 8.2 (Cary, NC).

Results and Discussion

Proximate Analysis

Proximate analysis results showed no differences in composition of samples from beginning, middle, and end of production ($p>0.05$) (data not shown). The percentage of fat in the raw milk was much higher than in other samples (Table 2). This was expected, as the raw milk was the only sample taken before skimming. The percentage of protein and ash increased as the moisture decreased in the evaporated and SMP.

Microbiological analysis also showed no significant difference between samples across production ($p>0.05$) (data not shown). Raw milk samples had higher coliform counts than other samples (Table 3). This result is not surprising because the raw milk is the only sample that has not undergone heat treatment. The mild heat treatment of the bulk tank sample is adequate to eliminate coliforms, which explains the decrease between raw and bulk tank samples.

Sensory Analysis

The sensory attributes of the milk changed significantly throughout the SMP production process ($p<0.05$) (Tables 4 and 5). The flavors of fluid milk and SMP are delicate and thus intensities are low, consistent with previous studies (Karagul-Yuceer and others, 2001, 2002; Drake and others, 2003). Milkfat flavor was high in the raw milk and decreased at subsequent stages due to skimming before entering the bulk tank. Metallic/serum and feed flavors were found in the raw and bulk tank milks and decreased to non-detectable levels with increasing heat treatment. These are flavors typically associated with raw milk. Cooked flavor increased as the heat treatment increased. The sweet aromatic flavor increased throughout the process, with the exception of the evaporated sample. SMP is typically much higher in sweet aromatic flavor compared to fluid milk (Drake and others, 2003) and this is likely due to caramelization compounds produced during heat treatment (Karagul-Yuceer and others, 2004). The volatile flavors, except sweet aromatic, were consistently higher in the undiluted evaporated sample than in other samples.

There was not a collection time effect for any processed (heat treated sample) ($p>0.05$), suggesting that equipment burn-on or soiling throughout 40 h of production does not impact sensory properties of the finished product. However, low levels (intensity = 0.75) of free fatty acid were detected in the raw milk from the first collection, indicative of hydrolytic rancidity. Free fatty acid flavors were not detected in subsequent processed samples from this collection point.

GC-O (Post peak intensity)

Thirty nine compounds were identified in milk samples throughout the SMP production process (Tables 6,7,8). Ethyl hexanoate was found in raw milk from the end of production samples. This compound was previously identified as one of the most potent odorants in raw cows' milk (Friedrich and Acree, 1998). Butanoic acid (cheesy) and hexanoic acid (sour/sweaty) were found in samples from each stage throughout the run. Other free fatty acids were found only from samples at the middle and end of the production run. These acids were pentanoic (sour/sweaty) and 2/3-methyl butanoic (cheesy/dried fruit) acids. Karagul-Yuceer and others (2001) documented these free fatty acids, caused by fat hydrolysis, as major contributors to skim milk powder flavor. Other flavors identified by post peak intensity were heat generated compounds, including methional, 2-acetyl-1-pyroline, and homofuraneol, which have potato, popcorn-like, and burnt sugar aromas, respectively. All of these compounds have been documented as thermally induced in SMP (Karagul-Yuceer and others, 2001). Hexanal and δ-decalactone were also found in some of the heated samples. Both compounds were noted as potent aroma-active compounds in heated milk (Friedrich and Acree, 1998). The intensities of these compounds increased from the raw milk to the skim milk powder, as the heat treatment increased. Few changes were seen throughout the production run from beginning to end. The most prominent difference between the timepoints was that there were more compounds identified in samples from the end of the run versus beginning and middle samples (Table 8). Compounds such as o-cresol (cowy), (E,E)-2,4-nonadienal (earthy/sweet/nutty), 2-methyl-3-furanthiol (brothy/dirty), and 2-acetyl-2-thiazoline (popcorn/sweet) could be due to burn-on residues and increased at the end of the production run.

AEDA

Aroma extract dilution analysis is a semi-quantitative method by which the most potent aroma active compounds can be characterized (Grosch, 1993). Volatile extracts are serially diluted and evaluated by GC-O until compounds are not detected and a dilution factor or $\log_3$ FD value is generated (Audouin and others, 2001). AEDA was performed on samples from the end of the production run based on the post peak intensity results, as there were a larger number of compounds, representative of all 3 timepoints, found in these samples. The most prominent changes in volatile compounds occurred at the major heating steps: HTST pasteurization, evaporation, and spray drying. Therefore, we conducted AEDA on the raw milk, HTST pasteurized milk, evaporated milk, and SMP (Table 8). Methional and 2-acetyl-1-pyroline have $\log_3$ FD values of 4 and 3, respectively in the HTST pasteurized milk. Methional had a $\log_3$ FD value of 3 in evaporated milk and 1 in SMP, indicating that it is an important compound in these samples. This compound likely contributes to the higher cooked flavor in these samples. Butanoic and 2/3-methyl butanoic acids had $\log_3$ FD values of 2-4 in all samples where the compound was detected. The $\log_3$ FD value of butanoic acid decreased from raw milk to SMP, and the $\log_3$ FD value of 2/3-methyl butanoic acid stayed constant among samples, but was not detected in the SMP. These compounds are most likely very important in milk flavor across the entire production process.

