

AN INTEGRATED APPROACH TO SENSORY ANALYSIS OF RICE FLAVOR

by

MARUJ LIMPAWATTANA

(Under the Direction of Robert L. Shewfelt)

ABSTRACT

Flavor is one key factor contributing to consumer acceptance and repeat purchase of rice. As a result, a systematic approach for rice breeders to select rice with favorable flavor traits is needed. Descriptive sensory analysis combined with chemical analysis provided an insight of sensory significance to interpret chemical data for a better understanding approach of rice flavor quality. By using trained panelists, this project aimed to develop rice flavor lexicon, identify sensory thresholds of character-impact compounds in different matrices and relate sensory descriptive notes to chemical composition of rice.

A rice flavor lexicon consisting of 24 descriptive notes was developed by eight trained sensory panelists to characterize the flavor of a broad spectrum of cooked rice (n=36). Of these 24 descriptive terms, 19 are aromatic notes and five are fundamental tastes and oral feeling factors. Eighteen aromatic terms were significantly present in most rice samples while some descriptors exhibited unique characteristic of a specific-rice type. Subsequent multivariate analysis indicated that 18 descriptive terms were required to fully understand the characteristics of rice flavor in greater details.

Orthonasal detection thresholds of selected volatile compounds varied among three matrices. Threshold values of all selected volatiles determined in deionized water were the

lowest followed by partially deodorized rice and corn starch. Partially deodorized rice was developed as an alternative matrix close to the actual cooked rice. Comparison of odor activity values (OAVs) between three matrices suggested that there was a matrix effect in that the spiked volatile compounds interacted with medium causing the suppression of odor perception.

Nineteen-aromatic descriptors from an established flavor lexicon, evaluated in 13 cooked specialty-rice types were regressed against the concentrations of aroma-active compounds derived from gas chromatography-olfactometry (GC-O) analysis. Significant models obtained from stepwise multiple linear regression were developed from most of aromatics descriptors including popcorn, cooked grain, starchy, woody, smoky, grain, corn, hay-like, barny, rancid, waxy, earthy and sweet aromatics. Compounds that were the prominent contributors to rice aroma include (*E,E*)-2,4-decadienal, naphthalene, guaiacol, (*E*)-2-hexenal, 2-acetyl-1-pyrroline and 2-heptanone.

INDEX WORDS: Rice, Sensory, Flavor lexicon, Volatile compounds, Thresholds, Gas chromatography-olfactometry

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MARUJ LIMPAWATTANA

B.S., King Mongkut's Institute of Technology Ladkrabang, Thailand, 1994

M.Fd.Tech., The University of Newcastle, Australia, 1999

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MARUJ LIMPAWATTANA

Major Professor:	Robert L. Shewfelt
Committee:	Stanley J. Kays Ronald R. Eitenmiller Manjeet S. Chinnan William L. Kerr

Electronic Version Approved:

Maureen Grasso
Dean of the Graduate School
The University of Georgia
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DEDICATION

To my beloved Mom and Dad who have brought me up with their great love and care, given me the best education and taught me with their great knowledge and experiences for both life and learn that a decent man should be. To my three dearest sisters, who have supported me and cherished me. I dedicate this work to all of you.

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CHAPTER 1

INTRODUCTION

Rice (*Oryza sativa* Linn.) is a well-known staple food for nearly half of the world population accounting for 20 percent of the world's dietary energy supply (FAO 2004). In 2006, world production of paddy rice was 638 million tonnes, equivalent to 429 million tonnes of milled rice (FAO 2007). Thailand, Vietnam and India are the top three of the world's largest milled rice exporting countries. The United States, the only non-Asian producer, ranks the fourth exporting about 3.3 million tonnes of milled rice which is nearly half of total gross production, to the world market (USDA 2007b). Although per capita consumption of rice in the United States was about 21 lb in 2005, consumption is expected to increase each year, primarily due to population growth in the United States including an increase of Asian-American and Hispanic-American populations (USDA 2007a).

Most rice is consumed as whole kernels and the acceptance varies from country to country and even between regions within a specific country (Juliano 1990; Suwansri and others 2002). Consumers in the United States prefer dry and fluffy to moist and chewy for cooked rice or vice versa compared to the consumers in Asia (Lee 1998). Asian consumers living in the U.S. would greatly accept rice based on appearance and aroma (Meullenet and others 2001). Minor changes in sensory properties, especially aroma, can make rice and its products unacceptable for the consumers.

The flavor of rice has been investigated by both instrumental and sensory methods. Descriptive sensory analysis is known as a primary tool to characterize products qualitatively and quantitatively (Munoz and Civille 1998). Numerous studies on rice properties merely emphasize texture and cooking (Champagne and others 1998; Meullenet and others 2000; Sesmat and Meullenet 2001; Mohapatra and Bal 2006) over flavor (Yau and Huang 1996; Champagne and others 2004). The expansive flavor lexicon for characterizing specific flavors in rice or a wide spectrum of rice samples was yet missing.

More than 300 volatile aroma components have been identified in rice but only a few contribute to the characteristic aroma of rice (Yajima and others 1978; Buttery and others 1982; Buttery and others 1988; Widjaja and others 1996; Jezussek and others 2002). Among these, 2-acetyl-1-pyrroline is the most important volatile emitting a popcorn-like aroma and is present in all parts of the plant except roots (Yoshihashi 2002). Determination of odor threshold is the initial approach to select volatile components that contribute to a characteristic aroma of the food from those that do not (Teranishi and others 1991).

Odor threshold values depend on the medium in which the component is dissolved (Rothe 1976). Although most of odor-threshold values are determined in air or water, the use of the partially deodorized rice as the medium of evaluation are deemed to be more realistic, than in air or water. Marsili (2007) suggested that sensory-threshold data should be determined in the same matrix as the sample being studied.

Efforts are ongoing by several U.S. rice breeding programs to develop rice cultivars that are acceptable to consumers. Rapid methods focusing on rice flavor quality are needed to assist rice breeders in making their selections from the multitude of lines. Recent studies by Yang (2007) investigated the odor-active compounds emanating from different types of cooked rice

using gas chromatography-olfactometry. With further statistical analysis by multivariate techniques, volatile compounds important to rice flavor were separated and characterized. As a result, sensory evaluation should be a critical component of those programs to provide a basis for screening of rice breeding lines.

The objectives of this study were:

1. To establish a descriptive lexicon with standard references for describing the flavor properties of a wide array of rice and use developed lexicon to characterize the important sensory attributes in rice flavor quality.
2. To determine the orthonasal detection thresholds of selected odor-active compounds responsible for the aroma of aromatic rice in three different matrices; water, corn starch and partially deodorized rice and determine the sensitivity range of the existing trained panelists.
3. To develop prediction models for sensory descriptors as functions of volatile components in rice flavor derived from gas chromatography-olfactometry (GC-O).

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CHAPTER 2
LITERATURE REVIEW

Rice production

Rice (*Oryza sativa* Linn.) is indigenous to Asia and provides a staple food for nearly half of the world population. The other cultivar, *Oryza glaberrima*, is only grown in Africa with a limited scale (Juliano 1985a). Two major ecogeographical races of *Oryza sativa* are japonica rice and indica rice. These rice races are grown in the temperate region and in the tropical and subtropical areas, respectively. Javanica rice belongs to the japonica race of *O. sativa* and is cultivated in Indonesia. In 2006, the world produced about 638 million tonnes of paddy rice which was equivalent to 429 million tonnes of milled rice (FAO 2007). Production is geographically concentrated in Western and Eastern Asia with more than 90 percent of world output. China and India account for more than half of world output but mostly for domestic consumption. Thailand, Vietnam and Pakistan are the top three of the world's largest milled rice exporting countries where the non-Asian producer like the United States ranks the fourth exporting about 3.3 million tonnes of milled rice to the world market (FAO 2007). The global rice trade is segmented into long-grain, medium and short grain, aromatic and specialty (primarily glutinous) rice which approximately accounts for 75 %, 12 %, 12 % and 1 %, respectively (FAO 2007). The major rice-producing states are: Arkansas, California, Florida, Louisiana, Texas, Mississippi, and Missouri. The international price of rice has been remarkably increased for high quality rice, which is strongly demanded in the world trade.

Rice composition

As shown in Figure 2.1, rough rice or *paddy* is the caryopsis or kernel of rice harvested with the hull or husk attached (or known as rice grain after threshing and winnowing). The hull constitutes about 20 % of the weight of rice and contains 25 % cellulose, 30 % lignin, 15 %

pentosan and 21 % ash (Hoseney 1994). Rice, after the hull is removed, is called *brown rice* which varies from 5 to 8 mm in length, and weighs about 25 mg. Brown rice consists of approximately 2 % pericarp, 5 % seed coat and aleurone, 2-3 % germ and 89-94 % endosperm. Hemicellulose in rice endosperm is low and composed of arabinose, xylose, and galactose-containing polymers, as well as protein and a large amount of uronic acids. During milling, bran is derived from the combination removal of the outermost layer of endosperm (aleurone), pericarp and seed coat and known as an excellent source of water soluble vitamins and vitamin E with little or no β -carotene or vitamins, C or D (Hoseney 1994). Milling results in the loss of lipid, protein, fiber, reducing sugar and total sugars, ash and minor components including vitamins, free amino acids and free fatty acids from grains (Heinemann and others 2005; Singh and others 1999). The thin-walled endosperm cells are tightly packed, with polygonal compound

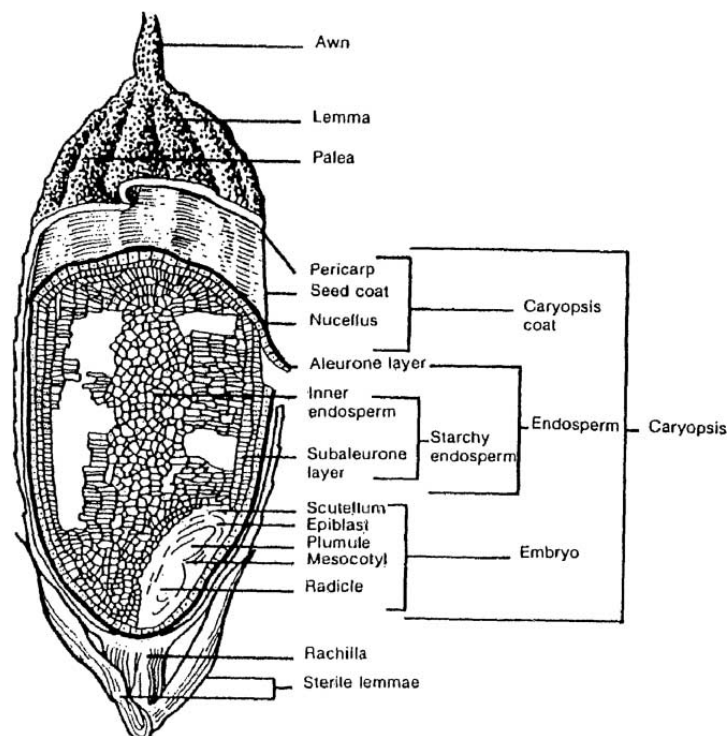


Figure 2.1 The structure of rice grain (Zhou and others 2002)

starch granules and protein bodies. The subaleurone layer is rich in protein and lipids and has smaller amyloplasts and compound starch granules than the inner endosperm. Flour is made when the contents and cell walls of the endosperm cells are reduced to appropriate particle size.

Rice starch granules fill within the endosperm cells and fraction into linear chain amylose and the branched amylopectin. Waxy rice contains 0-2 % amylose content compared with more than 25 % in non-waxy rice which in turn affects cooking characteristics, texture, water absorption ability, stickiness, volume expansion, hardness including the whiteness and glossiness of cooked milled rice (Juliano 1985). Suggested classification based on amylose content is waxy (0-5 %), very low (5-12 %), low (12-20 %), intermediate (20-25 %), and high (25-33 %) (Juliano 1985). The amylose content in japonica, indica, and javanica rice is in the range of 0-20 %, 25-33 % and 0-25 % respectively. The amylose chain has a helical conformation with six anhydroglucose units per turn yielding an internal hydrophobic cavity that allows to form inclusion compounds with other hydrophobic compounds by van der Waals bonds with adjacent C-hydrogen of amylose (Godet and others 1993). Monoacyl lipids and different volatile flavor compounds such as alcohols, aldehydes, lactones and terpenes are capable of inducing the helication of amylose and act as inclusion partners (Heinemann and others 2005).

Rice contains about 3 % lipids concentrated in the peripheral parts of the grain. Rice lipids can be classified as neutral lipids, glycolipids, and phospholipids with no difference in terms of the ratio between *japonica* and *indica* (Mano and others 1999). The lipid content of brown rice and milled rice range from 2.1-3.2% and 0.61-0.95%, respectively (Singh and others 1998). Juliano (1985) classified rice lipids into starch lipids which are associated with starch granules and non-starch lipids which are distributed throughout the grain. Starch lipids were mainly fatty acids (palmitic and linoleic acids) and phospholipids attributed to the formation of

the helical inclusion complex (Choudhury and Juliano 1980; Kitahara and others 1997). The major fatty acids of non-starch lipids were linoleic, oleic and palmitic (Hemavathy and Prabhakar 1987; Taira and others 1988). Rice oil contains a phenolic antioxidant (oryzanol), an ester of ferulic acid, and triterpene alcohols (Juliano 1985).

The protein content of rice is generally lower than that of the other cereals. For brown rice, the protein content ranged from 6.6-7.3 % but 6.2-6.9 % for milled rice (Singh and others 1998). Lysine is the first limiting amino acid based on human requirements, followed by threonine (Juliano 1985). Rice protein fractions such as albumin, globulin, prolamin and glutelin are soluble in water, salt, alcohol and alkali respectively and varied among rice. The distribution of different solubility fractions is uneven. Albumin and globulins are concentrated in the embryo and aleurone layer. The storage proteins (oryzenin and prolamin) occur in the highest amount in the endosperm. Glutelin (oryzenin) was the highest range (64-75 %) of the total protein (Basak and others 2002). Degree of polishing which removes the peripheral layer of the kernel was inversely correlated with the protein and fat content (Pal and others 1999).

Sensory evaluation methods

The Institute of Food Technologists developed the definition for sensory evaluation as “ a scientific discipline used to evoke, measure, analyze, and interpret reactions to those characteristics of foods and materials as they are perceived by the senses of sight, smell, taste, touch and hearing” (Dethmers and others 1981). Scientific principles drawn from food science, physiology, psychology and statistics are taken into account to elicit objective responses to the properties of foods (Piggott and others 1998). Sensory evaluation is concerned with precision, accuracy, sensitivity and avoiding false positive results (Lawless and Heymann 1999). Two

major classifications of sensory tests are developed according to their primary purpose and most valid use: analytical tests and affective tests. The analytical tests are classified as discriminative tests and descriptive tests. The purpose of these tests is to indicate the differences/ similarities and to identify and quantify the sensory characteristics respectively. The affective tests evaluate the preference and/or acceptance of a product.

There are several variations of discrimination tests including the Paired-comparison, Duo-Trio, Triangle, Ranking and Rating difference (Dethmers and others 1981), A-not A, 3-Alternative Forced Choice (directional difference), and Two-out-of-Five test (Meilgaard and others 2007). Difference tests are designed to measure difference, not the sameness. If the frequency of correct solutions is higher than that expected by chance, then a difference is declared.

Descriptive sensory analysis

Descriptive sensory tests are among the most sophisticated tools that use trained panelists to characterize the qualitative components and intensify the quantitative components (Lawless and Heymann 1999; Meilgaard and others 2007). Qualitative components are the perceived sensory attributes such as aroma, flavor, appearance, texture of the products. Quantitative components are expressed by the assigned values using a proper scale including category scales, line scales, and magnitude estimation (ME) (Meilgaard and others 2007). Improper use of scaling technique affects the validity and reliability of terms (Meilgaard and others 2007). All descriptive analysis methods require the training for the selected panel. Generally during the training period, a new panel will develop the sensory language to be used to describe the product with the aid of reference standards to align the concept of each panelists into a same way (Munoz

and Civille 1998). With exception to FCP method, terminology generation is done after the completion of training. Numerous papers discuss the selection of sensory panelists including screening tests (Issanchou and others 1995; McDaniel and others 1987) and monitoring panel performances (Derndorfer and others 2005; Pages and Perinel 2004; Malundo and Resurreccion 1992; Mangan 1992). A broad array of factors is suggested to select panelists for descriptive analysis including health status, allergies, availability, verbal creativity, concentration, motivation, team player, smoker, dietary habits, education, sensitivity, specific anosmias, previous experience, dentures, medication, use of products, supplements (Piggott and others 1998). Details for the numbers of hours required and training procedure are well elaborated elsewhere (Lawless and Heymann 1999; Stone and Sidel 2004; Meilgaard and others 2007).

There are several different methods of descriptive analysis including the Flavor Profile Method (FPM), Texture Profile Method (TPM), Quantitative Descriptive Analysis[®] (QDA), the Spectrum[™] method. The first two techniques have a commonality in that the consensus for all attributes is achieved from a highly trained panel. However, TPM is designed to better interpret the relationship between rheology and its nomenclature rather than to describe texture properties, unlike the FPM that is used to assess the flavor and aroma impressions of food. The latter two techniques differ markedly from FPM and TFM in that they are designed to take measurements from individual panelists and then generate a panel average, rather than generation of a group consensus profile as with FPM and TFM (Piggott and others 1998). Also, panelists are given more standards and training to reduce panelist variability and so increase discriminability between products and over time. Subsequent data analysis is introduced to remove unwanted variation. A detailed overview of all four techniques can be found in the Manual on Descriptive Analysis Testing for Sensory Evaluation (Hootman 1992). The Quantitative Flavor Profiling

(QFP) is a modified method from QDA developed by Givaudan-Roure, Switzerland (Stampanoni 1993). This method focuses on flavor only using language generated by flavorists who do not involved in the evaluation. Free-Choice Profiling is a variation of descriptive analysis which is different from QDA and the Spectrum method where consumers are allowed to use any number of their own attributes to describe and quantify product attributes. Data from this method is analyzed by generalized procrusted analysis. Time-intensity scaling (TI) is a special case of descriptive analysis, where a single characteristic is tracked as it changes over a period of time (Murray and others 2001).

