Application of Sensory and Instrumental Volatile Analyses to Dairy Products

A.E. Croissant, D.M. Watson, and M.A. Drake*

Department of Food, Bioprocessing, and Nutrition Sciences, Southeast Dairy Foods Research Center, North Carolina State University, Raleigh, North Carolina 27695;
email: maryanne_drake@ncsu.edu

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Abstract

Comprehensive food flavor analysis requires a multidisciplinary approach. This article presents a comprehensive review of the relationship between sensory and instrumental analysis in the research of food flavor. Common practices for aroma flavor compound isolation, separation, and identification are discussed with strengths and weaknesses of the respective methodologies. A review of whey protein flavor research is presented to demonstrate the range of techniques available for the investigation of food flavors. These techniques are applicable to all food categories. The complexity introduced by food texture regarding flavor analysis is discussed using the attribute creaminess as an example.

FLAVOR IN FOOD

The sensory experience of a food (flavor and texture) is multimodal and encompasses psychological and physiological responses. In the most basic form, flavor represents perception of the basic tastes, aromas, and feeling factors of a product. Given that there are only five basic tastes and a limited number of trigeminal responses, most of what is considered flavor actually refers to aroma-active volatile compounds that are perceived ortho- or retronasally by the olfactory epithelium. Considerable effort has been recently made to relate taste and aroma perception, and to define flavor as a combination of sensory modalities. Auvray & Spence (2008) reviewed multimodal flavor analysis and argued that taste, aroma, feeling factors, the trigeminal system, and visual and auditory properties make up flavor perception, which defines flavor not by sensory modality but by perceptual modality brought together by the act of eating. However, because of the wide assortment of aromatic compounds and the hundreds of combinations of these compounds, a tremendous amount of work remains to be conducted to elucidate the role(s) of volatile compounds in foods. The current review focuses primarily on the analysis of volatile compound flavor contributions in foods. The majority of food flavor experience consists of aroma properties, but nonvolatile constituents should also be considered in the investigation of food flavor. Nonvolatiles and volatile compounds may present additive or reductive effects on perceived aroma and/or taste. Subthreshold concentrations of volatile compounds have been shown to amplify taste intensity and vice versa (Pfeiffer et al. 2005). Flavor perception is also a combination of physiological and memory responses. Previous consumer experience with aroma compounds has been demonstrated to play a role in flavor synergy (Nguyen et al. 2002, Pfeiffer et al. 2005, Stevenson et al. 1999). In addition to sensory methodology, psychophysical, neuroimaging, and neurophysiological studies contribute to the body of knowledge explaining flavor and flavor perception. An understanding of the chemical and physical factors responsible for the attributes of interest as well as panelist training can aid in reducing misinterpretation of results.

SENSORY TECHNIQUES AND APPLICATIONS

Sensory analysis was practiced in one form or another long before the application of quantitative sensory analysis techniques. When considering food systems, all consumers evoke their own form of sensory analysis, whether by the determination of foods with recognized terms or by their own nomenclature or perceived taste. Grading and judging established a preliminary methodology for quality evaluation (Bodyfelt et al. 1988). Such techniques are still widely employed throughout dairy, muscle, and produce food systems. However, these techniques are defect oriented and limited to the assessment of good or poor quality foods for consumption. When considering some food products, such as produce, companies may employ internal grading to separate premium stock for sale or as fresh versus stock destined for further processing. Often times, this grading is based on appearance and size, and may be subject to electronic monitoring and automation. Human judging and scoring protocols are subjective, nonlinear, and cannot be analyzed statistically or related to consumer likes and dislikes (Drake et al. 2001, Singh et al. 2003). Grading and judging are intended to be utilized as rapid-assessment tools and do not serve to capture the distinct flavor profiles of products or to quantitatively measure consumer acceptance.

Recently, descriptive sensory analysis has become the preferred method of analytical (objective) sensory evaluation (Drake 2007). These methods are intended to enable a group of individuals to perform as a single instrument in order to profile products on all sensory characteristics. This approach can be used to compare products, evaluate quality, and relate to instrumental or consumer

responses (Murray et al. 2001, Singh et al. 2003). Each method requires the recruiting and training of a descriptive panel as well as the development or adoption of a sensory language (Drake & Civille 2003). Training is focused on use of the language with specific definitions and references. Terms are not defined as levels of good or bad but by intensities and attributes.

There are several approaches to the training and maintenance of a descriptive analysis panel. The flavor profile method and the texture profile method utilize panelists to form a group consensus (Lawless & Heymann 1999). Quantitative descriptive analysis® (QDA) and the Spectrum™ method are probably the two best-known approaches to descriptive panel training. QDA®, developed by Stone et al. (1974), uses nontechnical terms. Training is generally product or category specific, and references are used only to solve problems with terms. The panel leader does not participate with the 8–12 panelists but facilitates commentary. Data from QDA® is both quantitative and qualitative, but scaling is generally product specific (i.e., attribute intensities are only relatable to other products within the same category or product) (Lawless & Heymann 1999). The Spectrum™ method, developed by Civille of Sensory Spectrum (New Providence, NJ) (Lawless & Heymann 1999), relies on multiproduct, specialized training with references. Attributes are measured on a universal intensity scale and are thus not product specific; references are constant regardless of the product being evaluated. Training on this method is more time consuming, but a single panel can then readily evaluate multiple products or product categories and intensities from different products, and categories can be directly related to each other (i.e., a three in fruity flavor in cheese is the same as a three in fruity flavor of meal replacement beverages, etc.)

Lexicon development includes the designation of terms and references to describe the flavor attributes of a product or commodity. Drake & Civille (2003) reviewed the process for lexicon development as well as its application to understanding flavor. A good flavor lexicon is based on a diversity of products representing the range of flavors of the food product. A broad set of terms is developed then reduced to eliminate redundancies. Equally important is the variety and selection of food and/or chemical references to anchor the lexicon terms. The ideal lexicon maintains multiple references for each term, and is descriptive and discriminating (Drake & Civille 2003). A complete flavor lexicon coupled with a well-trained descriptive panel serves as the basis for flavor research. **Table 1** presents a compilation of sensory languages used to describe whey proteins and whey powders.

Civille & Lyons (1996) compiled a standardized definition and reference book of flavor descriptors that can be used to describe any product(s) or category. The dairy lexicons are applicable across all dairy products with requirements for addition and removal of some terms. Lexicons have been developed for numerous products and commodities, including but not limited to wine, cheese, peanuts and tree nuts, olive oil, fermented dairy products, distilled beverages and beer, chicken and meats, chocolate, rice, vanilla, and fruits (Drake & Civille 2003). Often with much overlap, lexicons have been developed to describe foods within the same category, such as different types of cheese (hard, semihard, soft; young versus aged; and specific types, such as regional cheeses) and different varietals of wine. Using specific and widely available references enables wider adoption of a flavor language.

Another type of sensory testing that generates quantitative data is affective testing. These tests involve consumers rather than trained panelists and 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 (Lawless & Heymann 1999). Data obtained from affective testing can be related to descriptive and/or instrumental data by external preference mapping.

Table 1 Sensory language for whey protein powders (SWP, WPC34–80, WPI)[a]