AEDA was also conducted on all three SMP timepoints (beginning, middle, and end) to determine if differences exist throughout the production process. Aldehydes have been documented to contribute to SMP flavor (Karagul-Yuceer and others, 2001). In these SMP, nonanal had a high $\log_3$ FD value (6) in the middle of the run SMP, but was not detected in the other SMP. (E,Z)-2,6-nonedienal had a $\log_3$ FD value of 3 in the beginning, 5 in the middle, and 1 in the end of run SMP. Previous research by Karagul-Yuceer and others (2001) found that heat induced compounds, such as methional and 2-acetyl-1-pyroline, as well as free fatty acids (butanoic acid and pentanoic acid) caused by hydrolysis of milkfats, are important odorants in SMP. The same study also suggested that the number of compounds, as well as their concentrations, contributes largely to

SMP flavor. Butanoic acid had a higher log_3 FD value in samples from the beginning of the run compared to the middle and end of the run. Propionic acid also had a high dilution factor in the beginning sample (log_3 FD value = 5) but less than 1 in subsequent samples. Methional and 2-acetyl-1-pyroline had lower log_3 FD values in the end of run samples than the other samples. These results suggest that fewer, more potent compounds make up the flavor of the beginning and middle samples, but more compounds (less potent) combine to make up the flavor of the end of production samples. Previous research has shown that raw milk has fewer common aroma-active compounds present (7) than heated milk (15) and that the potency of the compounds was different between the samples (Friedrich and Acree, 1998). Sensory differences were not detected between the three SMP. However, differences in volatile composition may contribute to differences in flavor stability and should be addressed in future work.

Quantification

Five acids (Butanoic, hexanoic, octanoic, decanoic, and 2/3-methyl butanoic acid) were chosen for quantification. These compounds were chosen because they are ubiquitous in all samples as well as within linear detection range of the GC-MS. Tables 9 and 10 show the quantification of compounds across SMP production. The only statistically significant difference was in the raw milk sample at the beginning of the run, which was consistent with sensory results. Sensory results indicated a low level of free fatty acid flavor in the raw milk from this set of samples. The concentration of 2/3-methyl butanoic acid in this sample was significantly higher than in other samples ($p<0.05$). Table 11 shows the quantification of each compound across the production run. There were no significant differences in the concentration of fatty acids among each SMP with the exception of 2/3 methyl butyric acid which was consistently higher in all products from the first production run. This result suggests that the free fatty acid flavor noted in the raw milk was not due to a problem in shipment of the raw milk sample, but something consistent in this sample of milk and thus in all product produced from this milk.

SPME

The mean relative abundance of selected volatile compounds found in each of the SMP is given in Table 12. There were few notable differences between compounds in the raw milk compared with other samples. However, the one exception was nonanoic acid. This particular compound was detected only in the raw milk samples. Dodecanoic acid was also detected at slightly higher levels in the raw milk samples. Several compounds, decanal, dodecanal, nonanal, and hexanal, were detected at higher levels in the evaporated milk and SMP than in other samples. Hexanal has previously been identified as being unique to heated milk samples (Friedrich and Acree, 1998). Nonanal was also noted previously to be an active aroma component in high-heat treated SMP (Karagul-Yuceer, 2001), so it is not surprising that these compounds were found at higher concentrations in the samples with the highest heat treatments.

Conclusions

The results of this study show that heat generated compounds increased in milk products which recieved higher heat treatment, such as pasteurized and evaporated milk, and SMP. Fatty acids did not largely differ in post peak intensity or $\log_3$ FD value across samples, indicating that their role in flavor was constant across samples. No sensory differences were found among the SMP from the beginning, middle or end of the production run, although instrumental differences were detected in the number of volatile compounds. It is important to note that heat treatments are the most important steps in SMP processing that contribute to the flavor of the final fresh product.