Development of flavor lexicons

Flavor lexicons are the lists of descriptors generated during the training phase of descriptive sensory analysis to characterize the flavor of products. The basis of lexicon composes of aromatics, tastes and feeling factors. A panel is provided with a set of reference which is basically the products in the category of interest in order to cover the wide array of flavor characteristics (Meilgaard and others 2007). Then, the references are provided for the grouped term and finally the redundant terms are eliminated by consensus or statistical methods (Lyon 1987).

A good descriptive term must be able to differentiate itself from similar sensation, identify the ascribable product and allow the panelist to recognize the product (Civille and Lawless 1986). Precise definitions would ensure that all panelists refer to a same sensory concept and reduce the ambivalence in meaning that may exist among panel members. The precise and clear in meaning will further provide a consistent panel performance and allow the repetition or comparison of the tests. Criteria underlying the construction of specific terms is

that the term should be uncorrelated with each other, related to the intrinsic natural structure, determined with a broad reference set and elemental not integrated (Civille and Lawless 1986). Lawless and Heymann (1999) recommended that descriptors should be singular (one-dimensional) rather than with combinations of several terms or ideas since the latter are prone to be confusing for panelists. Use of examples as an illustration but not to be in place of definition is acknowledged to avoid the ambiguity and reach the consensus (Meilgaard and others 2007). The writing guidelines using linguistic analysis are recently developed to properly define sensory descriptors in that the good definition composes of substitutability, paraphrasing and simplicity (Giboreau and others 2007). Substitutability means that reading a sentence using the definition in place of the descriptor should be possible without any change. Paraphrasing is concerned with the same meaning of the defined terms even though when different words are used to express. Finally, simplicity deals with the avoidance of complex wording in definitions but positive.

The useful tools during the training phase are reference standards. Reference may be food, chemicals or other substances that can be used to characterize or identify the attribute and also determine the intensities (Rainey 1986). The reference that is less complex in nature, reproducible, simply identified and diluted without changing character is recommended (Rainey 1986). Multiple references are also recommended for a better alignment of concept of product characteristics for panelists (Ishii and Omahony 1987; Ishii and Omahony 1991) since one single reference may not properly describe what panelists detect in the products. Several products may need to show that each has a similar characteristic, perhaps a different intensities but the same characteristic. The broad range of samples represented their flavor variation would enhance descriptors for which they are developed to be more discriminating (Meilgaard and others 2007).

Generally, panelists (trained or naive consumers) use their background information and reference points in their mind which is called the frame of reference to identify and quantify the perceive attributes (Munoz and Civille 1998). Without training, responses derived from these panelists vary widely if a common frame of reference is not developed for the panel. Reference can be qualitative, quantitative or both (Munoz and Civille 1998). Qualitative references associate with the use of collective terms from each panelist's past experience to identify a perception. Training programs in descriptive sensory method will establish the new qualitative frame of references for a panel to evaluate the products in the same way and thus shorten the training time (Rainey 1986). Focusing on one or two specific attributes provided with appropriate reference standards in each discussion is recommended as to increase panelists' motivation (Rainey 1986).

There are three types of quantitative references: universal, product specific and attribute specific (Munoz and Civille 1998) applied on scaling technique. Quantitative references are the intensities that panelists used to rate the strength of the perceived attribute. In the Spectrum™ method, a universal scale is used. The key feature of the universal scaling is that all attributes of any product are rated relative to the universal scale intensity reference (Meilgaard and others 2007). Therefore, comparative test across product or across attribute in the same product including different panel is achieved (Munoz and Civille 1998). A product specific scale rates attributes within the product. An attribute specific scaling rates each attribute independently with its own intense reference.

Numerous studies have been conducted to develop flavor lexicons for specific products including warm-over flavor (WOF) of meat (Johnson and Civille 1986), pond-raised catfish (Johnsen and others 1987), chicken (Lyon 1987), peanut (Johnsen and others 1988), carbonated

water (Harper and McDaniel 1993), noodles (Janto and others 1998), cheddar cheese (Drake and others 2001), sweet potato (van Oirschot and others 2003), floral honey (Galan-Soldevilla and others 2005), soymilks (N'Kouka and others 2004; Chambers and others 2006), soy sauce (Jeong and others 2004), Chardonal wine (Mirarefi and others 2004), French cheese (Retiveau and others 2005), rose apples (Vara-ubol and others 2006), nopalitas (Perez-Cacho and others 2006), lemon/lime carbonated beverages (Kappes and others 2006), American soybean (edamame) (Krinsky and others 2006), and green tea (Lee and Chambers 2007).

Flavor perception

Flavor is the most recognizable feature to define foods as it is a comprehensive stimulation of taste and odor receptors. Basic tastes are detected by gustatory receptors within mouth where aroma is then combined to make up all flavors. Theoretically, the human nose has an odor detection limit of about 10^{-19} moles (Mistry and others 1997) making it a sensitive tool for the detection of potent volatiles. Orthonasal aroma is perceived by smelling or sniffing which travels to receptor sites on the olfactory epithelium high in the nasal cavity. When food in the mouth is disintegrated during mastication, salivation and swallowing, retronasal aroma is released from food and enters the oral cavity. Concentrations and rate at which the flavor chemicals reach the receptors create the characteristic flavor profile of a food (Taylor 2002). Sniffing is a quick inhalation through the nose and different from smelling in that it has an average velocity of 27 L/ min and a volume of 500 cm³ within 1.6 s (Laing 1983). Three deep and quick sniffs are recommended for aroma assessment to prevent odor adaptation which leads to sensory fatigue and a drop in panelist sensitivity (Carpenter and others 2000). One key feature of sniffing underlying olfactory perception is that sniffing affects odorant intensity and identity

perception (Mainland and Sobel 2006). The aroma a consumer perceives is influenced by three factors: intrinsic food properties, process of eating with other related aspects such as the physiology, anatomy and physico-chemistry of the mouth, and the psychosocial and cognitive factor (Yeretzian and others 2004). Odor identification is successful when the odor of interest is familiarized, having its name associated with its character and aided in recall (Cain 1979).

Tastes can increase the apparent intensity of aromas. Flavor notes associated with tastes such as fruitiness can increase the perceived intensity of sweetness (Noble 1996). Abagaz and others (2004) used noseclips to partition taste from aromatic flavor notes in fresh tomato during sensory evaluation. They found that flavor notes were more pronounced when evaluated separately after taste perception making the taste descriptors better correlated with nonvolatile components. The more recent study indicated that most tastants can be smelled since blocking olfaction using noseclips reduce taste intensity ratings (Mojet and others 2005). The nature of the food matrix also plays an important role in overall process of food perception. Three mechanisms are involved in the interaction between aroma compounds and foods: the partition of flavor molecules between different phases of the food products, the diffusion of flavor molecules through the food and matrix and the binding of flavor molecule to food component (Taylor 1998). Retention of aroma compounds is influenced by the composition and the microstructure of the medium (Seuvre and others 2000).

A review indicated that volatile molecules interact with food ingredients and, thus, influence flavor perception (Guichard 2002). Regardless of the type of starch, retention increases with the polarity of the flavor molecule (Boutboul and others 2002). The amylose/amylopectin ratio of starch affected the retention of aroma compounds both from the formation of complexes and by a viscosity effect (Arvisenet and others 2002a). Another study

by this group indicated that the interactions occurred in starch matrices not only with amylose but with amylopectin (Arvisenet and others 2002b). Texture quality and flavor release from starch based food were reported to depend on starch/flavor interaction (Heinemann and others 2005). The more recent study confirmed that C-6 aroma compounds; (hexanol, hexanal, *trans*-2-hexenal and 2-hexenone) formed complexes with amylose resulting in aroma retention (Jouquand and others 2006).

Sensory thresholds

Food flavors commonly comprise complex mixtures of volatile compounds. Modern instrumental analysis of food flavors indicated that only a small number of these identified volatiles are of significance in determining the flavor (Grosch 2001). In aroma research it is necessary to select volatile components that contribute to a characteristic aroma of the food from those that do not. The first approach in evaluating the importance of different components in a particular food is to obtain information about their perception thresholds (Teranishi and others 1991). Aroma thresholds for food volatiles can vary from hundreds of ppm to sub-ppb levels. Therefore, compounds present in high concentration often provide little or no aroma activity; whereas, other aroma volatiles found at trace concentration may produce intense aroma activity.

Thresholds are the limits of sensory capacities including four types; absolute, recognition, difference, and terminal (Meilgaard and others 2007). Absolute or detection threshold is the minimum physical intensity detected without identification. Recognition threshold or identification threshold is the minimum physical intensity detected and identified correctly. Difference threshold is the smallest change in concentrations necessary to produce a noticeable difference. Terminal threshold is the maximum physical intensity detected above which increase

in intensity can not be detected. Volatiles present in food at higher concentrations than their minimum intensity detection are believed to contribute to the flavor of the food (Teranishi and others 1991).

Generally, it is accepted that aroma quality changes with concentration and the sensory threshold of the odorants. Published threshold values mostly were reported in water, air or other matrices (van Gemert 2003). Detection thresholds are not only useful measuring tools for specifying the potency of a flavor compound in food but also measuring an individual's sensitivity to some flavor compounds of interest (Meilgaard and others 2007). However, many factors such as genetic variability (anosmic, those who cannot perceive odor, or the aguesic, those who perceive no tastes), olfactory fatigue, and other factors such as temperature and humidity can influence one's ability to detect odor. Perception and rating of one attribute can differ among individuals, because assessors have different detection thresholds (Kilgast 1996).

Since most foods have high water content (70-90%), the odor thresholds of their volatiles are always cited in water. Odor threshold values depend on the medium in which the component is dissolved (Rothe 1976). A number of odor thresholds of key aroma compounds have been conducted in numerous foods including beer (Meilgaard 1993), wine (Martineau and others 1995), potatoes (Work and Camire 1996), bread (Rychlik and Grosch 1996), tomato (Tandon and others 2000), skim milk powder (Karagul-Yuceer and others 2004), apple juice (Eisele and Semon 2005), mango (Pino and Mesa 2006) and rice (Yang 2007).

Three classical psychophysical methods of determining thresholds are the Method of Constant Stimuli where different concentrations of the compound are presented in random order, the Method of Limits where the stimuli in ascending or descending order is presented, and the Method of Adjustment where the concentrations of the stimulus is slowly changed either by the

subject or the experimenter until the subject can just detect the stimulus (Durr 1994). The method of adjustment is the least accurate but the fastest and is well established in olfactometry (Durr 1994); whereas, the Methods of Limits becomes more useful (Meilgaard and others 2007). The short-cut Signal Detection Theory (SDT) measure of the size of small difference (d') has been proposed to investigate factors other than the subject's sensitivity that may influence the results of a threshold determination (O'Mahony 1979). However, this method is more time-consuming than other classical methods.

The principal underlying methods of limits is that detection thresholds are determined by the best-estimate criterion which is an interpolated concentration value presented. The scale steps of each odorant are used at a concentration factor of X which basically derived from the preliminary studies and indicated more accurate threshold values. Best-Estimate Threshold (BET) concentrations for the individual panelists were calculated as the geometric mean of the highest undetected concentration and lowest detected concentration. The lowest concentration detected is confirmed. The Best-Estimate Threshold for detection of the group will be calculated as the geometric mean of individual threshold values (ASTM 1997).

In other newer method, ASTM practice E 1432-04, the threshold is the stimulus level detectable by 50% of the population with a 0.5 probability. It determines individual threshold by fitting a response curve to the panelists' response to a range of concentrations. But the test requires five times more samples than the rapid method, and it must be repeated by adjusting stimulus levels until the threshold is determined for all panelists. Numbers of repetition and how to calculate the threshold as mentioned differentiate these two methods (ASTM 2004).

Sensory evaluation of rice flavor

Most rice is consumed as whole kernels and the acceptance varies from country to country and even between regions within a specific country. Most U.S. consumers prefer rice that cooks to produce dry and fluffy kernels. Other consumers, especially in Asia, prefer rice that cooks to be moist and chewy, with the kernels sticking together (Lee 1998). The acceptance of cooked rice by Asian people living in their countries has been reported (Juliano 1990) and by those living in the United States (Meullenet and others 2001; Suwansri and others 2002). Juliano (1990) indicated that Asian consumers preferred rice with high milling quality and high cooking quality as indicated by fewer broken rice with more polishing, and intermediate amylose content respectively. Meullenet and others (2001) found that appearance and aroma were the most important factors in determining the Asian consumer acceptance of cooked non-aromatic and aromatic varieties

Rice cultivars grown in the U.S. are divided by grain size and shape into three types: short, medium, and long grained. After cooking short and medium grain are moist and sticky while long grain are dry and fluffy. Only a small amount of waxy rice (with all amylopectin starch) is grown. However in other parts of the world, many rice cultivars are aromatic. U.S. long-grain cultivars are characterized by higher amylose content (23-27 %) than are short-and medium-grain types (15-21 %) (Hoseney 1994). There are numerous studies conducted on sensory properties of rice by descriptive analysis method.

Four commercial Taiwanese rice samples at different serving temperature (18° and 60° C) were studied using a modified quantitative descriptive analysis (Yau and Huang 1996). Samples were cooked and evaluated by 20 panelists using the developed sensory attributes which were grouped into appearance, aroma, flavor and texture. Descriptors used to evaluate

appearance were glossiness, whiteness and looseness. Aroma descriptors were hot-rice aroma, cold-rice aroma and brown rice aroma while sweetness was a flavor descriptor. Characteristics of texture were evaluated by kernelness, hardness, cohesiveness, stickiness, chewiness, and roughness. They found that serving temperature and samples affected the sensory properties of cooked rice.

Nine sensory texture attributes were developed to evaluate two long-grain rice and one – medium rice (Meullenet and others 1999). The samples were cooked and evaluated by 9 trained panelists using the Spectrum™ method. Sensory profile of long-grain rice was conducted by the Spectrum™ method using ten developed attributes by nine trained panelists (Meullenet and others 1999). The sensory attributes were overall flavor impact, starch note, cardboard note, sulfur note, and texture attributes including clumpiness, roughness, hardness, gluiness, moisture adsorption, cohesiveness of mass and geometry of slurry. The factors affecting the sensory properties of cooked rice were rough rice wet drying, drying temperature, storage temperature and storage condition. In terms of flavor attributes, starchy note and overall sensory impression decreased after four weeks of storage.

Twenty imported Jasmine rice samples from Thailand were assessed by the descriptive Spectrum™ methods for visual, flavor and texture. Descriptive aromatics and basic tastes lexicons were developed with reference standards by nine trained panelists (Suwansri and others 2002). Some flavor notes were used to describe aroma, aromatics and aftertaste including starchy, cooked grain, feedy, nutty, scorched, sweet aromatics, sulfury, heated oil, burlap, floral, woody, dairy notes and hot plastic. A metallic note was used to describe aroma, aromatics, aftertaste and feeling factor.

The descriptive analysis using the Spectrum™ methodology was used to categorize 79 rice cultivars developed in the United States (Bett-Garber and others 2001). In this study, twelve trained panelists adopted the sensory lexicon previously developed (Lyon and others 1999; Goodwin and others 1996). These descriptors were 1) aromatics: sewer or animal, floral, grain or starchy, haylike or musty, popcorn, corn, dairy and sweet aromatics, 2) tastes: sweet, sour or silage, 3) feeling factors: astringent, waterlike or metallic. Based on amylose content, protein content and sensory attributes, seven clusters of rice samples were categorized.

Sensory analysis of cooked rice was performed by the QDA® method to investigate the effect of milling ratio on sensory characteristics of short grain rice (Park and others 2001). Ten trained panelists developed twenty-three attributes of which nine attributes were used to describe flavor. The flavor descriptors were boiled egg white, puffed corn, dairy, raw rice, wet cardboard, hay-like, metallic, sweet taste and bitter taste. Puffed corn flavor, raw rice flavor, wet cardboard flavor, hay-like flavor and bitter taste were decreased with increased milling.

Cooked rice flavor intensities from 17 rice cultivars were low and similar with exception to grain flavor as described by the Spectrum™ descriptive analysis (Champagne and others 2004a). The finding also indicated that there were significantly different between the crop year sets in terms of the specific flavor attributes including hay-like, sweet aromatics, sour and astringent.

Milled white rice flavor was influenced by the moisture content and storage time before drying (Champagne and others 2004b). In this study, descriptive sensory analysis using the Spectrum™ method by 10 trained panelists showed that the intensity of sour/silage flavor and alfalfa/grassy/green bean flavor significantly increased in cooked rice derived from paddy rice with $\geq 24\%$ moisture content that was held for 48 hr. The flavor lexicon was again adopted from

Goodwin and his coworkers (1996). In another study, timing of field draining and harvesting date exhibited important effects on rice sensory (Champagne and others 2005). The spectrum™ method operated by ten trained panelizes found that drain and harvest dates did not significantly affect flavor of cooked rice samples. Some attributes such as floral, alfalfa/grassy/green bean flavor notes were not detected.