Descriptor	Definition	Reference/preparation
Aroma intensity	The overall orthonasal aroma impact of the rehydrated sample	Evaluated as the lid is removed from the cupped sample[c]
Sweet aromatic	Sweet aroma associated with dairy products	Colby Jack cheese shreds, mild Cheddar cheese[b]; 20 ppm vanillin in milk[c]; vanilla cake mix[e]; Quaker oatmeal (50 g soaked in 500 mL water)[e]
Milkfat	Aromatics associated with fresh whole milk	Heavy cream, δ-dodecalactone, 40 ppm[b]
Pasta	Aroma associated with water after pasta has been boiled in it[c]	Boil pasta in water for 30 min[c]; 2,4-decadienal, 20 ppm on filter paper in sniff jar[e]
Doughy/fatty	Aromatic associated with canned biscuit dough[c]	canned biscuit dough[c]; 1 ppm (*Z*)-4-heptenal in water[c]; (*E,E*)-2,4-decadienal (2 ppb in skim milk)[h]
Metallic/ meat serum	Aromatics associated with metals or with juices of raw or rare beef[e]	Raw beef steak or ground beef or juices from seared beef steak[e]
Cardboard/ wet brown paper	Aroma associated with cardboard and brown paper[e]	Cardboard in water[b]; cardboard paper[c]; 2 cm × 2 cm pieces of brown paper bag boiled in water for 30 min[e]; pentanal, heptanal, nonanal, 1-octen-3-one, & dimethyl trisulfide[f]
Animal/ wet dog	Aroma associated with wet dog hair[c]	Dissolve 1 bag of gelatin (28 g) in two cups of distilled water[c]
Brothy	Aromatics associated with vegetable stock or boiled potatoes[c]	1 ppm methional or freshly sliced potatoes[c]; drained broth from canned white potatoes[e]
Cooked	Aromatic associated with cooked milk[c]	Heated skim milk to 85°C for 30 min[b]
Musty	Aromatics associated with old books, decaying wood, or closed air spaces	Potting soil[b]
Buttery	Aromatics associated with fresh butterfat and sweet cream	Mild Cheddar[b]; 30 ppm diacetyl in water[d]
Cereal/grain	Aromatics associated with cereals and grains[e]	Cheerios, 50 g in 200 mL water[e]
Fruity	Aromatics associated with different fruits, particularly pineapple[e]	Ethyl hexanoate, 20 ppm on filter paper in sniff jar[e]
Catty	Aromatics associated with tomcat urine[e]	[2]-mercapto-[2]methyl-pentan-[4]-one, 200 ppm on filter paper in sniff jar[e]
Soapy	Aroma associated with medium-chain fatty acids and soap[c]	1 ppm lauric acid or shaved bar soap[c]; decanoic acid[h]
Fecal/dirty	Aromatics associated with animal excrement[e]	Skatole or indole, 20 ppm on filter paper in sniff jar[e]
Yeasty	Aromatics associated with fermenting yeast[e]	Freeze dried yeast packet, 7 g in 500 mL water[e]
Malty	Sweet fermented aromatic associated with dried sprouted grains[e]	Grape Nuts cereal, 20 g in 500 mL water[e]
Cabbage	Sulfurous aromatic associated with cooked cruciferous vegetables	Dimethyl trisulfide, boiled fresh cut cabbage[g]
Raisin/spicy	Aromatics associated with stewed raisins[h]	Boil 50 g dark raisins in 500 mL water[h]
Opacity	Visual term referring to the degree of opacity of the rehydrated protein solution[e]	Water = 0, whole fat fluid milk = 11[e]
Viscosity	Attribute evaluated in the mouth, place product in mouth (approximately 1 tsp), evaluate the rate of flow across the tongue[i]	Water = 1, heavy cream = 3, sweetened condensed milk = 12[d]
Cucumber	Aroma associated with freshly sliced cucumber[c]	1 ppm (*E*)-2-nonenal or freshly sliced cucumbers[c]

(*Continued*)

Table 1 (*Continued*)

Descriptor	Definition	Reference/preparation
Salty	Basic taste elicted by salts[i]	NaCl 2%[i]
Sour	Basic taste elicted by acids[i]	Citric acid, 1%[i]
Sweet	Basic taste elicited by sugars[i]	5% sucrose solution[i]
Astringent	Chemical feeling factor characterized by a drying or puckering of the oral tissues[i]	Soak six black tea bags in 500 mL water for 10 min; alum, 1% in water[c]
Bitter	Basic taste elicited by various compounds including caffeine and quinine[i]	Caffeine, 0.5% in water[i]

[a]Adapted from Carunchia Whetstine et al. (2005) & Russell et al. (2006).
[b]Karagul-Yuceer et al. 2003.
[c]Carunchia Whetstine et al. 2005.
[d]Mortenson et al. 2008.
[e]Russell et al. 2006.
[f]Whitson et al. 2010.
[g]Wright et al. 2006.
[h]Wright et al. 2009.
[i]Universal references in Meilgaard et al. 2007.

INSTRUMENTAL EVALUATION OF FLAVOR

The human nose is a more powerful tool for aroma evaluation than any machine to date. Flavor is, after all, a sensory experience. However, to understand flavor, sensory analysis techniques must be paired with instrumental techniques to identify specific compounds responsible for taste and aroma in foods. An understanding of the food matrix and the identification of flavor compounds of interest are required to determine the proper methods of extraction (McGorrin 2007, Reineccius 2006b). Factors for consideration include the physical state/composition of the product, sample size and availability, time, compound volatilities and stabilities, and resources. Each method of extraction has strengths and weaknesses in terms of sensitivity, time and resource commitment, and materials safety. Headspace analysis and solvent extraction remain the primary methods for volatile compound extraction. Some methods for direct analysis have been developed. It is important to note that because each extraction technique selects for certain compound classes, the most beneficial approach to flavor chemistry of foods combines more than one technique for analysis.

Aroma Extraction and Isolation Techniques

Although direct gas chromatography (GC) analysis of foods is theoretically possible, the resulting data may be inaccurate for a number of reasons: thermal degradation of nonvolatiles may result in false GC peaks; damage to the column and decreased separation efficiency (by water or other constituents); and column and sample contamination (Reineccius 2003). For these and other reasons, some form of sample extraction is required for a thorough and accurate analysis. Extraction techniques allow the researcher to preferentially solvate a class of compounds of interest (e.g., aroma volatiles) in an extracting solvent and then remove those compounds from the rest of the food matrix. However, extraction also picks up some unwanted organic material, so extraction is often carried out in tandem with purification or distillation of the extract. Researchers must understand that each method selects for some aroma compounds over others, so analysis and conclusions drawn must consider this bias (Reineccius 2006a).

DSE: direct solvent extraction

GC-O: gas chromatography-olfactometry

V-SDE: vacuum simultaneous distillation extraction

A-SDE: atmospheric simultaneous distillation extraction

Various methods exist for extracting the volatile components of foods into a form more easily analyzed. These methods include steam distillation, static and dynamic headspace sampling (DHS), direct solvent extraction (DSE), and vacuum distillation. Each method has distinct advantages and disadvantages, and each of these factors must be considered when choosing a method of extraction. In flavor chemistry, for example, high temperature extraction methods (e.g., steam distillation) often lead to artifact formation and ultimately inaccurate data (Reineccius 2006a). In the case of flavor chemistry, liquid solvent extractions are preferred; however, not all solvents extract organic compounds equally, some solvents are much more efficient than others (Prososki et al. 2007), and not all solvents may be used with gas chromatography-olfactometry (GC-O). Highly volatile compounds may also be masked by the solvent in GC analysis, so for these compounds headspace methods are preferred. Despite the drawbacks of using solvent extraction to recover aroma volatiles (can be time consuming, generates waste, nondiscriminatory, requires post-extraction purification and concentration steps that may lead to loss of volatile components), solvent extraction remains a preferred method of isolating aroma volatiles from food matrices.

Solvent extraction. DSE is generally performed by agitating solvent with crushed or ground analyte. A centrifugation step is often required to fully separate the solvent and sample layers. Diethyl ether (DE) is a very commonly used solvent for volatile extraction (Alewijn et al. 2003), although it has been shown to be less effective than other solvents, especially at extracting highly polar compounds (Prososki et al. 2007). Researchers noted that DE recovered only 35 compounds from sweet whey powder (SWP), as compared to 37 and 42 compounds recovered using methyl formate and methylene chloride, respectively. The study furthermore observed that DE recovered only 25%–50%, on average, of the volatile compounds known to be present across all classes of common food compounds. In a separate study, Alewijn et al. (2003) commented that despite its toxicity, acetonitrile was a superior solvent for the extraction of cheese volatiles, due to its ability to dissolve nearly all lipid-derived compounds. Despite these findings, however, DE remains a commonly used solvent for the extraction of aroma volatiles from food and is cited for use in numerous dairy and food flavor research papers (Drake et al. 2010, Song & Cadwallader 2008). DE is used because it is relatively safe to handle (Heath & Reineccius 1986), easily disposed, inexpensive, and most aroma compounds are readily soluble in organic solvents. DE extracts are considered safe for GC-O sniffing, unlike acetonitrile, methylene chloride, and to a lesser extent, methyl formate extracts. Supercritical fluid extraction offers the advantage of an evaporative solvent in CO_2. However, application to food products is limited by the high cost of equipment and the nonpolar nature of CO_2 (Zhang & Li 2010).

Another common solvent-extract technique is simultaneous distillation extraction (SDE), which simultaneously distills and solvent-extracts a sample (Chaintreau 2001). Samples are prepared for SDE by making a homogenous mixture of the sample with water. The sample and solvent are contained in separate flasks. Both flasks are boiled, vapors mix together, condense, and are separated into respective flasks by density. The extracting solvent is funneled into the solvent flask, and the process is repeated indefinitely. Historically, limitations to SDE were the relatively small sample size and the potential for artifact formation inherent to any extraction process that utilizes heat to drive the extraction (Majcher & Jelen 2009). Small sample size and solvent volume may be advantageous, resulting in an extract that requires no concentration step for analysis. SDE under vacuum has been utilized to reduce heat-generated artifact formation. Vacuum conditions reduce heating requirements by decreasing the boiling point of solvents and compounds. Higher boiling point solvents are required in V-SDE compared to A-SDE in order to reduce ice formation by increasing the condenser temperature and to condense the solvent at or near room temperature (Maignial et al 1992).