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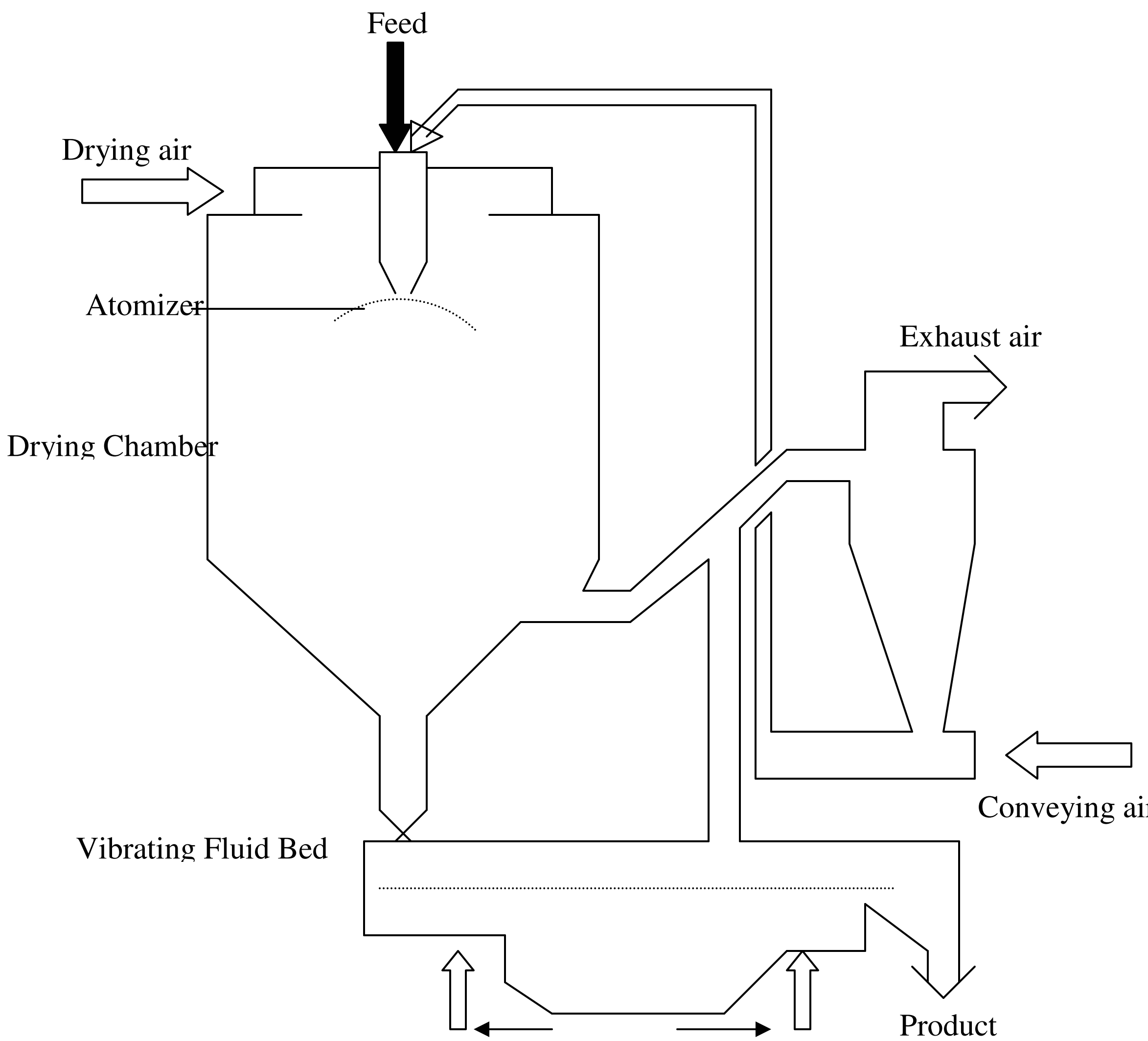
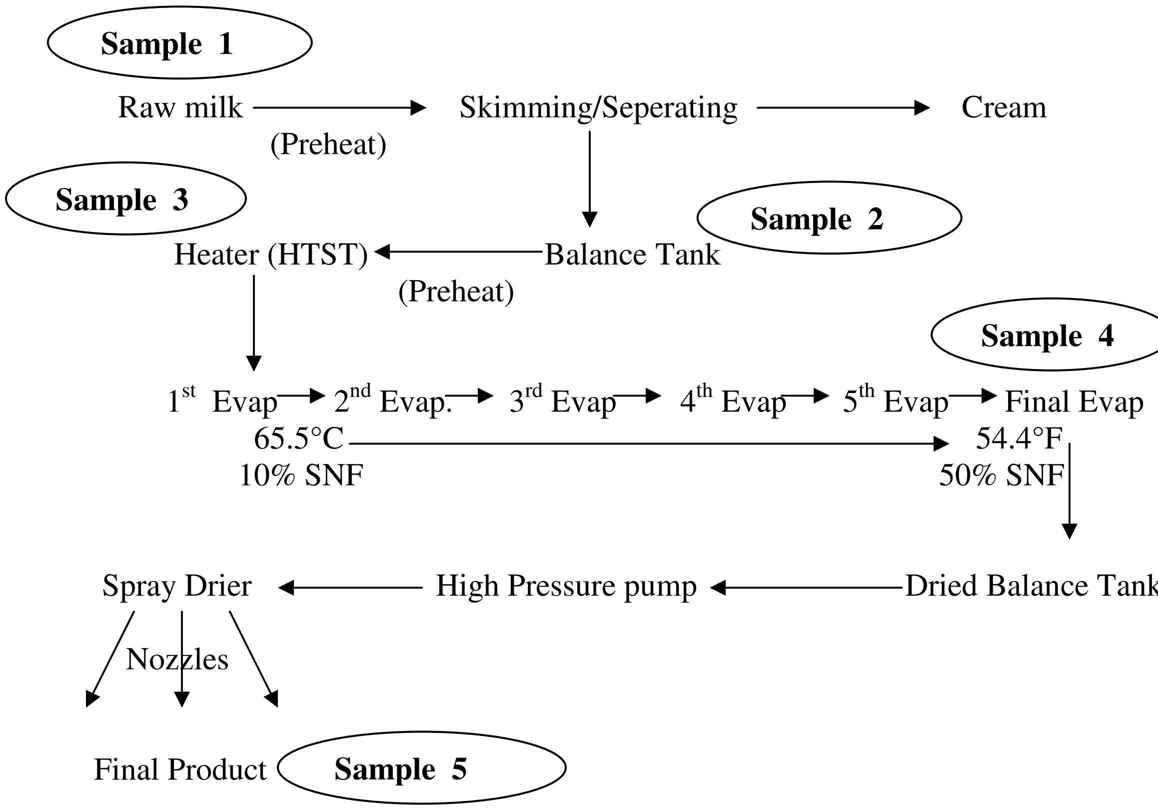
Figure 1: Diagram of a two-phase Spray Dryer



Adapted from Varnum and Sutherland, 1994

Figure 2: Product sampling points



Circled numbers represent sampling points.

Table 1: Descriptive sensory language for fluid milk and rehydrated SMP.