Thermally processed rice was sensorially evaluated against normal cooked rice using the QDA® method by twelve trained panelists. Although there was no difference between the samples for aroma and cooked-rice aroma, other desirable texture attributes such as plumpness and hardness were obtained with overall quality similar to the control rice sample (Prakash and others 2005).

Chemistry of rice flavor

Over the last three decades extensive research has been conducted in understanding the volatile compounds providing the characteristic aroma of cultivated rice (*Oryza sativa* L.) and rice product through instrumental analysis. Extensive studies on rice revealed that among 200 identified volatile compounds, only a few contributed to the characteristic aroma of rice (Yajima and others 1978; Buttery and others 1988; Widjaja and others 1996; Jezussek and others 2002).

Yajima and coworkers (1978) reported that in cooked rice by extraction of steam distillate, 100 compounds were identified including 13 hydrocarbons, 13 alcohols, 16 aldehydes, 14 ketones, 14 acids, eight esters, five phenols, three pyridines, six pyrrazines and eight other compounds. It is generally recognized that alcohols are formed by oxidation of lipids, aldehydes by oxidation of certain amino acids and fatty acids, and methyl ketones by beta-oxidation of fatty acids during heating.

The key compound responsible for the aroma of rice was firstly reported as 2-acetyl-1-pyrroline (2-AP), a lipophilic compound contributing to a popcorn-like aroma (Buttery and others 1982). Aromatic rice varieties contained 0.04-0.09 ppm of 2-acetyl-1-pyrroline; whereas, only 0.008 or less (10 times less) was found in ordinary rice varieties (Buttery and others 1983). In this study, odor threshold of 2-AP was given at 0.1 ppb. It was believed that 2-AP was thermally produced since it was detected in cooked rice and not raw rice. However, a recent study showed that 2-acetyl-1-pyrroline is present in all parts of the plant (stems, leave, grains) except roots (Yoshihashi 2002). Early study had suggested the simple method to differentiate between scented rice and non-scented rice by warming up the leaves in the closed vial (Nagaraju and others 1975).

Determination of odor thresholds for 64 known volatile compounds in cooked long grain rice was reported (Buttery and others 1988). Among those, the lowest threshold value was (*E,E*)-2,4-decadienal (0.07 ppb), followed by (*E*)-2-nonenal (0.08 ppb) and 2-AP (0.01 ppb) respectively. Based on odor unit value which was obtained by dividing the concentration of the compound in the food by its odor threshold in water, the major volatiles contributed to the long grain rice odor were identified. It is recognized that if the odor unit is less than 1 meaning that the threshold is not reached and, thus, the compound is unlikely to contribute to the overall odor.

Paule and Powers (1989) reported that 2-AP in Basmati 370 which was the aromatic rice from Pakistan and in Khao Dawk Mali 105 (Jasmine rice from Thailand) was positively correlated with descriptive terms ‘popcorn-like’ as described by non-oriental panel and ‘pandan-like’ as described by oriental panel. One key factor for successful odor identification is the common encountered terms (Cain 1979) since pandan leaves are popularly used as flavoring agent in the traditional Asian cooking.

The domestic (US-grown) aromatic rice called ‘Della’ was reported to possess the popcorn-like characteristic due to the presence of 2-AP (Lin and others 1990). The content of 2-AP in Della rice was almost four times more than that found in imported Jasmine from Thailand. In another study, Italian aromatic rice (line B5-3) was reported to contain more 2-AP compared to commercial Basmati (Tava and Bocchi 1999). It was reported that dry climate might be a factor for variations in 2-AP content of Khao Dawk Mali 105 (Jasmine rice) grown in different regions of Thailand (Yoshihashi and others 2004). Uncooked brown Jasmine rice (Khao Dawk Mali 105) was reported to have 2-AP as a key aroma compound similar to cooked Jasmine rice (Mahatheeranont and others 2001). Methods for quantification of 2-AP have been extensively reported and revised (Tanchotikul and Hsieh 1991; Bergman and others 2000; Sriseadka and others 2006).

The most important compounds identified in rice include alkanals, 2-pentylfuran, 2-acetyl-pyrroline, and 2-phenylethanol. Non aromatic rice has a higher amount of n-hexanal, other alkanals, and 2-pentylfuran than that of aromatic rice. Basmati rice had the highest amount of 2-phenylethanol and the lowest content of n-hexanal and exhibited the highest popcorn-like aroma (Widjaja and others 1996).

Yang (2007) studied the volatile profiles of cooked black rice and found that 31 out of 58 volatile compounds were odor-active compounds. Of these 31 compounds, hexanal, nonanal, 2-pentylfuran and 2-acetyl-1-pyrroline were present in high concentrations. 2-AP and guaiacol significantly contributed to the unique character of black rice. In another study by Yang (2007), volatile compounds emanating from pigmented rice samples (black and red) were compared to cooked white rice. Most of volatile compounds were shared in common among the three types

of rice, but only nine and two distinct volatile compounds were exclusively found in black rice and red rice, respectively.

The odorous compound 2-acetyl-1-pyrroline was found not only in the aromatic rice but also in cooked roasted beef and crusts of wheat and rye bread (Schieberle and Grosch 1985), wetted ground pearl millet (*Pennisetum americanum*) but with undesirable “mousy” notes (Seitz and others 1993), popcorn (Schieberle 1991), pandan leaves (*Pandanus amaryllifolius*) (Laksanalamai and Ilangantileke 1993), cooked sweet corn products (Buttery and others 1994). Synthesis methods of 2-AP by chemical reactions (Dekimpe and others 1993; Hofmann and Schieberle 1998; Harrison and Dake 2005) and by microbial cultures such as fungal strains and yeast strains (Rungsardthong and Noomhoom 2005) were reported. Another study demonstrated that proline was the nitrogen precursor of 2-acetyl-1-pyrroline (Yoshihashi and others 2002).

Since 2-AP is known to be unstable when stored, a great loss in aroma quality can occur in for aromatic rice. Methods to preserve the 2-AP content such as encapsulation using spray drying (Apintanapong and Noomhorm 2003), using low temperature drying (Wongpornchai and others 2004), and storage and handling at low temperature (Yoshihashi and others 2005) were reported.

Rice lipids are known to occur in both free and starch bound forms. Free lipids adhered on the surface of starch granules are easily extracted by solvent such as ether; whereas, bound lipids located inside the granules. The surface lipids are located in the bran streak that remains on the surface of the milled rice. Degradation of surface lipids in milled rice is related to lipase hydrolysis and subsequent oxidation, which produce off-flavors. The oxidative reactions results in the formation of monohydroperoxides which subsequently break down into volatiles including aldehydes, ketones, alcohols, furanones, acids, lactones and hydrocarbons (Grosch and

Wurzenberger 1983). Off-flavor development depends on temperature, time and exposure to air or oxygen, and the degree of milling (Ohta and others 1990).

Total surface lipids of commercially milled rice were simply reduced by 60-80 % after washing with deionized water with constant stirring (Monsoor and Proctor 2002). In addition, the total surface free fatty acids also decreased by more than 50 % compared to the control. Rice under storage for 7 days after washing exhibited a slight increase of free fatty acid and conjugated diene compared to the control.

The volatiles development in raw milled rice after 50 days of storage were studied (Lam and Proctor 2003). They found that partially milled rice contained more volatiles than fully milled rice. This was because the free fatty acid of residual bran are released by lipase hydrolysis or oxidized directly. Octanal, 2-nonenal, and hexanal which were the products of major fatty acids, i.e. oleic and linoleic acids, increased significantly. Octanal with a fatty character note and 2-nonenal with a rancid character note contributed the most to milled rice odor. A Later study conducted by this group found that broken milled rice resulted in greater exposure than head rice and; thus, it is more susceptible to greater off-flavor development (Monsoor and Proctor 2004).

Parboiled rice is the precooked rice processed by soaking, draining, steaming and drying (Ramesh and others 2000). Kato and others (1983) reported that parboiling significantly increased phenolic acid levels by about three times compared to the control. Also, the volatile components such as pentanal, hexanal, (E)-2-alkenals, (E,E)-2,4-decadienal, and 2 pentylfuran were increased while 1-pentanol, 1-hexanol, and 1-heptanol were decreased (Kato and others 1983).

Flavor analysis by gas chromatography-olfactometry (GC-O)

One key objective of flavor research is to identify the potent aroma components of a food. The methods involved in characterization of key aroma compounds including isolation and separation using different techniques, are merely screening methods that suggest key aroma components. These must be followed by sensory evaluation as to qualify and quantify the derived data (Reineccius 2000).

Preliminary isolation techniques are essential to flavor analysis including solvent extraction, headspace analysis, supercritical fluid extraction (SFE), solid phase extraction (SPE), solid phase microextraction (SPME) (Sides and others 2000). Solvent extraction, especially simultaneous distillation/extraction (SDE) using the Likens-Nickersons method, is a classical method that employs reduced pressure in order to reduce boiling point of flavor components and minimize thermally induced artifacts. The original headspace analysis method includes static recovery and a dynamic procedure (purge and trap). The latter method such as direct thermal desorption with cryogenic trapping allows this technique to be used for a wide array of compounds. The newer automated system called the short-path thermal desorber provides improved reproducibility. SFE is the extraction using a supercritical fluid, substance above its critical temperature and pressure. The temperature and pressure are optimized to control the solvating power and organic modifiers are added to selectively fractionate a sample. In SPE, aroma components recovered by solvent extraction or distillation are passed through a stationary phase cartridge. However, SPE poses many problems such low recoveries, carry over and high blank values and that make this method suitable for semi-volatile aroma compounds. Solid phase microextraction is a solvent-free, rapid sampling, low cost method but is easy to operate

with high sensitivity. Basically SPME fiber is a fused silica coated with an adsorbent and immersed directly into an aqueous sample or into the headspace above a liquid or solid sample.

Gas-chromatography-mass spectrometry (GC-MS) is generally used to selectively separate and characterize compounds based on their known spectral and chromatographic properties. Because key aroma compounds are usually volatiles or semi-volatiles, sample extraction followed by GC-MS is the most common analytical strategy for flavor analysis. With the aid of olfactometry, determination of odor-active compounds which contribute to the characteristics sensory properties can be obtained in that odorless compounds are eliminated from consideration. It is well known that many key aroma compounds occur at very low concentrations; i.e. at low odor thresholds indicating that the peak profile as seen from GC does not necessarily reflect the aroma profile of the food. Only a few (< 10) volatile compounds among hundreds of detected volatiles by GC are potent enough to define the sample's flavor profile (Marsili 2007).

One such approach proven to be useful to indicate the potency of odorants was the concept of odor activity value (OAV) which was defined as the concentration of an odorant relative to its human threshold (Acree and others 1984). OAV values less than one is unlikely detectable by humans and those with the higher values are assumed to be the most important. Based on the psychophysical laws, Steven's laws, which express a logarithmic or power relationship between the odor intensity and the physical concentration, OAV values do not correlate linearly with perceived intensity value and do not predict the odor intensity of compounds in combination (Mayol and Acree 2001).

The technique of gas chromatography-olfactometry (GC-O) provides sensory insights to chromatography analysis. GC-O methods are classified into four categories; dilution analysis

methods, detection frequency methods, posterior intensity methods, and time-intensity methods (van Ruth 2001). Generally, dilution techniques and time-intensity measurements are the two main GC-O methods.

Dilution analysis method is based on stepwise dilution to threshold including CHARM analysis (combined hedonic response measurement) (Acree and others 1984) and AEDA analysis (aroma extraction dilution analysis) (Grosch 1993). In both procedures, an extract is diluted, usually as a series of 1:1 or 1:2 dilutions (Grosch 1993) or 1:2 or 1:3 dilutions (van Ruth 2001), and each dilution is sniffed until no significant odor is detected. This may require several injections until the desired dilution at which odorous regions of aroma extract are no longer detected. Both methods are based on the odor detection threshold. For the AEDA, the dilution value is expressed as a flavor dilution (FD) factor which is proportional to the OAV of the compound in air. The primary difference between the two methods is that CHARM analysis measures the dilution value over the entire time of the eluting peak, whereas AEDA determines the maximum dilution value detected.

Detection frequency method employs a group of assessors rather than one or two (Linssen and others 1993). The intensity of a compound is calculated from the numbers of assessors who detect an odor active compound at the same time which poses a drawback for not being based on real intensities of compounds. Posterior intensity methods produce estimates of perceived intensity which are recorded after the peak has eluted from the column.

Time-intensity methods basically produce estimates of perceived intensity recorded and described by trained panelists simultaneously with the eluting peak using a 16-point scale time-intensity device (McDaniel and others 1990). The methods are based on magnitude estimation

of the odor intensity. This technique provides an Osmogram-an FID-style aromagram. Several trained panelists are required to perform this method.

Drawbacks of GC-O are related to the fact that it uses a human as a detector to sniff the effluent. Safety concerns are of importance to the sniffers who must be pre-screened for sensitivity and specific anosmia. Care must be taken to reduce experimental error and variation including sample preparation, room and sample temperature, time of day, duration of analysis, repetition of analysis, repeated standardization of sniffers and use of a standard lexicon and test conditions (Friedrich and Acree 2000).

Relating descriptive sensory data to volatiles compounds

Flavor cannot be measured directly by instruments as it is an interaction of consumer and product (Sydow 1971). Therefore, mathematical approaches in relating sensory data and chemical/physical data as shown in Table 2.1 are involved. Univariate statistical methods such simple linear regression might be less useful than multivariate methods in terms of interpretation of the sensory significance of complex volatile data. Correlation analysis is the most common use to determine whether a relationship exists. The correlation coefficient (r) generated lies between -1 to 1 indicating the degree of relationships (negative or positive, respectively) and significance of the relationships, that is, -1 or +1 is a perfect linear relationship between the independent variable (X) and the dependent variable (Y).

Multivariate statistical techniques illustrate pattern in volatiles that correspond with specific sensory aroma profile such as in wine flavor (Noble and Ebeler 2002). These techniques include correlation analysis, principal component analysis (PCA), cluster analysis, regression analysis, principal component regression (PCR), partial least square (PLS) regression and

discriminant analysis (Meilgaard and others 2007), canonical correlation and canonical variate (Piggott 1990). Reduction of the dimensionality of data set (linear combinations of independent variables that contain the maximum amounts of information) is the goal for multivariate methods. As a consequence, classification of individual samples based on their degree of similarity and prediction of properties are achieved (Marsili 2007).

The two most commonly used are regression/redundancy modeling and partial least square (PLS) modeling which poses some concerned features as shown in Table 2.2 (Williams 1994). Both multiple linear regression and partial least square regression rely on the linear nature of variable. Multiple linear regression in sensory analysis has many applications including relating sensory rating to instrument readings, predicting consumer acceptance, relating product characteristic to acceptance, relating process characteristics to acceptance and so on (Korth 1982). Simple transformations of data which are nonlinear in nature using squares and logarithms strengthen the relationship between the non-sensory score and sensory score (Korth 1982). It is advisable that any developed regression model is evaluated its predictive ability. This is done by separating the observations into two distinct sets, a training set which is used to build up the model, and a test set which is used to evaluate the model (Piggott 1990).

Sensory properties in tea aromas were related with gas chromatography peak using multiple linear regression (Togari and others 1995). In this study, seven descriptive notes were successfully correlated with the gas chromatography profile that was logarithmic transformed as indicated by the improve coefficient of determination. They found that linalool and jasmine lactone, 2-phenylethanol and jasmine lactone, and 2-phenylethanol highly contributed to the prediction of fresh floral, sweet floral, and sweet fruity, respectively.

Prediction models for tomato flavor were developed using multiple linear regression when sensory descriptors were regressed against the volatile and nonvolatile content (Tandon and others 2003). Aromatic descriptors were influenced by both volatile and nonvolatile components but volatiles provided more consistent relationships.

Other modeling such as generalized procrustes analysis (GPA) is used to match the two data sets that have been analyzed separately by PCA such as instrumental and sensory data. The consensus space derived from the average of the two spaces, is the explained variance needed to further conduct permutation tests to compare with the variance explained by random permutations (Noble and Ebeler 2002).

It is worth to note that no single method is completely perfect for all circumstances since the methods all have strengths and weaknesses. However, one thing that the methods share in common is the fact that they can only model from the data provided. As a consequence, to prevent the misleading caused by the model, the completion of sample set is necessary (Piggott 1990).

Table-2.1 Mathematical methods in sensory data and chemical/physical relationships (Williams and others 1988)

1.	Linear regression and correlation
2.	Non-linear regression using specific model
3.	Multiple regression using raw or transformed data
4.	Canonical variate analysis on defined groups
5.	Correlation and regression using principal axes, following principal component or canonical variate analyses
6.	Canonical correlation and regression
7.	Partial least squares regression
8.	Procrustes matching
9.	Redundancy modeling
10.	LISREL, maximum likelihood modeling

Table-2.2 Pros and cons of regression/redundancy modeling and partial least square regression
(Williams 1994)

Model	Pros	Cons
Regression/redundancy	Good predictability	Highly weighted to variables showing little overall variation With large numbers of variables, can produce unstable solutions
Partial least square (PLS)	Compromise between predictability and ability to summarize data	Like PCA regression, may overemphasize components accounting for large variations but of little sensory significance

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CHAPTER 3
FLAVOR LEXICON FOR SENSORY DESCRIPTIVE
PROFILING OF RICE¹

¹Limpawattana, M. and Shewfelt, R.L. To be submitted to Food Quality and Preference

ABSTRACT

A descriptive lexicon was developed and expanded to characterize the flavor of a broad spectrum of rice (n=36) using descriptive analysis method. Rice samples differed in terms of forms, types and specialty were used in this study. A trained descriptive panel (n=8) participated in terminology generation and validation including reference assignment. Univariate and multivariate analysis were used to determine the difference across the samples. Twenty-four attributes were required to fully describe the flavor. Eighteen descriptive terms were significantly present in most rice samples. Some descriptors are unique characteristic of specific rice. This lexicon will facilitate targeting the characteristic notes important to rice processors as well as producers.