Solvent extracts must be concentrated prior to GC injection, ideally without any loss of solvated organic material, while also removing coextracted nonvolatile material. Concentration under vacuum and distillation are two of the more common methods for concentrating solvent extracts. High vacuum transfer (HVT) is a vacuum concentration technique and relies on a large temperature gradient to vaporize the sample volatiles, forcing the volatile fraction through a carrier tube into a collection flask (Engel et al. 1999). Solvent-assisted flavor evaporation (SAFE) is a relatively modern method of separating volatile from nonvolatile food components, typically used in conjunction with solvent extraction. However, the SAFE method has also gained popularity due to the ability to isolate volatiles from numerous food matrices without solvent extraction (Engel et al. 1999). HVT has been used with both solvent-extracted samples and whole food samples, but drawbacks include requiring a large amount of time and space to operate, as well as limitations to the type of food material and solvent that can be utilized (Engel et al. 1999). SAFE offers several other advantages over HVT, as described by Engel et al. (1999). When using DE to extract Cheddar cheese, for example, the highly nonpolar ether also solvates the lipid fraction of the cheese; SAFE distills the ether extract phase from the lipid phase, yielding a flavor volatile extract free of nonvolatile material that can be further concentrated and analyzed.

SAFE: solvent-assisted flavor evaporation

Distillate may also be phase separated to aid in compound detection by decreasing the number of compounds in a given GC-FID (flame ionization detector)/GC-O/MS (mass spectrometry) injection. Acidic (AC) and neutral-basic (NB) fractions are common (Evans et al. 2009, 2010; Milo & Reineccius 1997; Suriyaphan et al. 2001; Whetstine et al. 2003, 2005). The purpose of separating a sample into multiple fractions is purely algebraic. Some of the compounds end up in the NB fraction, and the rest end up in the AC fraction, making identification easier by reducing problems of coelution or peak masking. Coelution is of particular interest to control, because two or more compounds may elute simultaneously, although it is possible that only one of the compounds will have aroma activity. GC-O analysis of fresh goat cheese samples (Whetstine et al. 2003) revealed 29 AC fraction compounds and 53 NB fraction compounds. Phase separation also aids in the identification of different compound classes. Free fatty acids, for example, will remain in the AC fraction, while many aldehydes, ketones, and esters remain in the NB fraction.

Headspace sampling techniques. Headspace sampling methods are broadly lumped into one of two categories: static or dynamic (Wampler 1997). Static methods sample the headspace of a known volume or mass of sample in a closed chamber after the sample has equilibrated with the headspace. Static headspace sampling is especially useful when the analytes of interest are low molecular weight and low boiling point compounds, which tend to elute early on a GC column and are consequently easily masked by a solvent peak (Wampler 1997). DHS methods, unlike static methods, do not allow the establishment of headspace equilibrium within the sample vessel. Volatiles are removed from the headspace by the flow of carrier gas and concentrated in a trap by adsorption or cold trapping. Lack of established equilibrium results in greater volatile transfer from the sample to the headspace. Carrier gas may also be bubbled through the sample in the case of liquid matrices. DHS methods may increase the sensitivity of GC analysis compared with static headspace methods by increasing the actual amount of analyte sampled from a sample matrix. However, dynamic methods typically involve much more complex instrumentation and a greater number of steps in the process compared with static headspace methods, thus introducing more potential for error and inconsistency (Wampler 1997).

Solid phase microextraction (SPME) began as a method for analyzing air quality and pesticide residues in water (Arthur et al. 1992, Pawliszyn & Arthur 1990) but has since been widely adapted to use in food and flavor chemistry research. Readers are encouraged to study Marsili (1997, 2002) for reviews on SPME application and methodology and Spietleun et al. (2010) for a more

HS-SPME: headspace solid phase microextraction

recent discussion on SPME fiber coatings. With headspace SPME (HS-SPME), a coated silica fiber is injected into the sample headspace and analytes are partitioned between the vapor phase, the sample matrix, and the polymer coating of the fiber (Quach et al. 1999). Direct immersion SPME, whereby the fiber is immersed in the sample, is a technique more associated with semi- and nonvolatile compounds than volatile flavor compounds (Kataoka et al. 2000). Immersion has also been shown effective for extraction of nonpolar and higher molecular weight analytes (Shirey 2000a,b). The mass transfer of volatiles from the headspace to the fiber is limited by the slower diffusion of analytes from the matrix. This imbalance can be overcome by changes to the headspace volume, pH, sample size, agitation, and temperature, as well as the addition and concentration of salt (Lee et al. 2003; Quach et al. 1999; Werkhoff et al. 2002; D.M. Watson, unpublished data). As different matrices will affect the partitioning of analytes, optimization of SPME parameters with each product type is vital to ensure recovery of intended compounds.

The SPME technique is applicable to gas and liquid chromatography analysis. An SPME unit consists of a fiber coated with a stationary phase, often dimethylsiloxane (nonpolar) or polyacrylate or divinylbenzene/Carboxen/poly(dimethylsiloxane) (polar) (Grosch 2007). The fiber coating is one of the most important factors for consideration, affecting compound selectivity, precision, and a representative volatile profile (Werkhoff et al. 2002). Some advantages of SPME are increased consistency and rapid measurement stemming from automation (Frank et al. 2004), equivalent methods for different phases, and heating of the sample is not required (dynamic SPME utilizes heat), reducing or eliminating the formation of artifacts (Harmon 2002). The greatest advantage of the SPME method over solvent extraction is the absence of solvents employed in the extraction. Lack of solvent and solvent disposal requirements makes SPME a less-costly volatile compound extraction method with reduced sample preparation time (Marshall 2003). Drawbacks to using SPME include linear range of the SPME fiber, competition between compounds for binding sites on the fiber can introduce inconsistency and error as well poor recovery of trace compounds, and reproducibility across multiple fibers with the same coating is questionable (e.g., in the event of fiber breakage) (Reineccius 2003).

Frank et al. (2004) utilized SPME-gas chromatography-mass spectrometry (GC-MS) and SPME-GC-O to analyze and identify aroma compounds in three types of cheeses, including Cheddar. Their basis for selecting SPME over solvent extraction methods was that important aroma compounds, especially those containing sulfur, were often present in foods below the limit of detection using conventional methods. A separate study, conducted by Bellesia et al. (2003), found that the recovery of volatiles by SPME was very comparable to the recovery of volatiles from the same samples by purge and trap headspace methodology. This finding is significant, as a prime factor slowing the widespread adoption of SPME was previous reliance on approved methods (Zhang & Yang 1994), such as purge and trap.

Gas chromatography. GC is used in flavor research to separate and identify unknown volatile compounds in food. GC analysis separates volatile compounds based primarily on polarity and volatility. Historically, columns were packed with a coated, granular material: The coating polarity was chosen to match the chemical properties of the compounds to be separated. As GC theory advanced, materials containing polar and nonpolar functional groups were introduced that allow for production of columns with a range of polarity (Reineccius 2003).

Careful column selection may make compound identification easier. Two commonly used columns are the DB-WAX (polar) and DB-5 (nonpolar) columns. Highly polar compounds, like free fatty acids, are well separated by polar columns (DB-WAX; also referred to as DB-FFAP). For this reason, polar columns are a good selection for any sample that is thought to contain a large constituency of acids (Reineccius 2003). Nonpolar compounds (e.g., lactones, esters, ketones, and

aldehydes) are better separated by more nonpolar columns (e.g., DB-5MS). Because most foods contain some quantity of both polar and nonpolar compounds, samples are typically analyzed on at least one type of each column.

An additional driver for multiple column analysis is to increase the certainty of identification of an unknown compound. Although coelution of compounds may be common on a single column, the likelihood that the same compounds will coelute across multiple columns is exponentially less. Therefore, analyzing a sample with multiple columns helps nullify the effects of coelution and to positively identify unknown compounds. Compounds eluting from a GC column have a specific retention time (RT) for a given column (in reality, on a given column a compound will elute within a given range of RTs). Retention indices (RIs) are more stable and comparable across time, machine, and location than RT values. The RI, or Kováts index, normalizes retention times by comparison against an *n*-alkane series (van den Dool & Kratz 1963).

Compounds eluting from the column are analyzed at the detector. Although several types of detectors are available for use with GC, FID, the human nose (GC-O), and MS provide the most diverse detection capabilities and therefore provide a better overall picture of food flavor (Reineccius 2003). FIDs respond best to organic compounds (C–C or C–H bonds), have a good linear response and high dependability (Reineccius 2003), and are the most common GC detectors (Reineccius 2006a).