Descriptor	Definition	Reference	Preparation
Cooked/sulfurous	-Aromatic associated with cooked milk	-Heated milk	-Heat pasteurized skim milk to 85ºC for 45 min
Sweet aromatic/ cake mix	-Sweet aromatic associated with dairy products	-Pillsbury-White cake mix -vanillin	-dilute 5mg of vanillin in skim milk
Vitamin/rubber	-Aromatic associated with vitamin supplements or rubber	-Enfamil – liquid Polyvisol vitamins	
Free Fatty Acid		Butyric acid	-20 ppm in skim milk
Metallic/Serum-like	-Aromatic associated with rare steak juice	Rare steak juice	
Sweet taste	-Basic taste associated with sweetness	-sucrose	-5% sucrose solution
Salty	-Basic taste associated with saltiness	-NaCl	-2% NaCl solution
Sour	-Basic taste associated with sourness	-citric acid	-1% citric acid solution
Bitter	-Basic taste associated with bitterness	-caffeine	-0.5% caffeine solution
Astringent	-Drying tongue sensation	-tea	-soak 6 tea bags in water for 10 min

Adapted from Drake and others 2003; Carunchia-Whetstine and others, 2002

Table 2: Proximate analysis for samples across SMP production

Analyte	Raw	BT	HTST	Evap	SMP
Ash (%)	0.68 (0.05)[1]	0.71 (0.04)	0.71 (0.04)	3.51 (0.11)	7.72 (0.02)
Fat (%)	3.52 (0.02)	0.06 (0.01)	0.06 (0.01)	0.29 (0.07)	0.67 (0.04)
Protein (%)	3.22 (0.02)	3.35 (0.01)	3.34 (0.02)	15.96 (0.62)	35.66 (0.09)
Solids (%)	12.13 (0.04)	9.08 (0.11)	9.09 (0.04)	44.54 (1.69)	ND
Moisture (%)	87.87 (0.04)	90.93 (0.11)	90.94 (0.05)	54.44 (1.66)	3.45 (0.13)

[1]Numbers in parentheses are standard deviations across samples.

Table 3: Microbiological analysis for samples across SMP production

Media	Raw	BT	HTST	Evap	SMP
VRB coliform CFU/mL	8230 (5800)	<10 (ND)	<10 (ND)	<10 (ND)	<10 (ND)
TPC CFU/mL	18570 (12500)	4300 (2400)	610 (550)	1170 (1150)	5000 (2300)
Thermospores CFU/mL	<1 (ND)	650 (920)	300 (370)	2130 (1980)	2770 (1330)

Table 4: Sensory attributes of SMP throughout the production process[D]

Attribute	Raw Milk	Bulk Tank	HTST	Evaporated	Evaporated (9% SNF)	SMP
Cooked	ND	1.92d	3.50c	6.00a	1.83d	4.25b
Caramelized	ND	ND	ND	3.67a	1.08b	1.58b
Milkfat	2.50a	0.92b	0.52c	1.25b	ND	ND
Sweet aromatic	1.42c	2.28b	2.77a	1.50c	1.08d	2.93a
Sweet taste	2.00c	2.67b	2.95ab	3.55a	1.41d	2.55b
Salty taste	0.58c	0.58c	ND	4.00a	1.50b	0.58c
Feed	1.42a	1.33a	1.42a	ND	ND	ND
Free fatty acid[1]	0.65	ND	ND	ND	ND	ND
Vitamin	ND	ND	ND	1.58a	0.50b	ND
Metallic/serum	1.67a	0.67b	ND	ND	ND	ND
Astringency	1.08d	1.25cd	1.67ab	1.75ab	2.25ab	2.46a

[A] Intensities are scored on a 15-point scale where 0 = none and 15 = very high (Drake and others, 2003; Meilgaard and others, 1999)
[B] Means in a row followed by different letters are different ($p<0.05$).
[C] ND=not detected
[D] There was not a collection time effect ($p>0.05$).
[1] There was a collection time effect for this attribute for raw milk collected at the beginning of the production run. See Table 5.

Table 5: Sensory attributes of each sample across SMP production

	Raw			BT			HTST			Evap			SMP		
Attribute	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
Cooked	ND	ND	ND	1.75	2.00	2.00	3.25	3.25	4.00	4.50 (2.25)[1]	3.00 (1.50)	4.50 (1.75)	4.25	4.00	4.50
Caramelized	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND (ND)	0.50 (ND)	1.50 (0.25)	ND	ND	1.75
Milkfat	2.25	2.75	2.50	0.50	1.25	1.00	0.50	0.75	ND	ND (ND)	0.75 (0.50)	ND (ND)	ND	ND	ND
Sweet aromatic	1.25	2.00	1.00	2.40	2.50	2.00	2.60	3.25	2.50	1.50 (1.50)	1.50 (0.75)	1.50 (1.00)	2.50	1.75	2.75
Sweet taste	1.75	2.00	2.25	2.00	2.50	3.25	2.50	3.15	3.25	2.50 (1.75)	3.25 (1.00)	4.00 (1.50)	2.00	2.25	2.50
Salty taste	ND	ND	ND	ND	0.50	1.25	ND	0.50	0.50	3.50 (ND)	2.00 (ND)	3.50 (1.25)	0.50	ND	0.25
Feed	ND	1.25	ND	ND	1.00	ND	ND	1.25	ND	ND (ND)	ND (ND)	ND (ND)	ND	0.50	ND
Free fatty acid[2]	0.75	ND	ND	ND	ND	ND	ND	ND	ND	ND (ND)	ND (ND)	ND (ND)	ND	ND	ND
Vitamin	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.75 (1.50)	ND (ND)	ND (ND)	ND	ND	ND
Metallic/serum	ND	ND	2.00	ND	ND	0.50	ND	ND	ND	ND (ND)	ND (ND)	ND (ND)	ND	ND	ND
Astringency	1.50	ND	1.75	1.50	1.00	1.25	1.75	1.00	2.25	2.00 (2.25)	1.00 (1.00)	ND (2.25)	2.25	1.75	1.80