Introduction

Rice plays a fundamental role in world food as it is the primary food source of more than half of the world population. In 2006, about 638 million tonnes of paddy rice was produced (FAO, 2007). Production is geographically concentrated in Western and Eastern Asia with more than 90 percent of world output. The United States is the second most important non-Asian producer of rice, exporting about 3.3 million tonnes. International price of rice have been remarkably increased for the high quality rice which is strongly demanded in the world trade (FAO, 2007).

Rice flavor is a significant factor in determining quality and consumer acceptability as exemplified by aromatic rice which is highly favored and commands a high market price. To most people rice is a rather bland food and minor changes in sensory properties, especially aroma, can make rice and rice products unacceptable to consumers. The flavor of rice has been studied by both instrumental and sensory methods. More than 300 volatile aroma components have been identified in rice (Jezussek, Juliano, & Schieberle, 2002; Maga, 1984; Widjaja, Craske, & Wootton, 1996). Among these, 2-acetyl-1-pyrroline (2-AP) was reported as the characteristic aroma compound yielding a popcorn-like aroma that is found predominantly in aromatic rice (Buttery, Ling, & Juliano, 1982). Sensory analysis methods have been performed to assess rice flavor characteristics; primarily using descriptive analysis methods (Champagne, Bett-Garber, McClung, & Bergman, 2004; Paule & Powers, 1989; Suwansri, Meullenet, Hankins, & Griffin, 2002; Yau & Huang, 1996; Yau & Liu, 1999). However, these studies did featured only a limited lexicon for characterizing specific flavors or the range of flavor types was limited.

Descriptive sensory analysis can characterize products both qualitatively and quantitatively (Munoz & Civille, 1998). Lexicons for describing characteristics of product have been developed for products such as catfish (Johnsen, Civille, & Vercellotti, 1987), peanut (Johnsen, Civille, Vercellotti, Sanders, & Dus, 1988), bread (Chang & Chambers, 1992; Lotong, Chambers, & Chambers, 2000), cheddar cheese (Drake, McIngvale, Gerard, Cadwallader, & Civille, 2001), French cheese (Retiveau, Chambers, & Esteve, 2005), soymilk (N'Kouka, Klein, & Lee, 2004), carbonated beverages (Kappes, Schmidt, & Lee, 2006), and green tea (Lee & Chambers, 2007).

Currently, several U.S. rice breeding programs are focusing on developing new rice cultivars with greater consumer acceptability. Consequently, rapid methods focused on rice flavor quality are needed to assist rice breeders in making progeny selections. As a result, sensory evaluation of rice should be a critical component in those programs. This study was undertaken to establish a descriptive lexicon with reference standards for describing the flavor properties of a broad spectrum of rice, and use the developed lexicon to characterize which sensory attributes are most important in rice flavor quality.

2. Materials and method

2.1 Samples

A broad spectrum of 36 domestic and imported rice samples (Table 3.1) represented the difference in forms, types and specialty (USA Rice Federation, 2007) were purchased from either an Asian supermarket in Atlanta, Georgia or directly from rice processors. Of these 36 samples, 15 were aromatic milled white imported Jasmine, three were aromatic milled white domestic Jasmine, three were aromatic milled white imported Basmati, one was aromatic milled

white domestic Basmati, two were domestic long grains, two were domestic medium grains, two were sweet imported rice, two were pigmented imported rice, two were non-aromatic brown rice, one was aromatic brown rice, two were parboiled non aromatic rice, and one was parboiled aromatic imported Basmati rice. Rice samples especially all imported Jasmine rice from Thailand were indicated as “New Crop 2006” on the package which meant that the rice was planted and harvested in 2005 and exported to overseas in the marketing year of 2006. All rice samples were individually weighed into 1.5-kg portions, stored in a zippered lock freezer bag (Ziploc Brand, S.C. Johnson & Son Inc., Racine, WI, U.S.A.) and placed in the freezer at -20 °C for further testing. Each sample was assigned a random three-digit number.

2.2 Panelists

The panel consisted of eight members who were recruited from graduate students and staff of Department of Food Science and Technology, University of Georgia. Panelists were pre-screened by questionnaire according to Meilgaard, Civille, & Carr (2007) on the basis of their health, food habits, availability and willingness to participate into the project. Results from the questionnaire indicated that they were free of food allergies and most importantly were free of respiratory problems. The panel composed of two males and six females, age ranging from 22 to 54 years, with prior experience in descriptive sensory analysis. The panel conducted and passed screening tests which primarily focused on basic taste, odor recognition, descriptive ability and ranking ability (Issanchou, Lesschaeve, & Koster, 1995). The principles and concepts of descriptive analysis of rice flavor using the Spectrum™ method (Meilgaard et al., 2007) were introduced to the panel. The Spectrum™ method is based on universal intensity scales (Munoz & Civille, 1998). Consequently, twelve-1.5 hours sessions were devoted to train the panelists. Of the twelve training sessions, four sessions were for actual testing of rice samples. During the

initial training sessions, the panelists generated the descriptive terms for aromas, tastes and oral feeling factors (Caul, 1957) using the 18 rice samples from the rice array as representatives for forms, types and specialty samples. At each session during the training period, panelists defined the developed language and established references (Giboreau et al., 2007; Rainey, 1986) for the terms generated. After panel agreement, standard references were chosen based on those previously published with the same specific attributes. The intensity of each reference was marked on a 150-mm unstructured line scale (Malundo, Shewfelt, & Scott, 1995). Since evaluation took place between the Fall 2006 and the Spring 2007, two new panelists (one male and one female within the same age range as above) were included in the group, while two male panelists had withdrawn. The Spring evaluation was done by eight panelists of whom six had participated in the Fall descriptive analysis test over the period of six-1.5 hour sessions. Two new panelists who had prior experience to descriptive sensory analysis and passed all screening procedures as mentioned above, were individually trained with the experimenter using the same set of standards and references that were previously established and able to integrate with other trained panelists. Panelists received premium chocolate treats as rewards for their participation in each session.

2.3 Preparation of cooked samples

Rice samples were retrieved from cold storage and allowed to equilibrate to room temperature for approximately 12 h before cooking. Micro-computerized rice cookers (Sanyo model ECJ-D100S, Sanyo, Japan) were used to cook all the rice samples. All the rice samples except pigmented rice were prepared using 1.5: 1 water: rice ratio or 300 g of rice in 450 ml of deionized water. The water: rice ratio for pigmented rice was 1:1. Cooking time among samples varied between 38-45 min based on types of rice and automatic operation programmed by the

cookers. The sample remained in the cooker until it was completely cooked as indicated by the light was switched to “warm” mode. The cooking chamber was coated with a non-stick Teflon minimizing any scorching of rice at the bottom.

2.4 Sample presentation

Without delay, the samples were transferred into preheated (75°C) 180-mL glass custard cups which were then sleeved with Styrofoam cups and covered with three-digit coded glass petridishes. Because temperature of cooked rice during an immediate “warm mode” was approximately 80 °C, panelists were informed to complete their evaluation before the temperature dropped to 60 °C as monitored by individual hand-held digital thermometers.

2.5 Sample evaluation

Thirty-three out of 36 rice samples were used to fully characterize the flavor of cooked rice after the attribute lexicon was developed. The three samples only generated a few descriptors; therefore, they were excluded from evaluation. Of these 33 samples, 17 rice samples were used to validate the lexicon during the Fall evaluation while 16 samples previously used to generate the lexicon during the Fall 2006 were reevaluated during Spring testing. The cooked rice samples were presented one at a time in random order across treatments to panelists in partitioned booths under fluorescent light and controlled room temperature. Panelists scored all attributes on a 150-mm unstructured line scale. References were introduced corresponding to the point on the line scales. Panelists recorded the scores for each attribute on paper ballots accordingly. Unsalted top crackers and water were provided for panelists to rinse their palates between samples. Each sample was evaluated in duplicated on separate testing days.

2.6 Statistical analysis

Analysis of variance (ANOVA) with Fischer's least significant difference (LSD) was conducted on Statistical Analysis Systems program (SAS) version 9.13 (SAS Inst., Cary, N.C., U.S.A) for each attribute. A two-way analysis of variance with samples and panelists as main effect was conducted to determine whether there were significant differences in the mean scores of the rice samples. The interaction between panelists and sample was also evaluated to ensure that it was not significant. If the sample by panelist interaction was significant in a given attribute, an adjusted F-test (mixed model ANOVA) was performed. Since replicates were randomized within the same session, so that no replicate effect is needed in the model (Lea, Rød-botten, & Næ, 1997). Significance was established at $p < 0.05$. For multivariate analyses, correlation analysis was conducted to determine the relationship among the significant attributes and principal component analysis (PCA) by the covariance matrix (Borgognone, Bussi, & Hough, 2001) and factor analysis with Varimax rotation were conducted to determine whether the attribute scores for rice sample could be explained by a smaller number of principal components that are linear combination of the initial variable using SAS. The visual demonstration of which terms were related and described the samples were provided by PCA biplots.

3. Results and Discussion

With the aid of the aroma lexicon previously developed for rice (Goodwin et al., 1996), grain (Civille & Lyon, 1996) and fundamental tastes (Meilgaard et al., 2007), a preliminary rice lexicon was generated and expanded (Table 3.2). These terms were used as a starting basis for the descriptive analysis panel. Because a broad spectrum of samples was used, a vast difference

in flavors among samples was found. Similar observations were noted when a French cheese lexicon was developed (Retiveau et al., 2005). Following the preliminary language, the refined 24 attributes selected by consensus were developed to fully characterize the sensory properties of the types of rice studied (Table 3.3). Among the attributes, three (smoky, sulfury and waxy) were found in less than 15 of the 33 samples (Table 3.4). It should be noted that terms that are not observed frequently in samples are yet relevant to the sensory language of the samples (Drake et al., 2001). Several of the terms have been used by previous researchers to describe rice (Meullenet et al., 2001; Suwansri et al., 2002). Terms common to both this research and those of Meullenet and coworkers (2001) include starchy, cooked grain, nutty, sulfury, floral, dairy, sweet aromatics, astringent and the basic tastes. Some new terms such as smoky, barny, buttery, rancid, waxy, earthy and green were included to better describe the flavor of brown, pigmented and parboiled rice (Table 3.4). However, some reference standards are different based on availability but are still sufficiently clear and distinct to identify the characteristic of those terms (Civille & Lyon, 1996). Some of the terms probably represent similar concepts but are described somewhat differently, such as wet cardboard and rancid, heated oil and waxy. Successful identification of aroma is partly due to the substances that panelists commonly encountered (Cain, 1979).

ANOVA performed on 24 attributes revealed that sweet aromatics, sweet, bitter, salty, astringent and metallic attributes were not significantly different between samples. In a descriptive panel analysis, it is common to detect a panelist effect since they used the scale differently for evaluation (N'Kouka et al., 2004). Panelist by sample interaction was used as the error term to calculate an adjusted F-test that considers panelist as a random effect in the mixed-

model ANOVA. Those attributes that were not significantly different among samples were kept in the vocabulary for further evaluation but not included in the PCA.

Correlation analysis of 18 significant attributes demonstrated that no attribute was highly correlated (i.e. > 0.80) with any other as shown in Table 3.5. Popcorn and starchy were positively correlated with buttery while smoky was negatively correlated. Cooked grain aroma was positively correlated with grain, nutty, buttery but negatively correlated with barny. Smoky, hay-like and waxy were positively correlated with earthy, while popcorn was negatively correlated. Nutty was positively correlated with buttery and waxy. Buttery was positively correlated with dairy while hay-like, barny and earthy were negatively correlated. The terms presented the highest significant correlation were nutty and waxy and also hay-like and earthy ($r = 0.58$). It could be explained that in some samples, these terms were present together and rated similarly as seen from Table 3.4. However, one imported Jasmine rice (JU) was the highest in nutty aroma but none in waxy note while the other imported Jasmine rice (JB) had moderate waxy aroma but lacked nutty aroma. It was also observed that the presence of hay-like note and earthy note demonstrated in the same fashion. For example, two imported Jasmine (JF and JR) and one non-aromatic brown rice (B2) exhibited high hay-like notes but lacked earthy note while one imported Jasmine rice (JU) was rated in moderate earthy note but lack the hay-like attribute.

Attributes that were found to be not significantly different were excluded from the data set prior to PCA. Furthermore, factor analysis with Varimax rotation was applied to the covariance matrix of rice means in order to clarify group individual terms. The first six factors on rice flavor explained 74.7 % of the variation. Each factor had eigenvalues greater than one and attributes with positive and/or negative factor loadings greater than 0.30 (Table 3.6). The principal component analysis biplots are presented in Figure 3.1 and 3.2 for aroma attributes

across samples. PCA visual applied to 18 aroma attributes of 33 rice samples, indicated that three components accounted for only 54.2 % of the variation in the data. PC1 was found to account for 28.1 % of variation in the data, while PC2 and PC3 were found to account for 16.7 % and 9.5 % of the variation, respectively. Factor 1 included earthy, hay-like, smoky, barny, woody inversely with dairy. Cooked grain, nutty, grain, buttery, starchy and waxy were positive loadings on factor 2 while green and sulfury were positively grouped on factor 3. The sample score of one imported sample (JE) exhibited a higher popcorn attribute than other Jasmine rice samples. Most of Jasmine rice samples were primarily characterized as having buttery, corn, dairy, grain, starchy, cooked grain, and nutty attributes. Imported Basmati samples (BV and BA) were characterized as having predominant hay-like and earthy attributes. Parboiled rice (PB and PM) and Sweet rice (TS) samples were characterized as having barny and woody attributes. Brown rice samples (B1) and (B2) were perceived as having more hay-like and earthy attributes compared to B3. The third component provided more details in that the green and sulfury aroma loaded most highly on PC3. Rice samples (MK, JD, JR, JF) loaded similarly in the area of green aroma in contrast to one imported Jasmine sample (JN).

It was evident that PCA was not effective for reduction of attributes that could adequately describe the rice samples. This finding was not surprising since the correlations were very weak indicating the presence attribute orthogonality in the original space, making it difficult to reduce the space to a smaller dimension (Khattree & Naik, 2000). Many attributes were presented independently from other attributes as indicated above. The same phenomenon as such has been found in lexicon development of French cheeses (Retiveau et al., 2005), soymilk (Chambers, Jenkins, & McGuire, 2006) and green tea (Lee & Chambers, 2007) where a wide range of samples was used ($n > 30$). Although, it was recommended that reduction of the number of

attributes would be beneficial (N'Kouka et al., 2004) but it was not the case in our study. Further elimination of attributes to get a smaller set of component would reduce the variability explained and thus it seems that the 18 significant attributes found are necessarily maintained to fully describe the flavor of a wide range of rice as such since some specific rice samples had unique characteristics.

4. Conclusion

This study developed and expanded a lexicon with references containing 24 attributes that can be used to characterize a wide range of rice samples. This lexicon covered a wider range of rice samples in the previous studies (Meullenet et al., 2001; Suwansri et al., 2002). The use of universal intensity scales also facilitates the addition of new terms that will allow for the addition new rice types in the future. Linking sensory flavor descriptors to chemical component found in rice is another area of research in our laboratory. Use of chemical references that exhibit the similar characteristic of the notes with relative to the intensity of food references would be applicable since all food references may not be available worldwide.