Mass spectrometry. MS first ionizes a molecule and then resolves the ionized molecule based on mass-to-charge (m/z) ratios in an electrostatic field (Smith & Thakur 2003). Detection and identification of analyte are based on the unique, predictable spectra created by the ionization of each compound. Mass spectra are compared to a computer database as well as spectra and retention index of an authentic standard for identification. Spectral retention indices are compared with GC-O or GC-FID retention indices for secondary affirmation that a compound is present in a sample. Researchers may also start with aroma properties and work their way back to confirmation by comparison to standards. MS detector function and types have been discussed (McMaster & McMaster 1998, Smith & Thakur 2003).

Quadrupole mass analyzers are the most common today, although ion trap analyzers are also commonly used (Smith & Thakur 2003). Sensitivity of both quadrupole and ion trap analyzers can be increased by selective ion monitoring (SIM) in which certain m/z ratios are detected and remaining ions are discarded (de Hoffmann & Stroobant 2002). This approach is useful when spectral information for compounds of interest is already known, providing greater resolution with less spectral information (Harmon 2002). Time of flight (TOF) analyzers contribute the ability to collect data in much shorter time frames at higher resolution compared with traditional scanning MS analyzers (Holland & Gardner 2002). Until recently, their contribution to flavor research was generally reserved for the analysis of large proteins, peptides, and polynucleotide molecules (McMaster & McMaster 1998). Two-dimensional (2D) GC × GC-TOFMS has been increasingly used in the investigation of volatile compounds and contaminants in food. Enhanced peak separation of GC × GC analysis compared with single GC allows for better characterization of compounds from complex matrices. 2D GC is generally paired with SPME and generally utilizes a polar and nonpolar column for each dimension. Application and advantages of GC-TOFMS in food analysis have been reviewed (Cajka & Hajslova 2006, Cajka et al. 2009, Williamson & Bartlett 2007).

High performance liquid chromatography (HPLC) and ion chromatography are widely used for the analysis of nonvolatile food components, i.e., taste components. Methods for analysis of taste compounds are generally well established. Additional techniques such as matrix-assisted laser desorption/ionization (MALDI) and electrospray ionization have seen increasing use in the

investigation of nonvolatile food components that may influence flavor and flavor perception (Turnipseed 2006).

Gas chromatography-olfactometry. A final detector type commonly used with GC is neither FID nor MS: It is the human nose. GC-O is a method of evaluating odor-active compounds. This technique provides additional confirmation of compound identity and aids in the determination of the aroma quality of individual compounds and their significance to the flavor of a food. The procedure for GC-O combines GC 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, while a chromatogram is established by the traditional detector, usually an FID. An additional injection is usually made separately on a GC-MS for comparison; GC effluent may also be split between the nose and an MS detector to obtain concurrent information and simplify analysis (Reineccius 2006a).

As with any analytical technique or methodology, GC-O has its advantages and its limitations. GC-O has high differentiating power, meaning the sniffer (if properly trained) is capable of detecting very subtle differences in column effluents. GC-O also affords the possibility to detect the aroma of compounds at a concentration below instrumental detection (Mistry et al. 1997). However, GC-O also has the distinct disadvantage of utilizing human responses to chemical stimuli, which are known to change over time, even with careful training (Mistry et al. 1997, Friedrich and Acree 2000). Human subjects must be carefully selected and calibrated by frequent lexicon and descriptive training. In addition, each sample should be sniffed on at least two different columns to account for poorly resolved compounds (Mistry et al. 1997). Because GC-O utilizes human response, this method is subject to inconsistencies. As demonstrated in **Table 2**, two sniffers may perceive the same compound differently. Sniffer training and experience with products of interest may help to alleviate such differences in recognition. GC-O should be used as a preliminary step in the investigation of aroma-active compounds. Compounds of interest are investigated further for positive identification, including comparison with chemical standards and sensory recombination techniques.

Table 2 Sensory descriptors of select compounds identified by two panelists in WPC80 by solvent extraction and gas chromatography-olfactometry[a]

RI (DB-5)	Compound	Sniffer 1 (female)	Sniffer 2 (female)
682	acetic acid	sour	vinegar
815	isopropyl butanoate	fruity/catty	tart fruit
862	butanoic acid	skunk(sulfur)/cheesy	sour milk
909	*Z*-4-heptanal	green/glue	plastic/waxy
947	2-acetyl-1-pyrroline	stale popcorn	brothy/meaty
965	dimethyl trisulfide	garlic/pungent	waxy/rancid/sulfur
1028	octanal	citrus	oranges/vitamin
1159	benzyl acetate	garlic	cabbage/potato
1188	(*E*)-2-nonenal	sweet/carpet/cucumber	carpet lingers
1202	dimethylsulfoxide	rubber	garlic
1292	phenylethyl acetate	cucumber/rosy	floral/plastic
1511	tridecanal	mothball	cinnamon

[a]Adapted from Evans et al. 2010, unpublished information.

Table 3 Summary of factors considered for different gas chromatography-olfactometry methods

	Dilution analysis?	Aroma	Intensity	Duration	# Responses
AEDA	Yes	+	−	−	−
CharmAnalysis	Yes	+	−	+	+
Osme	No	+	+	+	−
NIF/SNIF	No	+	−	+	+
Post Peak	No	+	+	−	−

(+) = positive response, (−) = negative response.

Just as the term extraction is limiting in its nonspecificity, the term GC-O is also limiting. GC-O describes a class of analysis methods, classified as detection frequency, dilution to threshold, or direct intensity (Delahunty et al. 2006). Those methods of olfactometry that are most common are postpeak sniffing, combined hedonic aroma response measurements (CharmAnalysis), Osme, aroma extract dilution analysis (AEDA), and nasal impact frequency/surface nasal impact frequency (NIF/SNIF). A summary of variables considered in GC-O methodology is presented in **Table 3**.

Postpeak sniffing is simply any method of olfactometry that splits the column effluent by some ratio (1:1 is most common) such that one direction of the split effluent is detected by FID or another detector, and the other half of the split effluent is evaluated by a human nose detector. CharmAnalysis is a dilution method developed at Cornell University to determine odor activity of compounds in a sample (Acree et al. 1984). CharmAnalysis is a combined hedonic aroma response measurement (Mistry et al. 1997, van Ruth 2001) based on a stepwise dilution of a sample, with randomized order of dilution presentation. This method notes both the sensory perception and intensity of an odor, as well as the duration of the perception. Charm values are calculated based on the number of panelists who perceived a particular odor and the dilution factor of the sample (Mistry et al. 1997, van Ruth 2001). An additional GC-O method is NIF/SNIF, a response frequency method. NIF/SNIF determines values based on the number of panelists who detect a certain aroma in an extract (Pollien et al. 1997).

AEDA, like CharmAnalysis, utilizes stepwise dilution of a sample extract to determine the most odor-active compounds in a sample. Unlike CharmAnalysis, AEDA does not take into account the time intensity of a particular odor or the number of coincident responses to an odor (van Ruth 2001). AEDA dilutions are carried out until no further odor is perceived. AEDA analysis assigns a flavor dilution (FD) value to a compound, which is the highest dilution at which a compound can still be detected by sniffing (Grosch 1993).

Osme was developed by McDaniel's group at Oregon State University for the aroma profile analysis of wine, hop oils, and beer (Miranda-Lopez et al. 1992). Osme, unlike AEDA and CharmAnalysis, is a nonserial dilution evaluation of aroma extracts and does not make any effort to determine odor activity values (OAVs) or odor importance to a sample. Osme is performed by four trained panelists using a 16-point scale (0 = none, 7 = moderate, 15 = extreme) to rank perceived odor intensity and the duration of the perceived odor (Miranda-Lopez et al. 1992, Mistry et al. 1997). An Osmegram is generated from the combined time-intensity averages for odors perceived by at least three of the four trained panelists.

Proponents of Osme support its use because it measures the odor intensity based on modern psychophysical theories (van Ruth 2001). AEDA and Charm have their limitations. They both assign an odor value to compounds and assume a linear relationship between compound concentration in a sample and its perceived intensity; both Fechner's and Steven's law disprove this assumption (van Ruth 2001). A second assumption inherent to AEDA and Charm that has

AEDA: aroma extract dilution analysis

received considerable discredit is that the same linear relationship (i.e., all slopes are the same) exists between all compounds and their perceived intensities. Many researchers have noted different linear relationships between concentration and perceived intensity for various compounds. Despite these criticisms and drawbacks, AEDA and Charm continue to be used for many aroma characterization and evaluation methods.

The electronic nose. The electronic nose (e-nose) or sensor array system was developed to mimic the discriminatory power of the mammalian olfactory system (Persaud & Dodd 1982). As is the case for all instrumental analysis, volatile and nonvolatile compounds are detected with e-nose systems, meaning that volatile compounds are detected by the machine. The presence of a volatile compound and its particular concentration in the food matrix do not necessarily relate to their role in flavor, as compounds may be present below sensory threshold and have no aroma activity and play little or no role in flavor. The application of e-nose systems to dairy products or any food product is thus limited by an understanding of the matrix, the compounds of interest, and their role in flavor (Ampuero & Bosset 2003). Dairy products have been analyzed by varied detector configurations but on a limited scope. E-nose holds suitable application for raw materials analysis and quality testing based on a strict set of parameters but does not hold as much power as human sensory analysis in terms of range of applicability to food products. As stated by Wilson & Baietto (2009) in a review of recent advances in e-nose technology, "A universal electronic nose capable of identifying or discriminating any gas sample type with high efficiency and for all possible applications has not as yet been built."