[1]Values in parentheses represent intensity of attributes in evaporated sample diluted to 9% SNF
[2] Free fatty acid was the only attribute impacted by sampling time

Table 6: Summary of aroma-active compounds identified in SMP throughout processing (beginning of run).

no.	compound	fraction	odor at the sniffer port[1]	RI[2]		post peak intensity at the sniffer port (n=4) (Log$_3$ dilution factor)[3]					
				DB-5MS	DB-WAX	Raw Milk	Bulk Tank	HTST	Evap-orated Milk	SMP	method of identification[1-4]
1	4-methyl-3-penten-2-one	NB	Plastic/solventy	788		1.5	ND	ND	1.6	1.3(<1)	RI, odor
2	hexanal	NB	Green/sweet	823		ND	ND	ND	1.5	4.0	RI, odor
3	unknwn	NB	Skunk	836		ND	2.0	ND	ND	ND	RI, odor
4	Butanoic acid	Ac	Rancid Cheese	858	1603	2.2	ND	1.8	1.5	2.5(5)	RI, odor, MS
5	Propionic acid	Ac	Cheesy/sour	865	1523	3.7	3.2	2.0	1.2	3.0(5)	Odor
6	unknown	NB	Fruity	866		1.3	ND	1.17	ND	2.3(<1)	RI, odor
7	methional	NB	Potato	933		ND	3.0	2	2	3(3)	RI, odor
8	2-acetyl-1-pyroline	NB	Popcorn	942		1.5	1.8	3.0	2.25	3.1(2)	RI, odor
9	DMTS	NB	Cabbage	987		ND	1.0	2.5	2	2.6(3)	RI, odor
10	1-octen-3-one	NB	Mushroom/metallic	995		1.9	1.8	1.0	1.9	2.5(1)	RI, odor
11	Hexanoic acid	Ac	Sour/sweaty	1013	1852	1.8	ND	1.5	1.5	1.5(1)	RI, odor, MS
12	octanal	NB	Fruity/citrus	1020		1.5	2.2	ND	1.6	2.5(<1)	RI, odor, MS
13	(E)-2-octenal	NB	Stale/musty	1084		1.8	1.5	1.2	1.5	ND	RI, odor
14	nonanal	NB	Earthy/fatty	1102		ND	2.0	ND	ND	ND	RI, odor, MS
15	unknown	NB	Earthy/burnt	1110		ND	ND	2.3	2.8	2.3(<1)	Odor
16	homofuraneol	Ac	Burnt sugar	1121	2045	ND	ND	ND	1.3	3.0(3)	RI, odor
17	3,6-nonadienal	NB	fatty	1123		2.0	2.0	2.0	2.2	2.8(<1)	RI, odor
18	unknown	NB	Oxidized/roasted	1137		ND	ND	2.0	2.2	2.5(<1)	Odor
19	(E,Z)-2,6-nonedienal	NB	Rosy/cucumber	1169		1.8	1.6	1.5	2.0	2.0(3)	RI, odor
20	Butyl hexanoate	NB	Cucumber/floral	1204		ND	ND	ND	ND	3.0(3)	RI, odor
21	decenal	NB	Old books	1229		ND	2.0	ND	ND	1.5(1)	RI, odor, MS
22	Unknown	NB	Rosy/waxy	1341		1.5	1.5	1.8	ND	ND	Odor
23	g-nonalactone	NB	Sweet/honey	1416		ND	ND	ND	ND	1.2(2)	RI, odor
24	g-decalactone	NB	Sweet/fruity	1524		1.2	ND	ND	ND	ND	RI,odor
25	g-dodecalactone	NB	peach	1695		1.5	1.5	2.0	ND	ND	RI, odor
26	d-dodecalactone	NB	peach	1736		1.0	1.0	2.5	ND	1.0(<1)	RI, odor

Table 6 continued:

[1] Odor description at the GC-sniffing port.
[2] Retention indices were calculated from GCO data.
[3]Flavor dilution factors were determined on a DB-5 column for NB compounds, and on a DB-Wax column for Ac compounds.
[4] Compounds were identified by comparison with the authentic standards on the following criteria: retention index (RI) on DB-Wax and DB-5MS columns, odor property at the GC-sniffing port, and mass spectra in the electron impact mode

Table 7: Summary of aroma-active compounds identified in SMP throughout processing (middle of run).