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Table 3.1 Rice samples used in the study

Product	Origin	Brand	Code
Jasmine	Imported/Thailand	Royal Umbrella	JU
	Imported/Thailand	Golden elephant	JE
	Imported/Thailand	Fook Cheung Pai	JF
	Imported/Thailand	Twin elephant	JW
	Imported/Thailand	Asian best	JB
	Imported/Thailand	Eagle	JG
	Imported/Thailand	Three ladies	JL
	Imported/Thailand	Sunlee	JS
	Imported/Thailand	Dynasty	JY
	Imported/Thailand	Golden camel	JC
	Imported/Thailand	Asian Taste	JT
	Imported/Thailand	Goya	JO
	Imported/Thailand	Vera	JV
	Imported/Thailand	La Triguena	JR
	Imported/Thailand	Imperial Dragon	JI
	Domestic/USA	Lundberg	JN
	Domestic/USA	Martin	JM
Della	Domestic/USA	Campbell	JD
Basmati	Imported/Pakistan	Arya	BA
	Imported/India	Vigo	BV
	Imported/India	Mahatma	BM
	Domestic/USA	Texmati	TX
Sweet rice	Imported/Thailand	Three rings	TS
	Domestic/USA	Jung Il Pum	KS
Pigmented	Imported/Thailand	Asian Taste	TK
	Imported/China	Assi	KK
Calrose	Domestic/USA	Botan	MC
Kokuho	Domestic/USA	Kokuho rose	MK
Long grain	Domestic/USA	Mahatma	LM
	Domestic/USA	Great Value	LV
Brown rice	Domestic/USA	Kroger's	B1
	Domestic/USA	Mahatma	B2
Parboiled	Imported Jasmine/Thailand	Asian Taste	B3
	Imported Basmati/Pakistan	Nobano	PB
	Domestic/US	Mahatma Gold	PM
	Domestic/US	Uncle Ben	PU

Table 3.2 Preliminary descriptors

Aroma		Basic tastes	Oral feeling factors
Acidic	Nutty	Sweet	Astringent
Alfalfa	Oily	Salty	Metallic
Animal-like	Onion	Bitter	
Balsamic	Popcorn		
Barny	Pyridine		
Burning	Rancid		
Buttery	Raw grain		
Caramel	Red bean		
Chalk	Rice hull		
Cooked clothes	Rotten/spoiled/rank		
Cooked grain	Rotten egg		
Cooked raw wheat	Rubbery		
Corn	Sea weed (kelp)		
Dairy	Sesame seed		
Dust-like	Sewer		
Earthy	Soapy		
Fatty	Soil		
Floral	Starchy		
Formalin	Sulfury		
Fried	Sweetpotato		
Fruity	Sweaty		
Grass-like	Water-like		
Green	Waxy		
Hay-like	Woody		
Meaty	Urine		
Medicine	Used clothes		
Musty			
New unfolded clothes			

Table 3.3 Sensory descriptors, definitions and references for cooked rice flavor evaluation

Attributes	Definition	References*
Popcorn	Aromatics reminiscent of popcorns	Orville Redenbacher's Gourmet [®] popping corn
Starchy	Aromatics associated with the starch of a particular grain source	Bob's Red milled rice flour : water (1:1)
Woody	Aromatics associated with dry cut fresh wood	Toothpicks
Smoky	Aromatics associated with any type of smoke flavor	Colgin Liquid smoking flavor
Cooked-grain	Aromatics associated with cooked grains	Nabisco Cream of wheat
Grain	Aromatics associated with overall character impression of grain such as corns, wheat, and oats	Grain mixture**
Sulfury	Aromatics associated with sulfurous compound	Hard boiled egg
Corn	Aromatics reminiscent of canned yellow cream-style corn	Libby's cream-style corn
Nutty	Aromatics associated with roasted nuts	Planters roasted peanut
Floral	Aromatics associated with flowers	Jasmine tea, jasmine scent
Dairy	Aromatics reminiscent of pasteurized cow's milk	2% pasteurized milk
Hay-like	A dry, dusty, slightly brown aroma	Kaytee natural Timothy hay
Barny	Aromatics reminiscent of barnyard and stocks (manure, urine, moldy, hay, feed, etc.)	Kroger white pepper
Buttery	Aromatics associated with natural fresh butter	Land O' Lake butter
Green	Aromatics (slightly sweet) associated with cut grass or green vegetable	Sunny creek organic alfalfa sprouts
Rancid	Aromatics associated with oxidized fats and oils	Canola oil aged 14 d at 80°C
Waxy	Aromatics associated with medium chain fatty acids	Candle wax
Earthy	Aromatics reminiscent of decaying vegetative matters and damp black soil	Sliced mushrooms
Sweet-aromatics	Aromatics associated with sweet tastes	Bordeaux cookies Pepperidge Farm
Sweet	Basic taste sensation elicited by sugar	2 % and 5 % sucrose solution
Salty	Basic taste sensation elicited by salts	0.2 % and 0.35 % NaCl solution
Bitter	Basic taste sensation elicited by caffeine	0.05 % and 0.08 % Caffeine solution
Astringent	Puckering or tingling sensation elicited by grape juice	Welch's Grape juice
Metallic	Chemical feeling factor stimulated on the tongue and teeth by metal	1 iron tablet/L

*Adapted from Civille & Lyon (1996), Goodwin et al, (1996), and Meilgaard et al., (2007); ** 2 tablespoons each for rice flour, yellow corn

meal, white flour and 1 tablespoon of oatmeal before grinding

Table 3.4 Mean of descriptive flavor attributes and least significant difference (LSD, $\alpha = 0.05$) (see sample code in Table 3.1)

Sample	Popcorn	Starchy	Woody	Ckdgrain	Smoky	Grain	Sulfury	Corn	Nutty	Floral	Buttery	Dairy
LM	25.19	30.50	12.75	24.56	0.00	10.00	12.31	0.00	8.25	8.75	12.19	7.06
MK	19.88	27.13	12.00	20.50	0.00	11.31	8.63	9.31	7.63	0.00	0.00	7.00
BA	25.13	19.69	22.94	18.13	13.94	7.31	8.25	0.00	9.81	11.81	6.31	0.00
BV	23.31	18.81	14.94	14.75	15.25	8.75	0.00	0.00	9.00	12.25	7.94	0.00
TX	36.88	19.19	19.50	21.88	9.69	11.75	0.00	15.63	0.00	8.50	9.00	0.00
TK	13.13	13.63	13.88	10.25	15.94	10.19	0.00	8.31	0.00	0.00	0.00	0.00
KK	11.44	15.38	13.56	15.50	12.75	6.44	9.00	21.13	8.25	11.94	0.00	0.00
KS	13.63	30.63	11.38	35.06	5.81	25.00	0.00	8.50	8.38	8.69	7.31	0.00
TS	19.75	15.00	18.19	10.69	9.69	9.44	0.00	9.19	0.00	6.63	0.00	7.19
B1	25.13	15.19	14.63	19.63	8.44	9.31	9.25	16.31	10.19	0.00	13.00	0.00
B2	13.56	8.19	14.00	6.00	7.94	6.94	8.13	9.69	0.00	6.00	0.00	0.00
B3	19.31	15.56	25.25	18.63	10.25	11.94	0.00	8.38	8.06	9.44	9.31	0.00
JM	31.69	29.31	10.38	19.44	0.00	8.50	9.00	15.19	10.13	9.38	13.75	7.63
JD	30.13	18.31	15.50	19.13	0.00	0.00	0.00	10.31	0.00	10.81	7.94	6.13
JN	30.25	17.56	17.75	12.63	0.00	9.38	0.00	9.25	0.00	8.31	8.06	7.13
JU	19.13	22.88	14.50	20.44	0.00	7.44	0.00	14.00	11.19	8.88	10.75	11.56
JW	21.19	22.06	12.06	24.19	0.00	7.38	0.00	9.50	0.00	11.38	10.50	6.88
JS	22.25	29.63	0.00	25.38	0.00	7.06	5.75	9.56	6.38	8.31	13.63	6.06
JY	27.63	28.38	16.88	0.00	0.00	12.00	0.00	9.31	7.63	16.13	9.50	0.00
JR	20.69	20.00	11.75	19.13	0.00	8.31	0.00	9.75	7.94	9.19	8.88	7.88
JV	28.63	26.81	13.31	21.38	0.00	8.69	0.00	12.44	9.56	12.44	13.44	10.56
JF	25.44	0.00	9.38	26.38	0.00	12.06	0.00	12.19	9.13	7.06	13.31	5.81
JC	24.00	20.13	11.88	19.00	0.00	11.69	0.00	13.25	8.44	10.38	10.44	7.19
JT	33.63	21.50	12.88	20.44	0.00	15.50	0.00	18.38	6.94	0.00	11.69	7.19
JL	35.19	26.06	13.38	20.69	0.00	16.56	0.00	19.94	8.44	7.13	9.38	7.88
JE	38.88	17.63	18.00	18.69	0.00	7.19	0.00	13.44	0.00	9.56	0.00	7.00
JB	26.44	14.13	10.69	11.69	7.44	7.06	7.63	11.50	0.00	10.00	6.25	7.63
JI	31.50	16.00	15.38	15.88	0.00	10.56	0.00	13.13	0.00	8.00	8.94	7.63
JO	37.06	18.31	19.06	17.06	0.00	8.19	0.00	11.88	0.00	6.13	9.44	0.00
JG	29.88	13.75	16.13	16.25	0.00	9.63	0.00	12.69	5.88	0.00	8.00	0.00
PM	12.63	7.56	13.06	0.00	9.56	0.00	0.00	0.00	0.00	10.81	0.00	0.00
PU	13.75	17.25	11.75	13.44	0.00	11.81	8.44	9.31	11.13	0.00	0.00	0.00
PB	17.19	8.91	22.38	6.69	11.19	9.31	0.00	0.00	0.00	0.00	0.00	0.00
LSD	10.99	8.99	7.84	8.40	3.84	6.06	4.78	5.87	4.25	3.99	6.75	3.66

Table 3.4 Mean of descriptive flavor attributes and least significant difference (LSD, $\alpha = 0.05$) (continued)

Sample	Hay-like	Barny	Green	Rancid	Waxy	Earthy	Swtarom	Sweet	Salty	Bitter	Astringent	Metallic
LM	8.94	0.00	8.56	0.00	0.00	6.38	0.00	12.25	10.25	11.75	13.31	7.00
MK	9.56	9.19	13.44	9.50	0.00	0.00	12.00	13.94	9.56	5.50	8.88	5.69
BA	15.75	6.81	0.00	0.00	8.44	11.63	6.25	9.38	10.38	14.06	7.94	5.38
BV	8.56	0.00	0.00	12.69	10.44	10.81	7.38	7.19	6.50	10.19	12.63	0.00
TX	8.19	16.25	0.00	0.00	0.00	0.00	8.00	9.13	9.13	9.25	7.00	5.19
TK	19.00	0.00	10.25	0.00	0.00	11.31	17.50	8.38	8.31	13.63	11.88	15.63
KK	19.69	11.75	8.31	0.00	8.31	16.25	8.06	11.19	9.94	9.69	14.00	5.25
KS	7.06	6.63	0.00	14.56	8.63	10.75	15.31	13.94	9.75	13.25	10.38	7.06
TS	9.44	12.44	0.00	13.06	0.00	0.00	9.13	5.69	7.50	10.25	10.25	8.31
B1	9.75	12.00	0.00	11.06	0.00	7.81	7.00	7.19	9.25	18.63	14.19	0.00
B2	10.94	11.81	0.00	12.75	0.00	0.00	7.06	7.56	9.31	10.81	9.69	6.69
B3	12.69	13.63	0.00	25.38	7.69	10.31	11.31	8.81	10.38	13.13	13.88	7.13
JM	0.00	0.00	0.00	11.06	7.31	0.00	11.06	13.13	9.88	12.31	5.50	5.50
JD	14.81	0.00	7.13	7.00	0.00	9.06	13.00	9.13	8.63	8.75	7.81	7.56
JN	9.25	12.56	0.00	8.19	0.00	8.25	9.69	9.31	8.88	6.31	0.00	9.31
JU	0.00	0.00	0.00	0.00	0.00	6.13	11.25	13.31	10.81	11.13	12.25	0.00
JW	9.25	0.00	0.00	0.00	0.00	0.00	13.44	12.94	7.69	13.06	9.88	0.00
JS	9.06	0.00	10.50	11.13	7.06	5.94	14.50	16.31	9.25	9.69	10.56	0.00
JY	12.19	7.94	11.69	10.00	7.00	7.81	12.56	10.81	11.00	12.06	9.94	4.69
JR	10.13	0.00	9.38	7.50	0.00	0.00	12.31	9.44	9.06	11.31	15.38	0.00
JV	8.69	0.00	10.75	10.06	7.31	0.00	13.38	15.06	8.13	16.94	8.31	0.00
JF	13.50	0.00	9.56	0.00	6.88	0.00	14.56	14.19	10.88	13.06	8.94	0.00
JC	8.25	8.94	0.00	12.38	7.44	7.75	10.50	14.19	13.44	13.44	14.94	0.00
JT	0.00	0.00	8.69	0.00	0.00	0.00	9.81	13.13	10.44	8.06	7.81	8.31
JL	8.56	0.00	0.00	9.88	0.00	0.00	12.13	12.63	8.81	5.31	10.38	9.50
JE	0.00	0.00	0.00	0.00	0.00	0.00	12.88	11.63	8.13	6.50	11.38	7.38
JB	0.00	11.63	7.56	0.00	5.56	0.00	9.38	10.31	9.63	8.69	9.50	9.19
JI	7.63	7.63	0.00	0.00	0.00	0.00	0.00	10.00	8.81	6.13	9.75	6.81
JO	9.50	12.81	0.00	0.00	0.00	0.00	6.63	11.63	8.94	5.56	9.88	5.94
JG	12.56	15.13	9.38	0.00	5.63	8.00	9.13	10.75	8.81	5.25	13.25	0.00
PM	9.81	9.44	0.00	7.94	0.00	0.00	15.63	7.94	12.31	6.63	11.88	0.00
PU	17.50	9.56	7.81	14.31	8.44	15.06	11.69	10.25	10.50	15.00	14.94	4.44
PB	8.00	13.13	9.00	9.81	0.00	8.50	9.44	11.19	11.81	7.69	7.63	12.75
LSD	6.79	5.01	5.34	5.83	3.40	5.29	7.66	5.75	5.42	7.55	8.04	3.94

Table 3.5 Correlation analysis of 18 significant attributes^a

	P	St	Wo	Ckg	Sm	Gr	Su	Co	Nu	Fl	Bu	Da	HL	Ba	Gn	Ra	Wx	Ea
Popcorn (P)	1.00																	
Starchy (St)	0.22	1.00																
Woody (Wo)	0.16	-0.27	1.00															
Cookedgrain (Ckg)	0.22	<i>0.44</i>	-0.32	1.00														
Smoky (Sm)	-0.46	<i>-0.39</i>	<i>0.39</i>	-0.33	1.00													
Grain (Gr)	0.04	0.34	-0.03	0.46	-0.10	1.00												
Sulfury (Su)	-0.23	0.18	-0.26	0.05	0.09	-0.13	1.00											
Corn (Co)	<i>0.37</i>	0.10	-0.21	0.28	<i>-0.35</i>	0.23	-0.05	1.00										
Nutty (Nu)	-0.13	<i>0.40</i>	-0.24	<i>0.40</i>	-0.16	<i>0.36</i>	0.32	0.13	1.00									
Floral (Fl)	0.11	0.26	0.01	0.00	-0.02	-0.24	-0.13	-0.11	0.04	1.00								
Buttery (Bu)	0.49	<i>0.41</i>	-0.27	0.54	<i>-0.46</i>	0.19	-0.08	0.25	<i>0.42</i>	0.26	1.00							
Dairy (Da)	0.33	0.34	<i>-0.37</i>	0.34	<i>-0.62</i>	-0.06	-0.08	0.30	0.08	0.18	<i>0.38</i>	1.00						
Hay-like (HL)	-0.45	-0.28	0.13	-0.18	0.34	-0.12	0.11	-0.19	0.05	-0.07	-0.31	<i>-0.55</i>	1.00					
Barny (Ba)	-0.12	<i>-0.37</i>	0.48	<i>-0.39</i>	0.33	0.02	0.09	0.00	-0.29	-0.23	<i>-0.37</i>	<i>-0.54</i>	0.22	1.00				
Green (Gn)	-0.16	0.08	<i>-0.35</i>	-0.05	-0.19	-0.02	0.17	-0.02	0.18	-0.25	-0.04	0.07	0.28	-0.18	1.00			
Rancid (Ra)	-0.33	0.14	0.05	-0.09	0.11	0.22	0.00	-0.18	0.27	0.03	-0.05	-0.18	0.11	0.18	-0.15	1.00		
Waxy (Wx)	-0.20	0.15	-0.16	0.15	0.16	0.22	0.17	-0.08	<i>0.58</i>	0.34	0.18	-0.25	0.22	0.00	0.12	0.33	1.00	
Earthy (Ea)	-0.45	0.02	0.17	-0.05	<i>0.40</i>	0.08	0.19	-0.22	0.34	-0.04	-0.19	<i>-0.52</i>	<i>0.58</i>	0.16	0.12	0.22	0.50	1.00

^aData in **bold**, ***bold italics***, and underlined character indicate significant at $p < 0.05$, $p < 0.01$ and $p < 0.001$ respectively

Table 3.6 Selected rotated factor loadings (higher than 0.30, in parentheses) of each significant attributes

Factor	Attributes	
	Positive loading	Negative loading
1	Earthy (0.76) Hay-like (0.66) Smoky (0.65) Barney (0.56), Woody (0.56)	Dairy (0.81)
2	Cooked grain (0.92) Nutty (0.62) Grain (0.60) Buttery (0.56) Starchy (0.48) Waxy (0.40)	
3	Green (0.82) Sulfury (0.45)	
4	Popcorn (0.84) Corn (0.38)	
5	Floral (0.72)	
6	Rancid (0.91)	

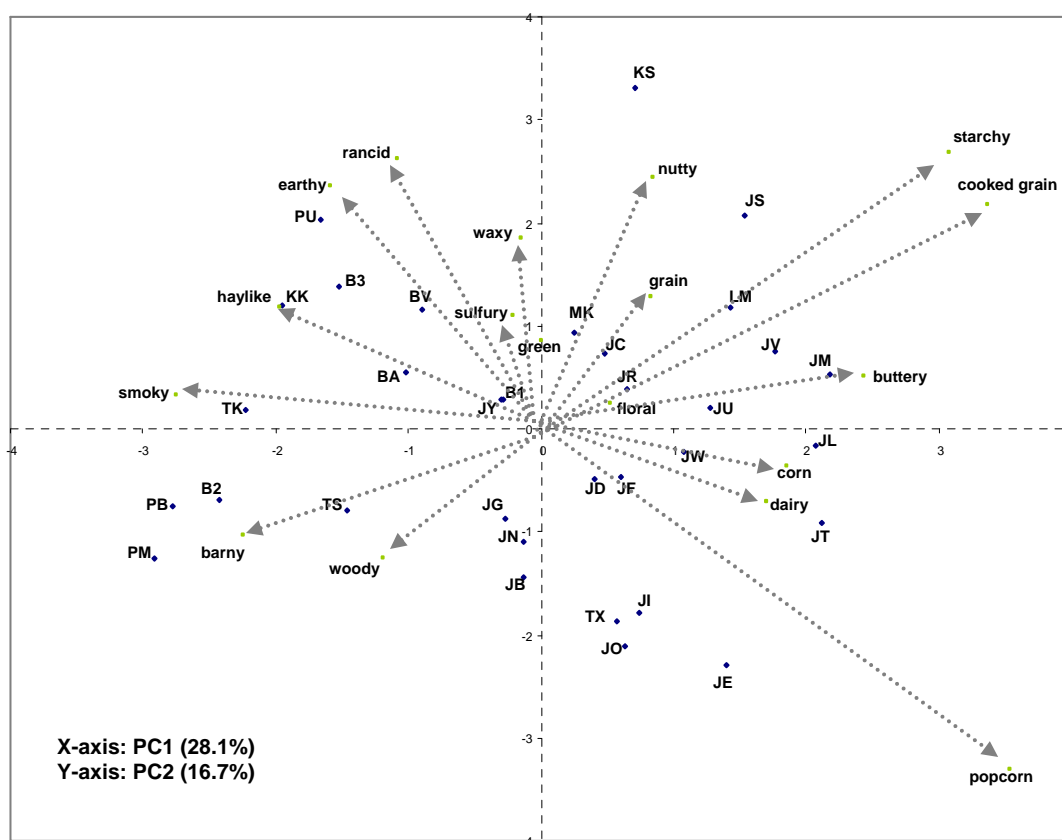


Figure 3.1 Principal component analysis (PCA) biplot of the covariance matrix of 18 significant mean sensory attributes ratings across the 33 rice samples. PC1 compared with PC2. See Table 3.1 for rice sample codes.