The application of the e-nose in food and beverage research has been focused on ingredient quality and origin, monitoring of manufacturing specifications, shelf life, and spoilage (Rock et al. 2008). Although numerous studies have been performed to assess the ability of the e-nose to differentiate food products, success is generally dependent on comprehensive descriptive sensory and/or instrumental analysis beforehand. For instance, Jonsdottir et al. (2004) related sensory attributes to GC-O and GC-MS to identify characteristic compounds of ripened roe. Sensory, GC, and e-nose data correlated well on the selected compounds of importance, but the e-nose by itself cannot determine quality unless a dataset is available for comparison. Given that dataset, any additional variation or off-flavor would require recalibration in order to differentiate samples in a meaningful manner.

LINKING SENSORY AND INSTRUMENTAL ANALYSIS

Identification of Flavor Attributes

Although 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 specific flavors in food (Drake & Civille 2003). This goal is challenging because instruments may detect many compounds that are present at levels below the human threshold. Likewise, there are flavor active components that contribute to food flavor at concentrations below instrumental detection. Concurrently, compounds may exhibit distinct aroma and taste properties in the food matrix compared with isolated analysis and influence flavor at concentrations below human threshold. Differentiating the small number of aroma compounds that are present in foods above the human threshold from those that are not becomes challenging when there are so many volatile compounds in the product (Drake & Civille 2003). One such method that assists in linking instrumental data to the sensory perception of flavor is GC-O.

As a basic review of flavor research techniques, descriptive sensory analysis describes the flavor of a food product, MS techniques provide information on volatile (and semivolatile to nonvolatile) compounds, and GC-O techniques provide information on the aroma activity of volatile compounds. By utilization of these processes, compounds can be identified as important aroma impact compounds in a food product. A compound identified with high odor activity and high concentrations within a food product is not enough to conclusively state its importance to aroma. OAV, threshold values, and model systems play an important role in flavor perception by way of investigation of the effect of the food matrix.

Odor activity values are calculated as the ratio of the concentration of a compound in food to the perception threshold of that compound in air, water, or other specified matrix (Audouin et al. 2001, Karagül-Yüceer et al. 2004). Therefore, OAV values less than one are theoretically not detectable by a human. A high OAV suggests a high degree of contribution to a food's overall aroma by a given compound. Threshold information has been published for numerous compounds (Burdock 2010) in varied matrices (van Gemert 2003a,b). However, dairy systems contribute a complex matrix that cannot be substituted by simple water and oil systems. Threshold values of flavor compounds should be determined in a matrix comparable to the food product of interest. Upon identification of important flavor compounds, descriptive analysis of model systems is employed as a tool to evaluate the contribution of individual compounds to aroma. Models are compared to the real food product for similarity. In the case of off-flavor characterization, this process is straightforward. Addition of the suspect compound to the food matrix free of the off flavor should recreate the off flavor (Wright et al. 2006). In the case of flavor characterization (e.g., strawberry flavor), this process is more challenging. Mixtures with individual compounds added (model addition or $N + 1$) or removed from the complete model (model subtraction, omission studies or $N - 1$) are utilized to compare contributions of each compound to the overall aroma (Buettner & Schieberle 2001, Karagul Yuceer et al. 2004). This process is not exact because typically only the primary (high aroma impact) compounds are selected for model studies (owing to their expense and time consumption), and volatile compounds at low or subthreshold concentrations as well as nonvolatile compounds and the food structure itself can influence the flavor perception of a product.

Analysis of Flavor in Liquid Whey and Dried Whey Powders: From Processing Through Storage

In recent years, the popularity and value of whey protein products have grown. In response to a growing body of knowledge of functionality and flavor, in addition to advances in separation technologies, whey protein ingredient use and whey protein products on the market have increased tremendously. Competition and the requirement for bland-flavored whey protein powders continue to drive innovation and research of a cheese-production byproduct of little value and quality mere decades ago. As a food category, whey protein powders are manufactured in a diverse manner. Understanding the effects of processing and storage on flavor has been undertaken by researchers deploying numerous technologies.

Although whey protein is defined as and expected to be bland and similar in flavor to fresh fluid whey or milk, thereby suitable for a wide range of food applications, the reality is that whey protein flavor variability exists between and within manufacturing facilities (Carunchia Whetstine et al. 2005, Wright et al. 2009). In order to evaluate differences in flavor profiles, numerous steps are required. Following the establishment of a lexicon, baseline sensory and instrumental information are required. Compositional values and range of flavors determine acceptability within the product category. Once the basic flavor profile has been established, deviations from the standard may be investigated. The investigation of the flavor chemistry and sensory properties of liquid whey

and whey protein powders is presented to demonstrate the range of techniques available to researchers investigating food flavors. Combining sensory science with instrumental tools provides the researcher opportunities to identify sources of variation and off-flavors between products. A summary of the techniques applied and conclusions of respective studies is presented in **Table 4**.

WPC: whey protein concentrate

WPI: whey protein isolate

Flavor is influenced along the entire manufacturing scheme of food products from the starting material through storage and packaging. Whey protein flavor chemistry and off-flavor assessment have been studied for decades. However, until recently, research failed to link the two by quantitative sensory assessment and instrumental analysis. Carunchia Whetstine et al. (2005) investigated the flavor properties of whey protein concentrate (WPC) and whey protein isolate (WPI) from different U.S. manufacturers by relating descriptive sensory and instrumental analyses. A lexicon was adapted from dried dairy ingredients. The terms applied in this lexicon are contained within **Table 1**. Similar to other dried dairy proteins, flavors were differentiated as dairy and nondairy flavors. Both WPC and WPI exhibited a range of undesirable flavors. Researchers employed solvent extraction followed by HVT to isolate aroma compounds. Compounds were then analyzed by GC-MS as well as GC-O. Representative samples were further analyzed by AEDA. Baseline volatile compound profiles of whey protein were established with lipid oxidation, protein degradation, and heat-generated compounds of primary concern. These findings are in agreement with Mahajan et al. (2004), who determined important aroma compounds in whey powder to be derived from the milk and starter culture as well as processing. Differences noted in ratios of aroma-active compounds in WPCs and WPIs to whey powders were attributed to the compositional differences between the product classifications (e.g., lower protein/higher lactose versus higher protein/lower lactose).

The conversion of liquid whey into dried whey protein involves numerous processing steps, providing the source of a wide range of variation between manufacturers and the creation or loss of aroma compounds. Storage of liquid whey is common in commercial manufacturing of whey powders. Tomaino et al. (2004) used descriptive sensory analysis, SPME GC-FID free fatty acid analysis, and DHS GC-MS to determine that extended storage of liquid Cheddar whey resulted in increased levels of common lipid oxidation products, decreased free fatty acids, and the development of cardboard flavor. However, the storage time was much longer than current manufacturing practices. Researchers also determined that different starter cultures affect the oxidative stability of liquid whey and that oxidation begins during cheese production based on the presence of oxidation compounds and cardboard flavor in fresh whey.

Carunchia Whetstine et al. (2003) investigated the impact of 23 commercially produced liquid whey from different mesophilic start cultures using descriptive sensory analysis, SPME GC-FID free fatty acid analysis, and DHS GC-MS. All three methods differentiated Cheddar liquid whey samples. Liaw (2009) also investigated the role of starter culture on the oxidative stability of fresh and stored liquid whey. Sensory and instrumental analysis differentiated Mozzarella from Cheddar cheese as well as fresh versus stored liquid whey. Descriptive sensory analysis and HS-SPME were performed on fresh liquid whey and liquid whey stored at 4°C for three days.