no.	compound	fraction	odor at the sniffer port[1]	RI[2]		post peak intensity at the sniffer port (n=4) (Log$_3$ dilution factor)[3]					
				DB-5MS	DB-WAX	Raw Milk	Bulk Tank	HTST	Evap-orated Milk	SMP	method of identification[1-4]
1	3-hydroxy-2-butanone	NB	Buttery	709		ND	1.0	ND	ND	ND	RI, odor
2	4-methyl-3-penten-2-one	NB	Plastic/solventy	781		ND	ND	ND	ND	1.5(<1)	RI, odor
3	hexanal	NB	Green/sweet	815		ND	ND	1.0	1.5	ND	RI, odor
4	Butanoic acid	Ac	Rancid cheese	835	1680	2.5	2.2	4.0	1.5	3.5(2)	RI, odor, MS
5	2/3 methyl butanoic	Ac	Cheesy/dried fruit	930	1605	3.8	3.0	ND	3.0	4.3(3)	RI, odor, MS
6	unknown	NB	Fruity	866		ND	1.5	ND	ND	ND	Odor
7	Pentanoic acid	Ac	Sweaty/waxy	930	1753	1.5	2.0	ND	ND	1.5(<1)	RI, odor, MS
8	methional	NB	Potato	933		1.0	2.0	1.6	3.1	2.8(2)	RI, odor
9	2-acetyl-1-pyroline	NB	Popcorn	942		ND	2.7	2.0	ND	2.7(2)	RI, odor
10	DMTS	NB	Cabbage	993		ND	1.8	ND	ND	1.2(3)	RI, odor
11	1-octen-3-one	NB	Mushroom/metallic	1000		2.0	1.2	ND	1.5	1.5(<1)	RI, odor
12	Hexanoic acid	Ac	Sour/sweaty	1013	1852	1.5	ND	ND	ND	ND	RI, odor, MS
13	octanal	NB	Fruity/citrus	1020		ND	ND	1.8	ND	2.0(1)	RI, odor, MS
14	(E)-2-octenal	NB	Stale/musty	1084		2.0	1.4	ND	ND	1.7(<1)	RI, odor
15	nonanal	NB	Earthy/fatty	1102		ND	2.5	ND	4.0	2.5(6)	RI, odor, MS
16	unknown	NB	Earthy/burnt	1110		ND	ND	ND	ND	2.0(<1)	Odor
17	homofuraneol	Ac	Burnt sugar	1121	2098	ND	ND	2.0	1.5	1.5(<1)	RI, odor
18	3,6-nonadienal	NB	fatty	1123		ND	1.5	2.0	2.5	ND	RI, odor
19	unknown	NB	Oxidized/roasted	1137		ND	ND	ND	3.0	2.5(2)	Odor
20	(E,Z)-2,6-nonedienal	NB	Rosy/cucumber	1169		ND	ND	ND	2.0	2.5(5)	RI, odor
21	Butyl hexanoate	NB	Cucumber/floral	1204		ND	ND	ND	2.5	ND	RI, odor
22	decenal	NB	Old books	1235		1.5	ND	ND	1.5	ND	RI, odor, MS
23	Octanoic acid	Ac	sweaty	1398	2117	2.0	1.7	ND	ND	ND	RI, odor, MS
24	g-nonalactone	NB	Sweet/honey	1416		ND	ND	ND	1.5	ND	RI, odor
25	o-AAP	NB	Grape/tortilla	1573		ND	ND	ND	ND	1.5(<1)	RI, odor
26	g-dodecalactone	NB	Peach	1695		1.5	1.5	2.0	ND	ND	RI, odor
27	d-dodecalactone	NB	Peach	1736		1.2	ND	ND	ND	ND	RI, odor

Table 7 continued:

[1] Odor description at the GC-sniffing port.
[2] Retention indices were calculated from GCO data.
[3]Flavor dilution factors were determined on a DB-5 column for NB compounds, and on a DB-Wax column for Ac compounds.
[4] Compounds were identified by comparison with the authentic standards on the following criteria: retention index (RI) on DB-Wax and DB-5MS columns, odor property at the GC-sniffing port, and mass spectra in the electron impact mode

Table 8: Summary of aroma-active compounds identified in SMP throughout processing (end of run).