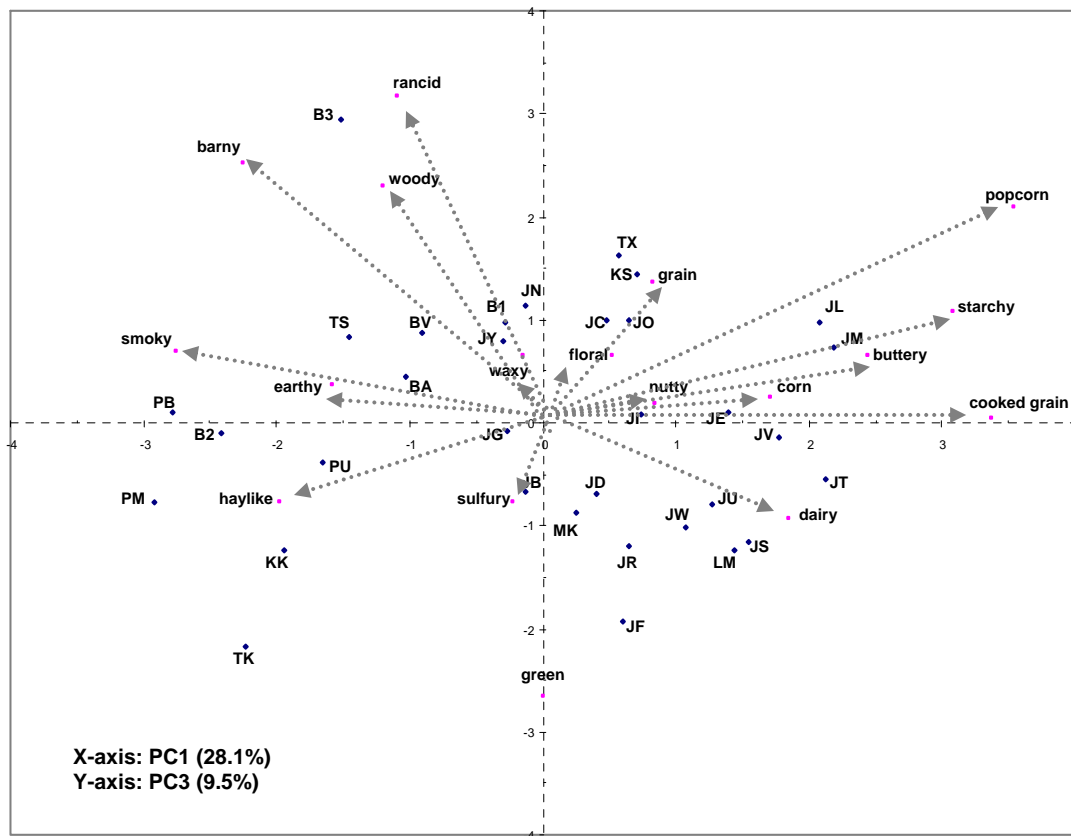


Figure 3.2 Principal component analysis (PCA) biplot of the covariance matrix of 18 significant mean sensory attributes ratings across the 33 rice samples. PC1 compared with PC3. See Table 3.1 for rice sample codes.

CHAPTER 4
THRESHOLD VALUES FOR SELECTED KEY AROMA COMPOUNDS IN RICE
USING DIFFERENT MATRICES¹

¹Limpawattana M, Yang DS, Kays SJ, Shewfelt RL To be submitted to Journal of Food Science

ABSTRACT

Orthonasal detection thresholds of selected compounds found in cooked aromatic rice were determined using the ascending method of limits in three different matrices; partially deodorized rice, water and corn starch. Threshold values of all selected volatiles determined in starch and partially deodorized rice were higher than threshold values in deionized water. Threshold values of all volatiles measured in starch were at least two times higher than threshold values in partially deodorized rice. Within the same compounds, individual thresholds in water were lower than those in starch and partially deodorized rice. The gas chromatographic profile of the control sample and partially deodorized rice revealed that hexanal was the major volatile compound in both samples followed by nonanal, heptanal, octanal and (*E*)-2-nonenal respectively, while 2-acetyl-2-thiazoline, an analogue to 2-acetyl-1-pyrroline, was not detected. Selected compounds were greater in the control sample than in the partially deodorized rice except (*E*)-2-nonenal. Volatile compounds in deionized water with odor activity values higher than one were hexanal, nonanal, and (*E*)-2-nonenal while heptanal and octanal were less than one. The results suggest that there was a matrix effect.

Keywords: thresholds, volatiles, rice, odor activity value, matrix

Introduction

Food flavors commonly comprise complex mixtures of volatile compounds which only a small number of these identified volatiles are of significance in determining the flavor (Grosch 2001). The first approach to select volatile components that contribute to a characteristic aroma of the food from those that do not is to obtain information about their perception threshold (Teranishi and others 1991). Volatiles present in food at higher concentrations than their minimum intensity detection are believed to contribute to the flavor of the food (Teranishi and others 1991). Generally, it is accepted that aroma quality changes with concentration and the sensory threshold of the odorants. Published threshold values mostly were reported in water, air or other matrices (van Gemert 2003). Detection thresholds are not only useful measuring tools for specifying the potency of a flavor compound in food but also measuring an individual's sensitivity to some flavor compounds of interest (Meilgaard and others 2007). It is recommended that sensory thresholds and odor activity values should be further reconfirmed key flavor volatiles after descriptive sensory analysis of flavor attributes is developed (Nursten and Reineccius 1996).

Food is a multi-component medium which recent studies indicated that volatile molecules interact with food ingredients and, thus, influence on flavor perception (Seuvre and others 2000; Guichard 2002; Heinemann and others 2005; Adhikari and others 2006). Since most foods have high water content (70-90%), odor thresholds of their volatiles are always cited in water. Odor threshold values depend on the medium in which the component is dissolved (Rothe 1976). A number of odor thresholds of key aroma compounds have been determined in numerous foods including vanillin (Powers and Shinholser 1988), beer (Meilgaard 1993), wine (Martineau and others 1995), potatoes (Work and Camire 1996), bread (Rychlik and Grosch 1996), tomato

(Tandon and others 2000), skim milk powder (Karagul-Yuceer and others 2004), apple juice (Eisele and Semon 2005) and mango (Pino and Mesa 2006).

Volatile compounds that contribute to the characteristic aroma of rice have been extensively studied and reviewed (Maga 1984). More than 200 rice volatiles have been identified but only some have odor threshold value low enough to make such contribution (Yajima and others 1978; Buttery and others 1988b; Widjaja and others 1996). Among these compounds, 2-acetyl-1-pyrroline is the most important volatile emitting a popcorn-like aroma firstly reported (Buttery and others 1982). Numerous studies have confirmed its intensity (Buttery and others 1983; Paule and Powers 1989; Tanchotikul and Hsieh 1991; Laksanalamai and Ilangantileke 1993; Jezussek and others 2002). Recent research on six different types of rice by Yang (2007) revealed that hexanal, heptanal, octanal, nonanal, (*E*)-2-nonenal, and 2-acetyl-1-pyrroline are important compounds based on calculation of odor activity values (OAV) which is derived from dividing its concentration with its threshold values determined in air.

In our studies, odor thresholds of selected compounds found to be important in cooked aromatic rice are determined using partially deodorized rice, water and corn starch as evaluation media. By using the partially deodorized rice as the medium of evaluation, thresholds are deemed to be more realistic, than in air or water. OAV (the concentration of an odor active compound divided by its odor threshold), were used to estimate the relative significance of each compound contributing to the aroma (Grosch 1993). Our objective was to determine the detection thresholds of selected odor-active compounds responsible for the aroma of aromatic rice in three different matrices; water, corn starch and freshly cooked partially deodorized rice using the ascending forced-choice method and determine the sensitivity range of the existing trained panelists.

Materials and Methods

Volatile compounds

The following potent volatile compounds contributing to cooked aromatic rice flavor were selected based on odor activity values (OAVs): hexanal, heptanal, octanal, nonanal, (*E*)-2-nonenal, and 2-acetyl-1-pyrroline (Yang 2007). OAV is obtained by dividing the concentration of an odor active compound by its odor threshold (Acree and others 1984; Grosch and Schieberle 1997). Food grade volatile compounds were purchased from Sigma-Aldrich, Flavors and Fragrance (Milwaukee, Wis., U.S.A.) with purity given in parentheses as follows: hexanal (98%), heptanal (92%), octanal (92%), nonanal (95%), (*E*)-2-nonenal (93%), and 2-acetyl-2-thiazoline (96%). Since 2-acetyl-1-pyrroline (2-AP) was not commercially available, an analogue, 2-acetyl-2-thiazoline (2-AT) was used due to its similar popcorn-like odor and low odor threshold (Adams and De Kimpe 2007). Previous studies on threshold and sensory model also reported the use of 2-AT in place of 2-AP (Buttery and others 1988a; Karagul-Yuceer and others 2004).

Rice samples

Milled long grain US grown rice (Mahatma Brand, Riviana Foods Inc., Houston, Tex., U.S.A.) in a 10-lb bag, purchased from the local supermarket in Athens, Georgia during Spring 2007, was individually weighed into 300g-portion, stored in a zipped lock bag (Ziploc Brand, S.C. Johnson & Son Inc., Racine, Wis., U.S.A.) and put in an air-tight plastic container in the refrigerator at 5 °C until further testing.

Partially Deodorized rice

Rice samples were taken out from cold storage and equilibrated to room temperature. The stored rice sample (200 g) was coarsely ground in a coffee grinder (MR.COFFE Brand:

model IDS-50, China) with 15 pulses and sieved using a commercial kitchen strainer. The rice was then washed with 400 mL of deionized water with constant stirring for 10 min (Monsoor and Proctor 2002) and filtered through a strainer. Washed rice was transferred to a 1-L Erlenmeyer flask containing 400 mL of pre-heated deionized water at 70 °C. The flask was connected to vacuum outlet and purged with purified N₂ gas at 60 mL/min (Yau and Liu 1999) for 10 min with the water-bath heating at 70 °C (Monsoor and Proctor 2004). Next, rice was separated from the suspension by filtering through cheesecloth; it was placed in a hot-air oven for 1 h at 80 °C. The process was repeated until the desired amount of dried rice samples was obtained.

Gas chromatography-mass spectrometry analysis of partially deodorized rice

To prepare samples for analysis, 60 g of partially deodorized rice was cooked with 90 mL of deionized water in a 1-L glass vessel covered with glass adaptor on a preheated hotplate for 25 min. The glass vessel was then placed in a water bath at 70 °C. An internal standard, benzyl acetate (10 ppm) was added before collection and analysis of the volatiles. The volatiles emanating from the various matrices were adsorbed on a Tenax trap. Headspace volatiles were pulled through the trap using an aspiration pump for 1 hour and then thermally desorbed using an automated short path thermal desorption system (Model TD-5, Scientific Instrument Services, Ringoes, N.J., U.S.A.) on the injector port of the gas chromatograph/mass spectrometer (GC-MS, Model 6890N/5973, Agilent, Palo Alto, Calif., U.S.A.). The volatiles were separated using a fused silica capillary column (0.20 i.d., 30 m and 0.25 µm film thickness). The collected samples were desorbed at 250 °C for 5 min at a helium flow rate of 10 mL/min and the analytes collected on the first 4 cm of the GC column using a CO₂ cooled cryofocus trap (-40 °C) (SIS 2" Cryo-Trap, Scientific Instrument Services, Ringoes, N.J., U.S.A.). After desorption, the cryofocus trap was rapidly heated to 200 °C and the analytes separated using temperature

programming. Desorbed volatiles compounds were injected into a column at 225 °C using helium flow rate of 1.0 mL/ min. The column temperature was programmed to hold at 40 °C for 1 min and then increase to 65 °C at 1.5 mL/ min and hold for 1 min, increase to 120 °C for 1 min at 2 mL/min, and finally increase to 280 °C for 5 min at 15 mL/ min. The operational MS conditions were as follows: ion source, 230 °C; electron energy 70 eV; multiplier voltage 1247 V; GC-MS interface zone 280 °C; and a scanning mass range (m/z) of 35 to 350 mass units.

Volatile component identification and quantification

Rice sample volatiles were positively identified by comparing the retention time and mass spectrum with those of authentic standards. Initial identification of compounds was based on comparing the mass spectrum with the Wiley library (7th ed., Wiley, N.Y., U.S.A.) and the National Institute of Standards and Technology of mass spectral database. Five identified volatile compounds representing character impact compounds of aromatic rice (Yang 2007) were selected. Quantification of selected compounds was carried out by establishing a calibration curve with four concentrations; 10, 100, 500, and 1000 ppm in hexane for each compound using authentic standards.

Sensory panel

A sensory panel consisting of eight members professionally trained in the Spectrum™ descriptive methodology (Meilgaard and others 2007) for rice flavor was convened. Six were female; two were male with age ranging from 23-54 years. The panel took place in a controlled sensory panel room (20 °C) containing partitioned booths equipped with fluorescent lights at the Food Process Research and Development Laboratory of the Department of Food Science and Technology at the University of Georgia, Athens, GA. Panelists were familiarized with the appropriate protocol prior to testing.

Sensory procedure

Orthonasal threshold evaluations for each volatile compound were determined in three media: deionized water, starch, and partially deodorized (PD) rice. Published thresholds in water were predetermined using three panelists including the experimenter, to establish the appropriate concentration for the panelists. Five successive concentrations of each substance, increasing by a factor of three based on preliminary assessment, were used, such that the panel threshold would be near the middle of the range (Meilgaard 1993). For the starch medium, volatile compounds in ethanol solution were dispensed in edible corn starch (Hodgson Mill, Effingham, Ill., U.S.A.), stirred consistently and diluted stepwise with corn starch (Rychlik and Grosch 1996). One presentation consisted of five triangles, each containing two controls and one sample (3-alternative forced choice) (ASTM 1997) except in PD rice. Sample size was 15 mL for deionized water media and 15 g for starch media. The samples were placed in 29.5 mL plastic (polystyrene) soufflé cups (Solo[®] Cup Company, Urbany, Ill., U.S.A.), capped and assigned 3-digit random codes. Cups within each set were presented in random order at room temperature.

Partially deodorized rice was spiked with each volatile compound at concentrations (5) differing by a factor of three before cooking in micro-computerized rice cookers (Sanyo model ECJ-D100S, Sanyo, Japan). Immediately after cooking, 30 g sample of the spiked PD rice was transferred into 120 mL styrofoam cups (Dart[®] Container Corp., Mason, MI, U.S.A.) and capped without delay in order to maintain the temperature above 70 °C. A modified ascending method of limits was used to determine odor thresholds by asking panelists to select the spiked sample from one of two samples (one containing the compound and another was the controlled PD cooked rice) (Tandon and others 2000).

Panelists were instructed to open the cups close to their noses and take three short sniffs of the headspace with their mouths closed starting from left to right and to decide which one is different from the other two except PD rice medium as indicated above. Even if they could not perceive a difference, a choice is needed to be made with certainty of judgment (sure/not sure) using the correction factor (Lawless and others 2000). Panelists were also instructed to provide odor descriptors for each of the volatile compounds in each of the three media. Panelists waited for 30 seconds between each set of samples before conducting next set and sniffed their sleeves to clear the nasal passageways between cups. The individual best-estimate threshold was calculated as the geometric mean of the last concentration, with an incorrect response, and the first concentration with a correct response. The group best-estimate threshold (BET) was calculated as the geometric mean of the individual best-estimate thresholds.

Results and Discussion

Best estimate threshold (BET) values of selected rice compounds calculated in deionized water, starch and partially deodorized rice are shown in Table 4.1. All volatiles except 2-acetyl-2-thiazoline were previously positively identified and quantified in aromatic rice samples (Yang 2007). 2-Acetyl-2-thiazoline, a sulfur-containing analogue of 2-acetyl-1-pyrroline reported for the first time in beef broth (Tonsbeek and others 1971) exhibits similar roasty, popcorn-like odor characteristics and low odor threshold (Adams and De Kimpe 2006). A 2-acetyl-2-thiazoline has been used in place of 2-acetyl-1-pyrroline, since the latter is not commercially available (Karagul-Yuceer and others 2004). Threshold values of all volatiles determined in starch and partially deodorized rice were higher than threshold values in deionized water. Odor thresholds of all compounds in deionized water were lower than those in published studies except (*E*)-2-

nonenal. Threshold values of all volatiles measured in starch were at least two times higher than threshold values in partially deodorized rice. The difference between BET among different medium of evaluation indicated that there was a matrix effect. Meilgaard and others (2007) stated that thresholds can vary widely depending on testing procedures, number of panelists, and matrix used.