In the processing of whey protein, it is quite common for liquid whey and concentrated whey, known as retentate, to be stored either for production efficiency reasons or for transport to another facility for further processing. The impact of liquid whey storage on flavor has been investigated (Tomaino et al. 2004, Liaw 2009). Both types of cheese whey resulted in increased cardboard aroma and lipid oxidation products with storage. However, Mozzarella cheese whey exhibited greater oxidative stability compared to Cheddar cheese whey. A maximum retentate storage time of 12 h has been suggested based on the results of a combination of descriptive sensory analysis, HS-SPME GC-MS, DSE SAFE GC-MS, and DSE SAFE GC-O and AEDA (Whitson 2010). This study analyzed Cheddar and Mozzarella WPC manufactured from retentate stored up to 48 h, an

Table 4 Summary of significant research studies of liquid whey and whey protein powders

Source	Sensory methods	Instrumental methods	Products analyzed	Research significance
Carunchia Whetstine et al. 2003	DSA	DHS GC-MS, SPME GC-FID FFA	Cheddar liquid whey	Milk source, Cheddar starter cultures, and processing parameters impact the flavor & aroma volatile compound profiles of commercially produced liquid whey Liquid whey flavor is variable between and within manufacturing facilities
Karagul-Yuceer et al. 2003	DSA	DSE HVT, GC-MS; DSE HVT, GC-O, AEDA	Cheddar liquid whey	Liquid whey flavor is impacted by starter culture, milk source, and processing Liquid whey flavor is variable between and within manufacturing facilities
Carunchia Whetstine et al. 2005	DSA	DSE HVT, GC-MS; DSE HVT, GC-O	Cheddar WPC80; Cheddar WPI; Mozzarella WPC80	Established a baseline flavor and aroma volatile compound profile for WPC80 and WPI Compounds of interest are products of lipid oxidation, protein degradation, and heat generated
Mahajan et al. 2004		DSE SAFE, GC-MS; DSE SAFE, GC-O	SWP	Sources of aroma compounds of interest in SWP can be from milk, cheese type, lipid oxidation, caramelization, and Maillard browning
Tomaino et al. 2004	DSA	SPME, GC-FID; Purge & trap GC-MS	Cheddar liquid whey	Oxidation reactions begin during cheesemake Different starter cultures affect the oxidative stability of liquid whey produced on a pilot scale Liquid whey storage results in increases in lipid oxidation, cardboard flavors, and loss of fresh flavors
Campbell et al. 2010	DSA	HS-SPME, GC-MS	Cheddar liquid whey; Mozzarella liquid whey	Cheddar liquid whey less oxidative stability compared to Mozzarella liquid whey Both Cheddar and Mozzarella liquid whey decrease dairy flavors and increase nondairy flavors with storage
I.W. Liaw, unpublished data	DSA	HS-SPME, GC-MS; DSE SAFE, GC-MS; DSE SAFE, GC-O, AEDA	Cheddar liquid whey; Mozzarella liquid whey	Both Cheddar and Mozzarella liquid whey increase in cardboard flavor and lipid oxidation products with storage Mozzarella liquid whey has greater oxidative stability compared with Cheddar liquid whey
M.E. Whitson, unpublished data	DSA	HS-SPME, GC-MS; DSE SAFE, GC-MS; DSE SAFE, GC-O, AEDA	Mozzarella WPC80 retentate; Cheddar WPI retentate; Cheddar WPI; Mozzarella WPC80	Lipid oxidation and protein degradation products are the main aroma compounds of interest in WPC80 and WPI Longer retentate storage time corresponds to increased lipid oxidation products, cardboard and serum flavors, and aroma intensity Maximum suggested retentate storage time prior to spray drying is 12 h to maximize shelf life
Mortenson et al. 2008	DSA	DSE SAFE, GC-MS DSE SAFE, GC-O	Cheddar WPC34, WPI; Mozzarella WPC34; WPI; agglom. Mozzarella WPC34, WPI	Benzoyl peroxide bleaching, agglomeration, and filtration method had no significant effect on flavor of WPC34 and WPI

(*Continued*)

Table 4 (*Continued*)

Source	Sensory methods	Instrumental methods	Products analyzed	Research significance
Javidipour & Qian 2008		HS-SPME, GC-MS	Cheddar & Mozzarella WPC80; agglom. Cheddar & Mozzarella WPC80	Lipid oxidation compounds increased with storage at elevated temperatures Instantized WPC80 had a higher off-flavor formation compared with nonagglomerated WPC80 Argon flushing decreased the formation of volatile compounds
Liaw et al. 2010	DSA	HS-SPME, GC-MS	Cheddar liquid whey; Mozzarella liquid whey; Cheddar WPC66;	Mozzarella liquid whey greater oxidative stability than Cheddar liquid whey during storage WPH and ascorbic acid as antioxidant treatments reduce cardboard flavor and lipid oxidation products WPH is more effective antioxidant compared to ascorbic acid but imparts potato/brothy flavor to WPC
Croissant et al. 2009	DSA	HS-SPME, GC-MS	Cheddar liquid whey; Cheddar WPC70	Liquid whey flavor is representative of WPC flavor; treatment effects can be measured in liquid whey, eliminating need to produce WPC for analysis Bleaching increases cardboard flavor and lipid oxidation products in liquid whey and WPC With equivalent bleaching efficacy, hydrogen peroxide bleaching produces less desirable WPC compared with benzoyl peroxide
Wright et al. 2009	DSA; Ingredient application; Consumer acceptance	HS-SPME, GC-MS	agglom. Cheddar WPC80, WPI; instant Cheddar WPC80, WPI; instant Mozzarella WPC80; nonagglom. Cheddar WPC80, WPI; nonagglom. Mozzarella WPC80	Whey powders developed cucumber, raisin, fatty, and brothy flavors with storage; cardboard flavor increased and sweet aromatic flavor decreased with storage Lipid oxidation volatile aroma products and flavors increased with storage of all samples, but to a greater extent in agglomerated and instantized whey powders Descriptive sensory with consumer acceptance suggests the shelf life of agglom. whey powders is 8–12 months, nonagglom. powder shelf life is 12–15 months
Whitson et al. 2010	DSA, n-1 model systems	HS-SPME, GC-MS	Cheddar WPI; cardboard	Cardboard flavor is of primary concern to whey protein products Aroma volatile compounds and concentrations responsible for cardboard flavor were determined using model system WPI Although hexanal is a primary indicator of lipid oxidation, it does not contribute to cardboard flavor in whey protein products
Wright et al. 2006	DSA, threshold, OAV, model systems	HS-SPME, GC-MS DSE SAFE, GC-O, AEDA	Cheddar WPI; Cabbage off-flavor Cheddar WPI	DMTS is a common volatile compound found in WPI Above threshold level, DMTS contributes cabbage flavor in WPI

Abbreviations: AEDA, aroma extract dilution analysis; agglom., agglomerated; DMTS, dimethyl trisulfide; DS, descriptive sensory analysis; DSE, direct solvent extraction; FID, flame ionization detector; FFA, free fatty acids; GC-MS, gas chromatography-mass spectrometry; GC-O, gas chromatography-olfactometry; HS-SPME, headspace solid phase microextraction; HVT, high vacuum transfer; nonagglom., nonagglomerated; OAV, odor activity values; SWP, sweet whey powder; WPC, whey protein concentrate, WPI, whey protein isolate.

industrially relevant holding timeframe. Retentate was spray dried at 0, 6, 12, 24, and 48 h intervals. WPCs were stored at room temperature and analyzed at intervals over 12 months. Sensory analysis of retentate and powders revealed decreased desirable dairy flavors and increased undesirable nondairy flavors with increasing retentate storage time. This trend was maintained in WPC and WPI throughout 12 months of storage. Although storage of all powders resulted in increasing undesirable flavors, shorter retentate storage time resulted in decreased undesirable flavors.

Given that lipid oxidation products are believed to be the most important compounds when considering whey protein, methods for control of oxidation are vital to the continued improvement of flavor. The application of antioxidants to liquid whey was investigated by Liaw et al. (2010) to determine their impact on the flavor of WPC. Ascorbic acid, nitrogen blanketing, and whey protein hydrolysate (WPH) were added separately to Cheddar and Mozzarella liquid whey following pasteurization and fat separation. Liquid whey and WPC were analyzed by descriptive sensory analysis and SPME GC-MS. Results were consistent with previous studies in that Mozzarella liquid whey was more stable in storage than Cheddar whey. Cheddar WPC was produced with a control, WPH, and ascorbic acid treatments. Both antioxidant treatments yielded lower cardboard intensities and lower relative abundance of lipid oxidation products in WPC compared with the control. However, ascorbic acid was a less effective antioxidant than WPH. The addition of WPH resulted in a potato/brothy aroma, an undesirable flavor in whey that is commonly associated with WPH.

An additional source of liquid whey oxidation is the bleaching step. Bleaching of liquid whey and concentrated liquid whey retentate is required for the production of yellow Cheddar cheese and resulting uncolored WPC and WPI. Cheddar cheese whey serves as the major source of liquid whey in the United States, and colored Cheddar is the primary type manufactured. Currently, peroxides (hydrogen and benzoyl) are utilized in the bleaching step with great manufacturer variability in bleaching conditions, including time, temperature, concentration, and total solids at time of bleaching (Kang et al. 2010). Croissant et al. (2009) performed descriptive sensory analysis and SPME GC-MS to quantify the impact of the two common, approved oxidative bleaching agents on WPC flavor. Both hydrogen peroxide (HP) and benzoyl peroxide (BP) treatments resulted in objectionable flavor compared with the unbleached control. With the same bleaching efficacy, HP was more detrimental to whey flavor than BP. Contrary to the findings of Croissant et al. (2009), Mortenson et al. (2008) concluded that bleaching did not significantly affect the flavor of WPC34 and WPI. However, different whey streams with different processing conditions, storage times, and starter cultures obtained from different manufacturers were compared. Lack of control of processing conditions beyond bleaching introduced variability, making a true comparison difficult in this instance.