no.	compound	fraction	odor at the sniffer port[1]	RI[2] DB-5MS	RI[2] DB-WAX	post peak intensity at the sniffer port (n=4) (Log$_3$ dilution factor)[3] Raw Milk	Bulk Tank	HTST	Evaporated Milk	SMP	method of identification[1-4]
1	Diacetyl	NB	Buttery	640		ND	ND	1.5(<1)	ND	ND	RI, odor
2	2/3 methyl butanal	NB	Chocolate/malty	686		1.5(<1)	ND	ND	ND	ND	RI, odor
3	4-methyl-3-penten-2-one	NB	Plastic/solventy	794		1.5(<1)	1.2	1(<1)	ND	1.3(<1)	RI, odor
4	Ethyl butyrate	NB	fruity/sweet	815		2.5(3)	1.7	1.8(<1)	1.5(<1)	ND	RI, odor
5	2-methyl-3-furanthiol	NB	Brothy/dirty	830		ND	ND	1.5(<1)	ND	3.0(<1)	RI, odor
6	Butanoic acid	Ac	Rancid Cheese	858	1615	4.2(4)	3.5	3.3(2)	3.0(2)	3.1(3)	RI, odor, MS
7	2/3-methyl butanoic acid	Ac	Cheesy/dried fruit	930	1688	3.5(3)	4.1	4.0(3)	1.5(3)	ND	RI, odor, MS
8	Pentanoic Acid	Ac	Sweaty/waxy	930	1758	ND	ND	ND	3.6(<1)	3.3(<1)	RI, odor, MS
9	Propionic acid	Ac	Cheesy/sour	865	1477	3.7(<1)	3.2	2.0(<1)	1.2(<1)	2.5(<1)	RI, odor
10	unknown	NB	Fruity	866		3.5(<1)	ND	1.5(<1)	ND	ND	RI, odor
11	methional	NB	Potato	933		3.5(1)	2.2	2.8(4)	2.1(3)	2.7(1)	RI, odor
12	2-acetyl-1-pyroline	NB	Popcorn	942		ND	2.0	2.2(3)	1.8(<1)	2.1(<1)	RI, odor
13	Ethyl hexanoate	NB	fruity	972		2.0(<1)	ND	ND	ND	ND	RI, odor
14	DMTS	NB	Cabbage	987		3.5(<1)	2.6	3.1(4)	2.5(<1)	3.0(1)	RI, odor
15	1-octen-3-one	NB	Mushroom/metallic	995		1.5(<1)	ND	2.0(<1)	2.0(<1)	1.2(<1)	RI, odor
16	Hexanoic acid	Ac	Sour/sweaty	1013	1830	ND	ND	2.5(<1)	4.5(<1)	ND	RI, odor, MS
17	octanal	NB	Fruity/citrus	1020		1.7(<1)	1.5	2.0(<1)	2.0(<1)	2.0(<1)	RI, odor, MS
18	2-acetyl thiazole	NB	pocorn	1038		ND	ND	2.0(<1)	ND	2.0(<1)	RI, odor
19	(E)-2-octenal	NB	Stale/musty/fatty	1082		1.5(1)	1.0	ND	1.5(<1)	2.0(<1)	RI, odor
20	2-acetyl-2-thiazoline	NB	Popcorn/sweet	1100		ND	ND	ND	ND	3.0(1)	RI, odor
21	nonanal	NB	Earthy/fatty	1102		1.5(3)	1.5	2.5(3)	ND	ND	RI, odor, MS
22	2-phenethanol	ND	Rosey/cuke	1117		2.2(1)	3.5	ND	ND	2.5(<1)	RI, odor
23	homofuraneol	Ac	Burnt sugar	1121	2065	ND	ND	1.5(1)	1.5(<1)	1.5(1)	RI, odor
24	3,6-nonadienal	NB	fatty	1133		ND	1.0	2.5(<1)	2.5(<1)	2.8(<1)	RI, odor
25	unknown	NB	Oxidized/roasted	1137		ND	ND	2.0(1)	2.2(<1)	2.5(<1)	Odor
26	o-cresol	NB	cowy	1154		ND	ND	ND	ND	2.0(<1)	RI, odor
27	(E,Z)-2,6-nonedienal	NB	Rosy/cucumber	1169		2.0(1)	2.5	2.0(<1)	2.5(<1)	2.0(1)	RI, odor

Table 8 continued:

28	(E)-2-nonenal	NB	Old books	1186	2.2(<1)	1.9	2.4(<1)	1.8(<1)	2.5(<1)	RI, odor
29	2-isobutyl-3-methoxy pyrazine	NB	Bell pepper	1193	ND	ND	2.5(<1)	1.5(<1)	2.2(1)	RI, odor
30	Butyl hexanoate	NB	Cucumber/floral	1204	ND	2.0	ND	ND	ND	RI, odor
31	(E,E)-2,4-nonadienal	NB	Earthy/sweet/nutty	1220	1.5(1)	2.0	2.0(<1)	ND	ND	RI, odor
32	decenal	NB	Old books	1229	ND	1.5	ND	ND	3.5(<1)	RI, odor, MS
33	unknown	NB	Rosy/waxy	1341	1.5(1)	1.5	1.8(<1)	ND	ND	Odor
34	skatole	NB	fecal	1501	2.5(<1)	ND	2.5(1)	1.7(<1)	1.8(<1)	RI, odor
35	g-decalactone	NB	Sweet/fruity	1524	2.5(<1)	2.5	2.25 (<1)	ND	ND	RI, odor
36	g-octalactone	NB	coconut	1563	3.0(<1)	ND	ND	1.7(<1)	ND	RI, odor
37	6-(Z)-dodecen-g-lactone	NB	Peach/waxy/ coconut	1609	ND	1.8	2.5(<1)	2.0(<1)	1.3(1)	RI, odor
38	g-dodecalactone	NB	peach	1695	ND	2.5	2.7(3)	1.5(<1)	1.2(<1)	RI, odor
39	d-dodecalactone	NB	peach	1736	2.0(3)	2.2	2.0(<1)	ND	2.0(<1)	RI, odor

[1] Odor description at the GC-sniffing port.
[2] Retention indices were calculated from GCO data.
[3] Flavor dilution factors were determined on a DB-5 column for NB compounds, and on a DB-Wax column for Ac compounds.
[4] Compounds were identified by comparison with the authentic standards on the following criteria: retention index (RI) on DB-Wax and DB-5MS columns, odor property at the GC-sniffing port, and mass spectra in the electron impact mode

Table 9: Quantification of selected compounds across SMP production

Concentration (ppm)