In this study, we chose the ascending forced-choice method of limits, ASTM E-679 procedure, because it is a rapid test to approximate group thresholds using limited size data sets (ASTM 1997). Eight trained panelists participating in descriptive sensory analysis were prescreened for their sensitivity and reproducibility. The data from this study was considered normally distributed and, therefore, the geometric mean was reported as the group detection threshold (Costell and others 1994). The lower odor threshold values determined in this experiment, as compared to the published values, was attributed to the sensitivity of panelists.

Figure 4.1 shows the individual thresholds for each panelist of each compound in deionized water, starch and partially deodorized rice. For the same compounds, individual orthonasal thresholds in water were lower than those in starch and partially deodorized rice. Log standard deviation from the geometric mean of the panel can describe the individual differences in sensory threshold. The log standard deviation varied from the most uniform at 0.11 for (*E*)-2-nonenal in starch to the least uniform at 1.08 for octanal in partially deodorized rice (Table 4.1). Individual differences in sensory threshold for aroma compounds as such were demonstrated by Meilgaard (1993). None of the panelists used in this study was found to be insensitive to rice volatiles tested. Some panelists were, however, very sensitive and could consistently detect compounds present at the very low concentration tested (Table 4.3). A high proportion of the

panelists was able to detect 2-acetyl-2-thiazoline at the lowest concentration tested in starch indicating that the lower concentration is probably needed.

We used a dilution factor of three as described by ASTM (1997) which is usually reported in detection threshold tests (Engen 1982). As a result, it appeared to cover the range of concentrations that was perceived by our panelists. Panelists were requested to give a descriptor, aided by relevant descriptors provided, if they could identify the flavor at a certain concentration level. Panelists appeared to be consistent in the descriptors they gave rather than using a wider vocabulary based on their personal experience, mostly showed similarities in all three media (Table 4.2).

A matrix effect was expected, due to the components of the food matrix interacting with flavor compounds and impacting flavor release into the headspace. The odor thresholds for all volatiles in starch were higher than thresholds determined in deionized water. The increase ranged from < 35-fold for nonanal to almost 300-fold for hexanal. A similar trend of increases was observed in odor thresholds determined in the partially deodorized rice but to a lesser amount than those determined in starch. The increase in thresholds was probably caused by amylose which binds flavor compounds by formation of inclusion complexes that result in a physical entrapment of the ligand and modification of the starch structure (Rutschmann and Solms 1990). Generally, milled rice may be classified based on amylose content as waxy (1-2 %), low (7-20%), intermediate (20-25%), and high (>25%) (Juliano 1985). Commercial corn starch has an amylose content of about 25-26%. The highest retention of volatile substances was previously reported in an amorphous polymeric matrix (Castellano and Snow 2001) but, in our studies, the corn starch was merely dispersed into the ethanolic solution of volatiles without any heat treatment. The other difference between the BET in corn starch and partially deodorized

rice may be partly due to preparation method. Panelists were required to shake the samples well before sniffing so that the consistency of the concentration in a gaseous phase was assumed. However, the compounds were dissolved in 1% ethanol solution to improve their solubility with the assumption that it would not be perceived. As a result, this solvent carrier, to some extent, might interfere with the aroma perception of volatiles. It was reported that subjects were unable to differentiate blanks containing ethanol at their threshold level of only 0.03% (Krajewska and Powers 1988). Our results suggested that ethanol has irritative properties when used as solubilizing agent in threshold tests.

The third medium of evaluation was PD rice which we developed as an alternative matrix close to the actual cooked rice. Comparison between the gas chromatography profile of the control sample and PD rice as shown in Figure 4.2 revealed that hexanal was the major volatile compound in both samples followed by nonanal, heptanal, octanal and (*E*)-2-nonenal respectively while 2-acetyl-2-thiazoline was not detected. The tested compounds were greater in the control sample than in the PD rice except (*E*)-2-nonenal. Hexanal, 2-nonenal and heptanal are breakdown products of linoleic acid while octanal, nonanal are secondary oxidation products of oleic acids (Grosch 1987). Linoleic acid is much more susceptible to oxidation than oleic acid (Galliard 1989). The initial concentration of hexanal, heptanal and (*E*)-2-nonenal in the control sample probably reflects the higher concentration of linoleic acid as compared with oleic acid. The concentration of (*E*)-2-nonenal in PD rice was higher than that in the control, probably due to lipid autoxidation. Rice lipids are known to occur in both free and starch bound forms. Water washing of milled rice was reported to reduce the total surface lipid and free fatty acids (FFA) contents before storage about 60-80% and decrease the rate of increase of FFA during storage (Monsoor and Proctor 2002). However, the rice/water ratio and washing time were not

the key factors affecting lipid and FFA contents (Monsoor and Proctor 2002). Extraction of free lipids adhered on the surface of starch granules could be done by using ether at room temperature; whereas, bound lipids located inside of starch granules require ethanol as an extractant at high temperature (Yoshihashi and others 2005).

PD rice samples were presented in insulated Styrofoam cups at a temperature no less than 70 °C at which a consumer would eat cooked rice. This higher temperature would increase the amount of volatiles in the headspace, and thus, decrease orthonasal odor threshold with relative to odor threshold perceived in starch. However, 2-acetyl-2-thiazoline was shown to be unstable during heat treatment in the presence of water, but a significant stabilization was observed in oil (Hofmann and Schieberle 1995). Therefore, fat-containing food matrices might enhance the stability of 2-acetyl-2-thiazoline. In our studies, almost panelists could detect 2-acetyl-2-thiazoline at the lowest concentration added, probably because the concentration was not low enough to observe instability. Application of this compound to non-aromatic cooked rice using Indian style cooking where butter is added probably increases the roasted and popcorn-like flavor notes of the rice similar to cooking with the authentic Basmati rice.

It is now known that odor activity values are used to screen which compounds are most likely to contribute to the food flavors rather than to reliably determine the key aroma components (Reineccius 2000). By using concentration of selected compounds determined in the control sample, the odor activity value is different among the three matrices. Odor activity values (Table 4.4) for volatile compounds in deionized water, higher than one, were hexanal, nonanal, and (*E*)-2-nonenal while OAVs for heptanal and octanal were less than one. None of volatile compounds determined in starch and partially deodorize demonstrate OAVs of more than one. Basically compounds with OAV values greater than one are likely to be detectable

depending on flavor release and odor suppression for other components in the sample which in turn probably explain our results in this case. However, OAV values do not correlate linearly with perceived intensity values and do not predict the odor intensity of compounds in combination (Mayol and Acree 2001). We selected the prominent volatiles based on OAVs derived from GCO techniques which generally do not consider interaction between odorants. It was suggested that one would not expect OAV to be an accurate indicator of the contribution of an odorant to a mixture (Audouin and others 2001). Our studies give an indication that partially deodorized rice is unlike liquid food that is easy to deodorize and obtain a homogenous blend of volatiles when spiked. It is also difficult to extract the composition of non-volatile component which is yet remain in the rice without using invasive method. These compounds that are below threshold concentration might play an important role to the overall aroma and flavor perception in terms of enhancing or suppressing each other.

Conclusion

The partially deodorized (PD) rice was developed and used as a medium to determine orthonasal detection thresholds of selected compounds found in cooked rice as compared to water and corn starch. A matrix effect was observed among these three media on the thresholds. Although odor activity values calculated from the derived thresholds using the concentrations of each compound determined in the control rice did not surpass the common value of one to indicate their contribution, but determination of threshold in the same matrix as the sample under investigation is recommended.

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Table 4.1- Best estimate thresholds (BET) for orthonasal odor thresholds determined for selected compounds in different matrices and published values in the same medium for comparison

Compound	Matrix	BET ^a	Log Standard Deviation	Selected published Threshold (µg/L)
Hexanal	Deionized water	2.78	0.34	10.5 ^b
	Starch	832.1	0.17	
	Partially deodorized rice	380.7	0.73	
Heptanal	Deionized water	2.06	0.43	5.8 ^b
	Starch	180.9	0.16	
	Partially deodorized rice	64.8	0.79	
Octanal	Deionized water	0.98	0.31	8 ^b
	Starch	39.8	0.16	
	Partially deodorized rice	8.7	1.08	
Nonanal	Deionized water	2.06	0.37	5 ^b
	Starch	71.0	0.14	
	Partially deodorized rice	29.8	0.61	
(E)-2-Nonenal	Deionized water	0.09	0.72	0.08 ^b
	Starch	21.3	0.11	0.53 ^d
	Partially deodorized rice	4.9	0.88	
2-Acetyl-2-thiazoline	Deionized water	0.12	0.62	1 ^c
	Starch	15.7	0.08	
	Partially deodorized rice	4.6	1.00	

^a BET of 8 panelists and define unit as µg/L for deionized water medium and as µg/Kg for starch and partially deodorized rice media

^b Values quoted from van Germert (2003)

^c Karagül-Yüceer and others (2004)

^d Rychlik and Grosch (1996), define unit as µg/Kg

Table 4.2- Odor descriptors for selected compounds in different matrices

Compound	Deionized water	Starch	Partially deodorized rice
Hexanal	Grass/green	Penetrating/fruity	Green/herbal/soapy
Heptanal	Floral/fatty	Floral/herbal	Rancid/soapy
Octanal	Orange/fruity	Lemon/fatty	Citrus/oily
Nonanal	Waxy/fruity	Rose/rancid	Floral/citrus
(<i>E</i>)-2-Nonenal	Stale/oily/fatty	Penetrating/fatty	Medicine-like/rancid
2-Acetyl-2-thiazoline	Popcorn/honey	Popcorn/roasty	Popcorn/sweet

Table 4.3- Panelist BET range values determined for selected compounds in different matrices

Compound	Panelist BET range ^a		
	Deionized water	Starch	Partially deodorized rice
Hexanal	1.73 to 16.96	435.6 to 1199.5	42.0 to 1537.9
Heptanal	0.62 to 9.81	117.0 to 293.8	4.5 to 362.9
Octanal	0.62 to 3.25	26.3 to 71.0	0.11 to 108.7
Nonanal	0.68 to 5.66	47.2 to 103.9	3.2 to 138.3
(<i>E</i>)-2-Nonenal	0.02 to 2.43	16.2 to 28.2	0.23 to 50.6
2-Acetyl-2-thiazoline	0.02 to 2.65	14.1 to 21.4	0.11 to 26.0

^a BET of individual and define unit as µg/L for deionized water medium and as µg/Kg for starch and partially deodorized rice media

Table 4.4- Odor activity values (OAVs) of selected compounds in different matrices

Compound	Con. in long grain rice ($\mu\text{g/Kg}$)	Deionized water	Starch	Partially deodorized rice
Hexanal	29.74	10.7	0.04	0.07
Heptanal	1.52	0.74	0.01	0.02
Octanal	0.77	0.79	0.02	0.09
Nonanal	2.08	1.00	0.03	0.07
(<i>E</i>)-2-Nonenal	0.25	2.78	0.01	0.05
2-Acetyl-2-thiazoline*	ND	ND	ND	ND

*Not detected in the control rice sample

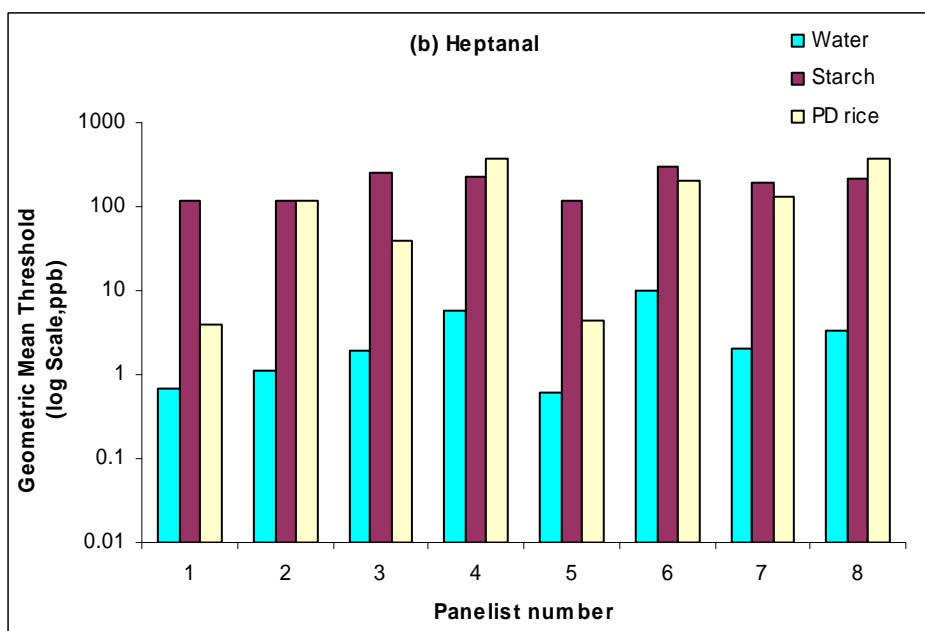
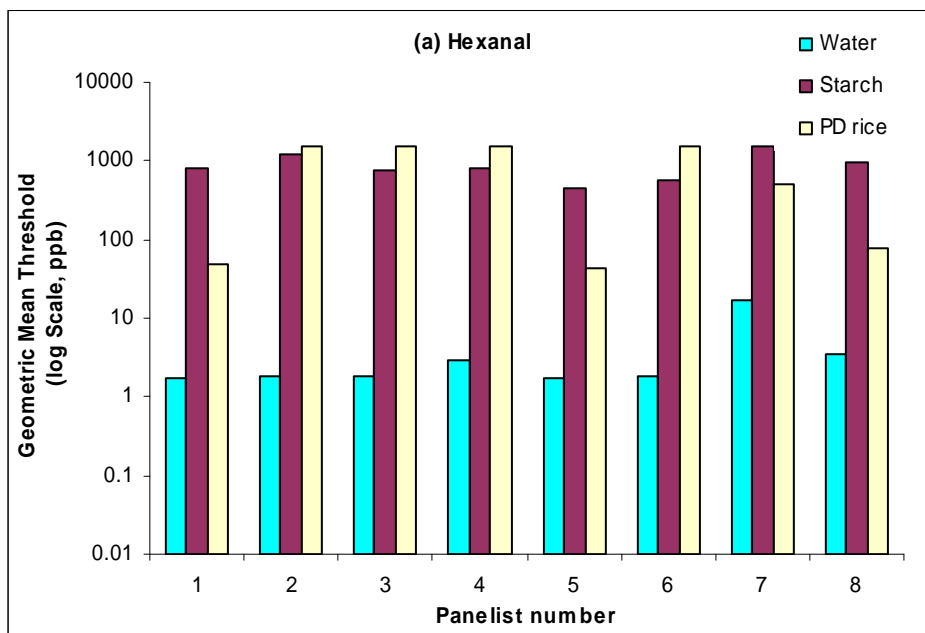


Figure 4.1- Individual best estimate threshold for (a) hexanal (b) heptanal (c) octanal (d) nonanal (e) (*E*)-2-nonenal (f) 2-acetyl-2-thiazoline in different matrices

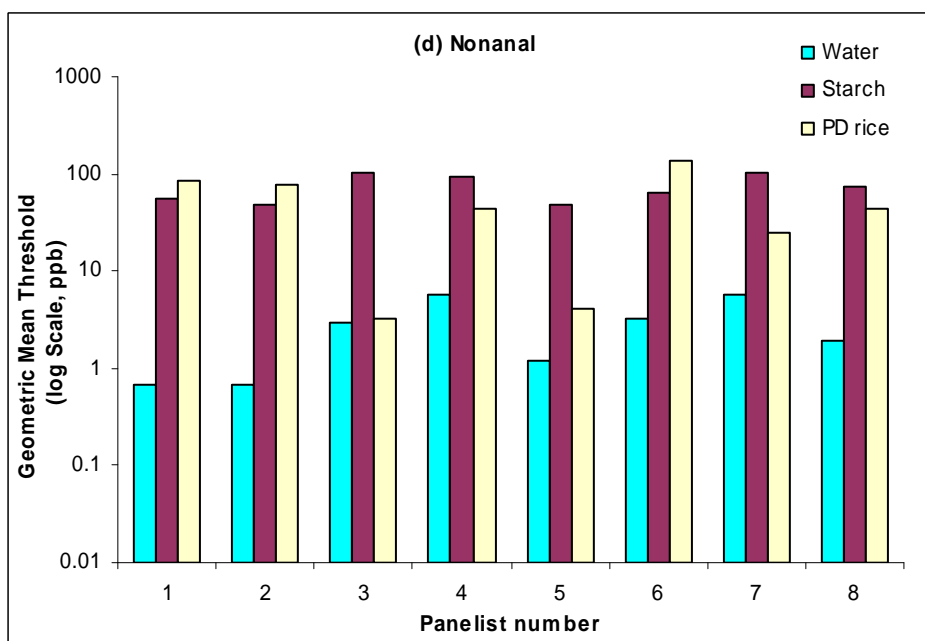
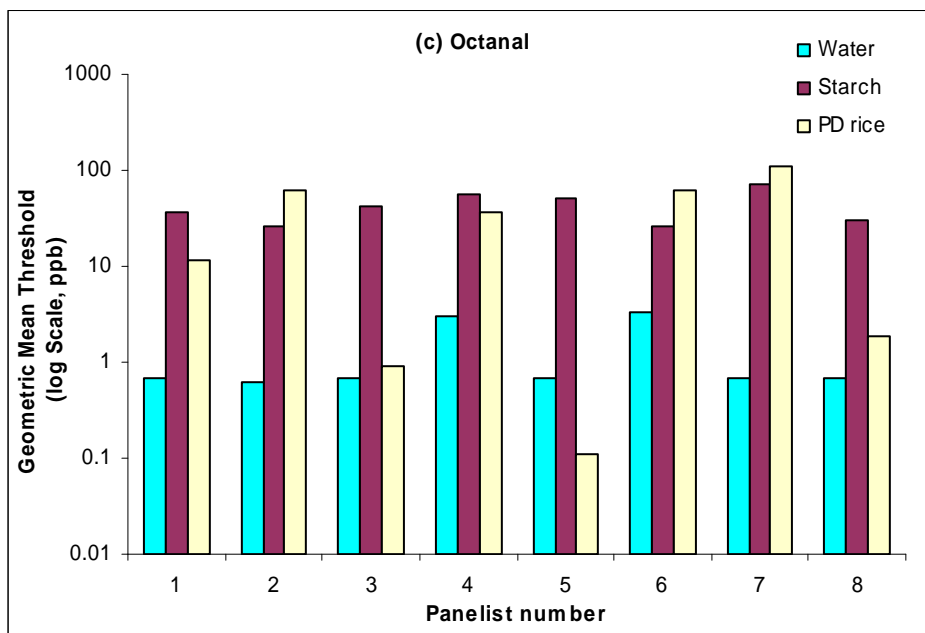