Food ingredients may undergo postprocessing treatments in order to increase value by providing additional functional properties. Whey protein powders are often subjected to agglomeration—the addition of steam, whey, or lecithin—to aid in dispersion. Javidipour & Qian (2008) investigated selected compound formation by HS-SPME GC-MS in agglomerated and nonagglomerated WPC80 over 15 weeks of storage at elevated temperatures. Generally speaking, lipid oxidation products increased with temperature and storage time. Agglomerated WPC80 had higher off-flavor formation than nonagglomerated. Argon flushing reduced off-flavor formation with storage. Although researchers did not apply sensory analysis to their products, compounds were selected based on previous research combining instrumental and sensory analysis. Wright et al. (2009) identified the agglomeration step as a source of reduced storage stability through the application of sensory and instrumental analysis. The shelf life of agglomerated and nonagglomerated WPC80 and WPI was investigated by combining descriptive sensory analysis, SPME GC-MS, and consumer acceptance testing. Descriptive sensory analysis and instrumental analysis were performed at intervals throughout storage. Flavor carry-through in beverages was

investigated to determine the consumer acceptance of flavored protein beverages made with fresh or stored whey proteins. Although numerous variables may be investigated within a given research project, prudence dictates that representative samples are chosen for ingredient application and subsequent consumer testing. Samples were chosen based on the data compiled by the trained sensory panel and volatile compound profiles. Shelf life was determined based on the consumer acceptance scores of flavored protein beverages made from fresh and stored WPC80 and WPI.

Cardboard flavor has been observed in many dried dairy products, specifically whey protein (Carunchia Whetstine et al. 2005, Evans et al. 2009, Russell et al. 2006, Wright et al. 2006). Cardboard flavor is commonly associated with whey proteins and has a negative effect on consumer liking in ingredient applications (Wright et al. 2009). Whitson et al. (2010) used a combination of sensory and analytical methods to determine the chemical compounds responsible for cardboard flavor in whey protein. The identification of specific lipid oxidation products as the source of cardboard flavor will aid in the ongoing effort to pinpoint the major factors associated with improving whey flavor by focusing instrumental detection methods. Sensory analysis was required to identify and compare representative WPI cardboard samples. Instrumental analysis was required to correlate volatile compounds with aroma. Numerous compounds have been detected in whey protein products (**Table 5**); determining which aroma compounds are of interest to a specific flavor requires preliminary investigation. Researchers evaluated samples of paper and cardboard soaked in water to identify the type of cardboard that represented the cardboard aroma found in whey protein. A WPI sample with high cardboard flavor was used as comparison for all testing. A stepwise approach to identify important aroma compounds is required. Often, one compound is not solely responsible for a flavor but rather a combination of compounds in varying concentrations (Carunchia Whetstine et al. 2006, Whetstine et al. 2005). Compounds were evaluated individually and in combination. Each compound was analyzed across a concentration range to account for changes in aroma (Avsar et al. 2004, Drake & Civille 2003, Karagul-Yuceer et al. 2004). Omission or subtraction models ($N - 1$) were used to identify the compounds that contributed most to cardboard aroma.

Through a combination of instrumental and sensory techniques applied to whey protein, researchers have developed a sensory language, identified sources of off-flavors, designated processing steps important to flavor, and determined compounds responsible for common aromas. Nonetheless, the whey protein flavor code has yet to be cracked. Numerous processing steps, conditions, and other variables require research in order to meet the lofty goal of a bland dairy protein ingredient with subtle dairy flavors.

LINKING LEXICONS AND FLAVOR CHEMISTRY TO THE CONSUMER

Preference Mapping

The ultimate goal of the sensory scientist and flavor chemist is to relate product attributes to the consumer. The determination of flavor attributes and the identification of compounds responsible for flavor serve the industry only when this data is converted to yield a better quality or higher-value food product. Descriptive sensory analysis yields a wealth of information but trained panelists function as part of an instrument rather than representing the consumer and determining product likes and dislikes. Through the use of preference mapping, volatile data and/or descriptive sensory data (aromatics, flavor, and/or texture attributes) can be modeled on consumer preference data. Preference mapping describes more than one technique, differentiated by the data used to locate products on the axes (Meilgaard et al. 2007, Drake 2007). Internal preference mapping places products by consumer acceptance values. External preference mapping places products using

Table 5 Volatile compounds identified in liquid whey and dried whey products[a]

Volatile compound	Whey type[b]	Volatile compound	Whey type[b]
(*E*)-2-hexenal	WPC80, agglom. WPC80, WPI, agglom. WPI	acetone	liquid
(*E*)-2-nonenal	liquid, SWP, WPC80, WPI	benzaldehyde	liquid, WPC80, agglom. WPC80, WPI, agglom. WPI
(*E*)-2-octenal	liquid, SWP, WPC80	butadiene	liquid, WPC80, WPI
(*E,E*)-2,4-decadienal	liquid, SWP, WPC80, WPI	butanoic acid	liquid, SWP, WPC80, WPI
(*E,E*)-2,4-nonadienal	SWP, WPI	butanol	liquid
(*E,E*)-2,4-octadienal	SWP	decanal	WPC80, WPI
(*E,Z*)-2,4-decadienal	SWP	decanoic acid	SWP, WPC, WPI
(*E,Z*)-2,4-nonadienal	SWP	delta-decalactone	liquid, SWP, WPC80, WPI
(*E-Z*)-2,6-nonadienal	liquid, SWP, WPC80, WPI	delta-dodecalactone	SWP, WPC80, WPI
(*Z*)-1,5-octadien-3-one	WPC66	delta-octalactone	SWP
(*Z*)-2-nonenal	liquid, SWP, WPI	delta-undecalactone	SWP
(*Z*)-4-heptanal	WPC80	diacetyl	liquid, SWP, WPC80, agglom. WPC80, WPI, agglom. WPI
(*Z*)-4-heptenal	SWP	dimethyl disulfide	SWP, WPC80, inst. WPC80, agglom. WPC80, WPI, agglom. WPI
1,2-propadiene; alkenyl	liquid	dimethyl sulfide	liquid, WPC80, WPI
1,5-octadienone	WPI	dimethyl trisulfide	liquid, SWP, WPC80, agglom. WCP80, WPI, agglom. WPI
1-dodecane	liquid	dimethylamine, allyl	liquid
1-hexen-3-one	WPC66	dodecanoic acid	SWP, WPC35, WPI
1-nonen-3-one	WPC66	ethanol	liquid
1-octen-3-ol	WPC80, agglom. WPC80, WPI, agglom. WPI	ethyl acetate	liquid
1-octen-3-one	liquid, SWP, WPC80, inst. WPC80, WPI	ethyl hexanoate	WPC66
1-pentanol	WPC66	formic acid	SWP
1-propanol	liquid	furfuryl alcohol	SWP
2,3-methylbutanal	WPC66	gamma-decalactone	SWP, WPC80
2,3-methylbutanol	liquid	gamma-dodecalactone	SWP
2,3,5-trimethylpyrazine	SWP	gamma-hexalactone	SWP
2,3-dimethylpyrazine	SWP	gamma-nonalactone	WPC80
2,3-methylbutanoic acid	liquid	gamma-octalactone	WPC66
2,3-pentanedione	WPC66	heptanal	liquid, SWP, WPC80, inst. WPC80, agglom. WPC80, WPI, agglom. WPI
2,5-dimethylpyrazine	SWP	heptanoic acid	SWP, WPC80, WPI
2,5-dimethyl-4-hydroxy-3-(2H) furanone (Furaneol)	SWP, WPC80, WPI	heptanone	liquid
2,5-octanedione	WPC66	hexanal	liquid, SWP, WPC80, inst. WPC80, agglom. WPC80, WPI, agglom. WPI

(*Continued*)

Table 5 (*Continued*)