	Raw			BT			HTST			Evap			SMP			
Compound	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	Threshold (ppm)
Butanoic Acid	59.1a[1] (48.5)[3]	21.3 (22.1)	4.5a (3.5)	37.1a (25.1)	1.3a (0.4)	1.3a (0.6)	58.6a (95.5)	2.0a (2.5)	0.2 (0.06)	45.3a (48.5)	2.4a (1.5)	0.8a (0.5)	35.8a (27.5)	1.6a (1.5)	3.8a (2.5)	0.90[2]
Hexanoic Acid	6.4a (6.7)	12.6a (13.4)	3.5a (2.9)	1.2a (1.2)	1.5a (0.1)	1.6a (0.7)	3.5a (5.6)	3.2a (4.3)	0.2 (0.1)	1.4a (0.9)	2.0a (1.3)	1.0a (0.8)	1.8a (1.7)	1.2a (1.3)	4.6a (3.9)	2.8[2]
Octanoic Acid	390a (502)	6.7a (6.3)	2.2a (1.9)	59.2a (41.6)	3.0a (0.2)	2.0a (1.4)	61.6a (98.7)	6.4a (9.3)	0.3 (0.1)	15.2a (117.7)	1.9a (1.1)	1.3a (1.3)	11.3a (10.0)	1.4a (1.7)	5.9a (5.0)	3.0[2]
Decanoic Acid	8.5a (12.1)	0.1a (0.1)	0.1a (0.1)	3.2a (2.9)	0.1a (ND)	0.1a (0.1)	2.4a (3.8)	0.2a (0.4)	ND	2.8a (5.6)	0.1a (0.1)	0.1a (0.1)	3.9a (5.8)	ND	0.3a (0.2)	10.0[2]
2-methyl Butanoic Acid	3077a (3846)	0.9b (1.2)	0.6b (0.1)	230b (160)	1.4b (ND)	1.3b (0.7)	210b (341)	3.2b (4.0)	0.8b (0.2)	60.4b (58.7)	3.0b (2.5)	1.8b (1.3)	90.4b (71.2)	1.9b (1.9)	5.4b (3.1)	2.4[2]

[1] different letters denote statistically significant differences.

[2] Thresholds reported orthonasally in water (Rychlik and others, 1998)

[3] Number is parentheses are standard deviations across 4 samples

Table 10: Quantification of selected compounds across SMP production (ppm)

	Concentration (ppm)					
Compound	Raw	BT	HTST	Evaporated	SMP	Threshold
Butanoic Acid	36.0a (41.6)	15.6a (23.5)	16.8a (50.5)	16.2a (33.2)	13.7a (21.8)	0.9[2]
Hexanoic Acid	7.2a (7.6)	1.4a (0.8)	2.2a (3.8)	1.5a (1.0)	2.5a (2.8)	2.8[2]
Octanoic Acid	197a (388)	25.1a (38.0)	19.2a (52.2)	6.1a (11.4)	6.2a (7.3)	3.0[2]
Decanoic Acid	4.3a (9.1)	1.3a (2.3)	0.7a (2.0)	1.0a (3.2)	1.4a (3.6)	10.0[2]
2-methyl Butanoic Acid	1539a (3007)	92.8b (150)	58.6b (181)	21.7b (41.9)	32.6b (56.7)	2.4[2]

[3] Thresholds reported orthonasally in water (Rychlik and others, 1998).
Means in a row followed by different letters are different ($p<0.05$).
Numbers in parentheses are standard deviations across samples

Table 11: Quantification of selected compounds throughout SMP production (ppm)

Concentration (ppm)

Compound	SMP 1	SMP 2	SMP 3	Threshold
Butanoic Acid	35.8a (27.5)	1.6a (1.5)	3.8a (2.5)	0.9[1]
Hexanoic Acid	1.8a (1.7)	1.2a (1.3)	4.6a (3.9)	2.8[1]
Octanoic Acid	11.3a (10.0)	1.4a (1.7)	5.9a (5.0)	3.0[1]
Decanoic Acid	3.9a (5.8)	ND	0.3a (0.2)	10.0[1]
2-methyl Butanoic Acid	90.4a (71.2)	1.9b (1.9)	5.4b (3.1)	2.4[1]

[1] Thresholds reported orthonasally in water (Rychlik and others, 1998).
Means in a row followed by different letters are different ($p<0.05$).
Numbers in parentheses are standard deviations across samples

Table 12: Relative Abundance of compounds identified by SPME

Concentration (ppm)

Compound	Raw	BT	HTST	Evap	SMP
Undecanoic	0.04	0.48	0.03	1.09	0.91
Acid	(0.05)	(0.92)	(0.08)	(1.29)	(0.79)
Decanal	0.30	2.32	0.11	6.50	4.21
	(0.45)	(4.08)	(0.29)	(8.68)	(3.71)
Dodecanal	0.41	0.65	0.03	1.94	1.57
	(0.06)	(1.12)	(0.07)	(2.58)	(1.28)
Nonanal	0.18	0.92	0.05	5.81	1.52
	(0.32)	(1.65)	(0.13)	(7.99)	(2.31)
Hexanoic	0.03	0.40	ND	ND	0.55
Acid	(0.02)	(0.97)			(0.93)
Heptanal	ND	ND	ND	0.37	ND
				(0.52)	
Octanoic	47.8	374.2	18.4	28.7	706.0
Acid	(68.6)	(659.3)	(48.6)	(37.6)	(642.4)
Nonanoic	5.1	ND	ND	ND	ND
Acid	(11.39)				
n-Decanoic	0.16	1.23	0.06	3.53	2.29
Acid	(0.20)	(2.18)	(0.16)	(4.74)	(2.09)
Butanal	0.73	0.95	ND	0.07	0.86
	(1.62)	(2.24)		(0.09)	(1.67)
Dodecanoic	0.22	ND	0.01	0.05	ND
acid	(0.47)		(0.01)	(0.07)	
Hexanal	0.04	0.06	0.01	0.14	0.19
	(0.08)	(0.14)	(0.02)	(0.20)	(0.21)

ND = Not Detected

Numbers in parentheses are standard deviations across samples