Volatile compound	Whey type[b]	Volatile compound	Whey type[b]
2,6-dimethylpyrazine	SWP	hexanoic acid	liquid, SWP, WPC80, WPI
2-acetyl-1-pyrroline	liquid, SWP, WPC80, WPI	hydrocarboxyl	liquid
2-acetylpyridine	WPC66	isobutyric acid	liquid
2-acetylpyrrole	SWP	maltol	liquid, SWP, WPC35
2-acetylthiazole	SWP	methional	liquid, SWP, WPC80, WPI
2-butanol	liquid	methyl propanoic acid	liquid
2-butanone	liquid	nonanal	liquid, SWP, WPC80, agglom. WPC80, WPI, agglom. WPI
2-butanone	liquid	nonanoic acid	WPC80, WPI
2-ethylpyrazine	liquid, SWP	nonanol	WPC80, inst. WPC80
2-ethyl-1-hexanol	WPC80, agglom. WPC80, WPI, agglom. WPI	nonanone	liquid
2-furfural	liquid	*o*-aminoacetophenone	WPC80, WPI
2-heptanol	WPC80, inst. WPC80	octanal	liquid, WPC80, agglom. WPC80, WPI, agglom. WPI
2-heptanone	liquid, WPC80, agglom. WPC80, WPI, agglom. WPI	octanoic acid	liquid, SWP, WPC80, WPI
2-isobutyl-3-methoxypyrazine	liquid, WPC80, WPI	octanol	WPC80, inst. WPC80
2-isopropyl-3-methoxypyrazine	WPC66	*p*-cresol	SWP, WPC66
2-methoxyphenol (guiacol)	WPC80	pentanal	liquid
2-methoxy-3-isopropylpyrazine	liquid	pentanoic acid	liquid, SWP, WPC80, WPI
2-methyl propanoic acid	SWP	phenol	liquid
2-methyl-3-furanthiol	liquid, WPC66, WPI	phenyl ethyl acetate	WPC80
2-nonanol	WPC80, inst. WPC80	phenylacetaldehyde	SWP, WPC66, WPI
2-nonanone	liquid, WPC80, agglom. WPC80, WPI, agglom. WPI	propan-1-ol, alkyl	liquid
2-octanone	WPC80, inst. WPC80	propanoic acid	liquid, SWP, WPC66
2-pentylfuran	liquid, WPC80, agglom. WPC80, WPI, agglom. WPI	skatole	SWP, WPC66
2-phenethanol	WPC80, WPI	toluene	WPC80, agglom. WPC80, WPI, agglom. WPI
2-propanol	liquid	fatty acids	
2-propionyl-1-pyrroline	SWP	butyric	liquid
2-undecanone	WPC80, agglom. WPC80, WPI, agglom. WPI	caproic	liquid
3-hydroxy-4,5-dimethyl-2-(5H)-furanone (Sotolon)	liquid, SWP, WPC80, WPI	caprylic	liquid
3-methoxy-4-hydroxy benzaldehyde (vanillin)	WPC80	capric	liquid

(*Continued*)

Table 5 (*Continued*)

Volatile compound	Whey type[b]	Volatile compound	Whey type[b]
3-methyl butanoic acid	SWP	lauric	liquid
3-methyl furan	liquid	myristic	liquid
4-methyl octanoic acid	WPC80, WPI	palmitic	liquid
9-decanoic acid	SWP	palmitoleic	liquid
acetaldehyde	liquid	stearic	liquid
acetic acid	liquid, SWP, WPC80, WPI	oleic	liquid
acetoin	liquid	linoleic	liquid

[a]Adapted from Drake et al. 2009b.

[b]Cheese source. liquid: Cheddar, Gouda, Mozzarella, Paneer, Quarg, rennet casein, acid casein, lactic acid casein; SWP: Cheddar; WPC80: Cheddar, Mozzarella, Monterey Jack; inst. WPC80; Cheddar, Monterey Jack; agglom. WPC80; Cheddar, Mozzerella; WPI: Cheddar; agglom. WPI: Cheddar.
Abbreviations: SWP, sweet whey powder; WPC80, whey protein concentrate 80% protein; inst. WPC80, instantized WPC80; agglom. WPC80, agglomerated WPC80; WPI, whey protein isolate >90% protein; agglom. WPI, agglomerated WPI.
Carunchia Whetstine et al. 2003, 2005; Croissant et al. 2009; Drake et al. 2003, 2009a; Gallardo-Escamilla et al. 2005; Javidipour & Qian 2008; Karagul-Yuceer et al. 2003; Mahajan et al. 2004; Russell et al. 2006; Tomaino et al. 2004; Wright et al. 2006, 2009.

descriptive sensory values or even instrumental values. Partial least-squares regression analysis is the most common type of modeling approach used with external mapping (Tenenhaus et al. 2005).

Given the proper line of consumer questioning, preference mapping provides consumer segment information and respective liking attributes. Murray & Delahunty (2000) investigated consumer preference for Cheddar cheese and cheese packaging. Demographic information allowed characterizations of consumer segments, and relationships between purchasing habits and liking were characterized. Preference mapping is equally suited for correlation of instrumental analysis with consumer liking. Pham et al. (2008) utilized HS-SPME GC-MS and GC-O to identify aroma impact compounds and descriptive sensory and consumer acceptance testing to determine drivers of liking of dry-cured hams. All products tested had similar volatile compound profiles but differed in relative concentrations. Consumer testing provided information on aroma compounds that may be responsible for higher and lower acceptability scores.

Check-all-that-apply (CATA) (Dooley et al. 2010) and open-ended questions (Ares et al. 2010) have been proposed as alternative approaches to gain additional product information from the consumer. Modeling of both approaches compared well with external preference mapping. Both methods provide additional information in the consumers' language rather than the trained panel. However, both methods results in data that is not comparable to descriptive language and intensities. Previous studies have also suggested that the use of open-ended questioning may influence consumer overall liking scores.

Texture

Barden et al. (2009) determined that flavor does not impact texture perception in WPI gels by consumers or trained panelists. Conversely, texture has been shown to influence the perception of flavor (Lubbers 2006) and overall consumer liking (Yates & Drake 2007). Although the relationship between texture, aroma release, and flavor perception is outside the scope of this review, the term creaminess is discussed briefly in order to highlight difficulties encountered when attempting to relate flavor chemistry to consumer response. "Texture is a sensory property" (Szczesniak 2002); as such, terms have been established for the sensory analysis of texture (Meilgaard et al. 2007). However, the term creaminess is a source of ambiguity in the sensory language, with

applications in both texture and flavor, and probably implying different things with different foods. Representing texture, creaminess has been correlated with fat content (Richardson-Harman et al. 2000), particle size (Kilcast & Clegg 2002), viscosity or thickness (Daget et al. 1988, Frost & Danhoj 2007), and smoothness and consistency (Elmore et al. 1999, Frost & Danhoj 2007). From a flavor and consumer perspective, creaminess has been related to dairy and nondairy flavors (Frost & Danhoj 2007, Kirkmeyer & Tepper 2005, Richardson-Harman et al. 2000) and positively influencing overall liking (Kirkmeyer & Tepper 2005, Weenen et al. 2005). The term has been researched in numerous dairy products (Richardson-Harman et al. 2000) and is commonly used to describe appearance, texture, and flavor attributes (Elmore et al. 1999). Understanding the relationship between consumer perception and liking related to creaminess and texture/flavor profiles is important given the growing interest in low-fat foods, especially dairy products. The difficulty of quantifying this attribute is based on its lack of definition in descriptive sensory analysis and the inability of the consumer to differentiate flavor, texture, and liking. Generally, studies investigating the relationship between creaminess and consumer liking have shown positive correlation. However, differentiation between the physical and chemical properties of creaminess and the sensory perception of creaminess has not been achieved (Frost & Danhoj 2007). Frost & Danhoj (2007) concluded that texture properties correlated creaminess in liquid and semisolid dairy products, but the flavor properties correlated creaminess in weak gels (i.e., yogurts).

Creaminess perception includes both flavor and texture sensations (Tournier et al. 2007). Instrumental aroma analysis has also been utilized to investigate the role of volatile and semivolatile compounds on creaminess. Schlutt et al. (2007) identified several lactone compounds as contributors to creaminess flavor. These compounds, γ- and δ-octadecalactones and γ- and δ-eicosalactones, contribute to creaminess by influencing the melting behavior of cream in the oral cavity rather than having a direct influence on flavor. Only one compound when added to whipped cream above threshold, δ-tetradecalactone, was shown to affect creamy flavor. Creaminess in dairy products is a complex term involving compounds that elicit a creamy feeling factor, enhancing sensory attributes (e.g., cooked, milk fat), sensory attributes that decrease creamy perception (sour, bitter, e.g.,), and texture qualities (e.g., viscosity, particle size). A greater understanding of creaminess will only be accomplished by a complete approach utilizing sensory and instrumental techniques.

SUMMARY POINTS

1. Descriptive sensory analysis is a powerful analytical method to evaluate food products.
2. Sensory and instrumental analyses are complementary in the investigation of food flavor.
3. Sensory properties and flavor compound analysis can be related to consumer response to determine drivers of liking of food products.

FUTURE ISSUES

1. The effects of texture on flavor perception.
2. The relationship between the psychological and the physical consumer response.
3. Real-time measurement of flavor release and flavor perception by way of in-mouth analysis.

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Contents

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