Figure 4.1- Individual best estimate threshold for (a) hexanal (b) heptanal (c) octanal (d) nonanal (e) (*E*)-2-nonenal (f) 2-acetyl-2-thiazoline in different matrices

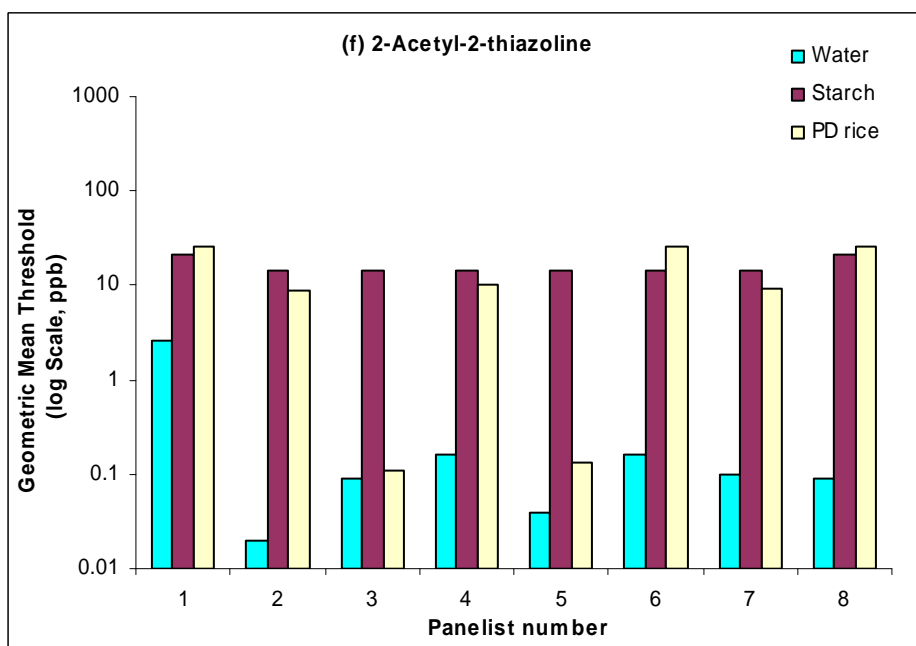
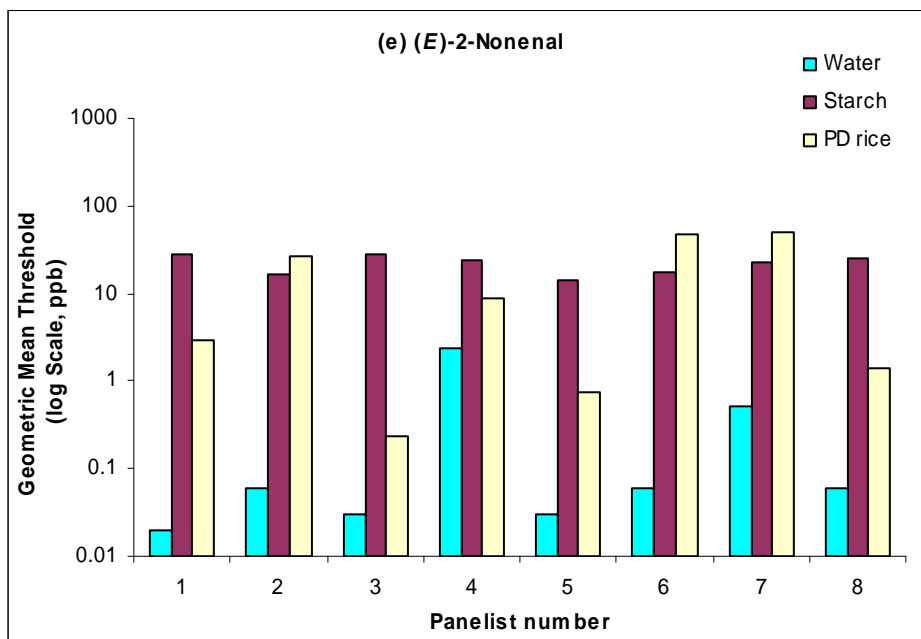


Figure 4.1- Individual best estimate threshold for (a) hexanal (b) heptanal (c) octanal (d) nonanal (e) (E)-2-nonenal (f) 2-acetyl-2-thiazoline in different matrices

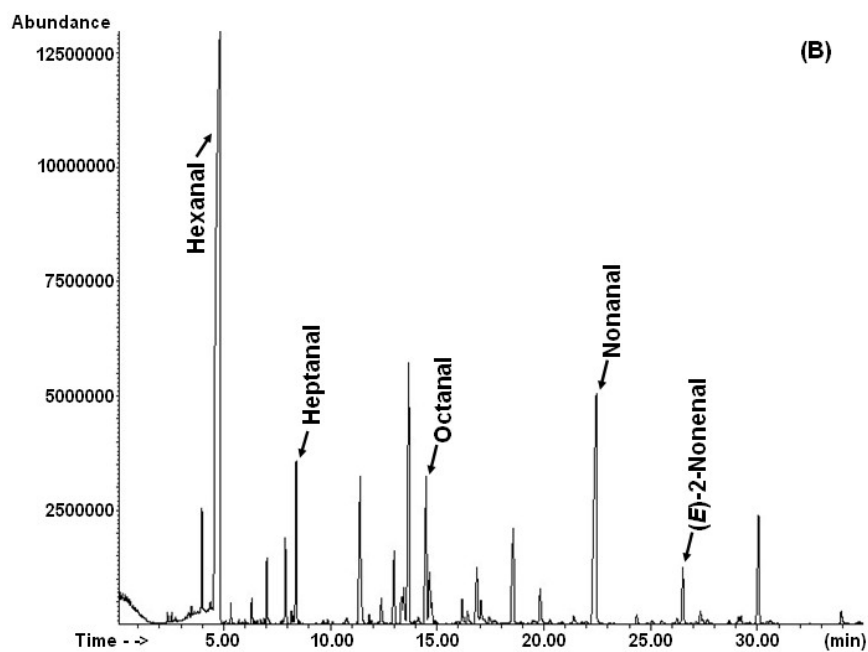
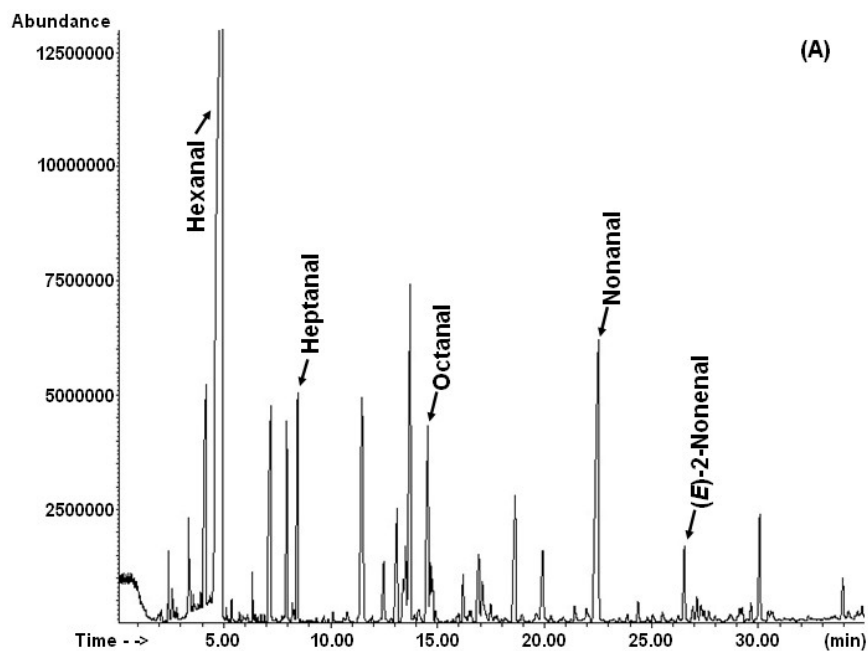


Figure 4.2- Gas chromatogram of specific aroma active compounds in long-grain non-aromatic rice (A) and partially deodorized rice (B)

CHAPTER 5
RELATING SENSORY DESCRIPTORS TO VOLATILE COMPONENTS OF
SPECIALTY RICE FLAVOR¹

¹Limpawattana M, Yang DS, Kays SJ, Shewfelt RL. To be submitted to Journal of Food Science

ABSTRACT

Nineteen aromatics attributes in cooked specialty rice samples evaluated by descriptive sensory analysis were statistically correlated to the concentration of aroma-active compounds derived from gas chromatography-olfactometry (GC-O) analysis. Significant prediction models were developed for most aromatic descriptors including popcorn, cooked grain, starchy, woody, smoky, grain, corn, hay-like, barny, rancid, waxy, earthy and sweet aromatics using Stepwise multiple linear regression. (*E,E*)-2,4-decadienal, naphthalene, guaiacol, (*E*)-2-hexenal, 2-acetyl-1-pyrroline, 2-heptanone contributed most to these sensory attributes. These models help provide a link between sensory characteristics of commercial rice samples and desirable traits to be used in selection by rice breeders.

Keywords: rice, aroma-active compound, regression model, descriptor, volatile

Introduction

Rice serves as the main staple for people in more than half of the world. The demand for production of rice with high quality in the global market has remarkably increased in recent years (FAO 2006). Many factors influence the palatability of rice (Lyon and others 1999; Champagne and others 2004; Bett-Garber and others 2001; Meullenet and others 1999). Consumer acceptance of specialty rice such as Jasmine rice was driven by its prominent sensory characteristic including flavor and aroma (Suwansri and others 2002). Previous research indicated that consumers would pay more in trade of full-flavored products (Bruhn and others 1991) and the finding would inevitably be applied to all food products including rice.

Descriptive sensory analysis by trained panelists is the primary tool that can be used for assessment of these specific properties such as aroma and flavor (Yau and Huang 1996). Since the sensory properties of cooked rice are subtle, numerous studies emphasize texture and cooking quality (Sesmat and Meullenet 2001; Mohapatra and Bal 2006; Meullenet and others 2000; Leelayuthsoontorn and Thipayarat 2006; Hirannaiah and others 2001; Champagne and others 1998) over flavor (Yau and Huang 1996; Champagne and others 2004).

Extensive studies on rice revealed that among 200 identified volatile compounds, only a few contributed to the characteristic aroma of rice (Yajima and others 1978; Buttery and others 1988; Widjaja and others 1996; Jezussek and others 2002). The key compound responsible for the aroma of rice was reported as 2-acetyl-1-pyrroline which possesses a popcorn-like aroma (Buttery and others 1982) and is present in all parts of the plant except roots (Yoshihashi 2002). Non-aromatic rice has a higher amount of n-hexanal, other alkanals, and 2-pentylfuran than aromatic rice (Widjaja and others 1996).

At present, gas chromatography-olfactometry (GC-O) is widely used as the powerful method to estimate the sensory contribution of a single aroma compound to food flavors. The relationships between volatile compounds and descriptive attributes of cooked rice were previously reported (Paule and Powers 1989; Yau and Liu 1999) but sensory descriptions were not related directly to individual aroma compounds (Wilkie and others 2004). Consequently, more information on compounds that lead to the prediction of the sensory properties of rice is needed to be elucidated.

The objective of this study was to develop prediction models for sensory descriptors based on the volatile components derived from GC-O that would be useful to help select rice cultivars containing a satisfactory flavor to produce improved quality in rice breeding program.

Materials and methods

Rice samples

Thirteen specialty rice samples originally harvested from National Institute of Crop Science (NICS), Suwon, South Korea in 2006 were air-freighted to the University of Georgia for chemical and sensory analysis. Upon arrival, the samples were individually sealed in air-tight glass containers at -20°C until further evaluation. Of the 13 samples, two were aromatic white milled rice, three pigmented rice, two glutinous rice, and six premium rice (known for flavor in Korean markets) (Table 5.1).

Sensory panel

Eight panelists (2 men and 6 women, age range 22 to 54 y) previously screened on the basis of their health, availability and willingness to consume rice and trained in the descriptive sensory analysis using the Spectrum™ methodology (Meilgaard and others 2007) participated in

this study. The panelists were chosen from students and staff of Department of Food Science and Technology at the University of Georgia and received six-1.5 hour training sessions with the wide range of references using the Spectrum™ technique (Meilgaard and others 2007). Rice flavor attributes were evaluated using a modified descriptive Spectrum® method utilizing a 150-mm line scale (Tandon and others 2003) using a flavor lexicon developed by Limpawattana and Shewfelt (2007). The sensory flavor profile included 24 attributes that were categorized into aromatics, taste and oral feeling factor terms. Evaluation was carried out by sniffing and tasting. The panel was calibrated by the provided reference standards which were also marked on a 150-mm unstructured line scale. Panelists received premium chocolate treats as rewards for their participation in each session.

Sample preparation and evaluation procedure

Micro-computerized rice cookers (Sanyo model ECJ-D100S, Sanyo, Japan) were used to cook all the rice samples after equilibration to room temperature of rice samples for approximately 12 h. All the rice samples except black rice were prepared using 1.5: 1 water: rice ratio (by weight) while that of black rice was 1:1 (Limpawattana and Shewfelt 2007). Cooking time was varied between 38-45 min depending on the rice types. Upon completion of cooking as indicated by the light switching to “warm” mode, the samples were transferred without delay into preheated (75 ± 2 °C) 180-mL glass custard cups insulated with Styrofoam cups and covered with a three-digit coded glass petridish. Panelists were required to complete their evaluation before the temperature dropped to 60 °C as monitored by individual hand-held digital thermometers. Cooked rice samples were served monadically in random order across samples to panelists in partitioned booths under fluorescent light and controlled room temperature. Panelists were instructed to smell the samples while keeping their mouth closed but when tasting

they were told to hold their breath. Panelists scored all attributes on paper ballot using a 150-mm unstructured line scale with references standards assigned preselected points on the line scales. Unsalted-top crackers and water were provided for panelists to cleanse their palates between samples. Sensory testing was conducted in three sessions in the morning. At the beginning of each session, reference rice samples represented the same types of rice to be tested were presented and evaluated as “warm-up” samples. The entire evaluation was conducted at the Food Products Research and Development Laboratory in the Department of Food Science and Technology at the University of Georgia.

Sample preparation by dynamic headspace sampling

One hundred grams of rice sample was cooked with 150 mL of distilled water on the hotplate in the all-glass system. Headspace volatiles generated from cooked rice were collected at 70°C for 30 min using air purified by passing it through a charcoal filter at 150 mL/ min, into the container with the rice sample and then through a Tenax trap using an aspiration pump. After sampling, the Tenax trap was thermally desorbed at 250 °C for 5 min via an automated short path thermal desorption system (Model TD-5, Scientific Instrument Services, Ringoes, N.J., U.S.A.) on the injector port of the gas chromatograph/mass spectrometer. The desorption volatiles were retrapped on the first 3 cm of the column using a CO₂ cooled cryofocus trap (-40 °C) (SIS 2” Cryo-Trap, Scientific Instrument Services, Ringoes, N.J., U.S.A.) and subsequently flash heated to 200 °C. The analytes were separated on the column using temperature programming. One mL of 18.34 mg/L trimethylpyridine (TMP) solution in 0.1 M HCl was used as an internal standard for 2-AP. Three replications were analyzed for each rice sample.

GC-MS and Gas Chromatography-Olfactometry

Chromatography was performed using an HP5-MS Model 6890N/5973 gas chromatograph-mass spectrometry (Agilent Inc., Palo Alto, Calif., U.S.A.) equipped with fused silica capillary column (0.20 i.d., 30m, 0.25 μ m film thickness,). The injector temperature was 225 °C with a split ratio of 0.5:1. Helium was used as the carrier gas at 2.0 mL/min. The column temperature was held at 40 °C for 1 min following sample injection and then programmed at 1.5 mL/min to 65 °C which was held for 1 min, at 2 mL/min to 120 °C for 1 min, finally at 15 mL/min to 280 °C for 5 min. The electron energy at 70 eV was used for the ionization. Volatiles exiting the column were split between the mass spectrometer for identification and quantification and the sniffing port with humidified purged air for description and intensity assessment (ODO II, SGE Intl., Austin, Tex., U.S.A.). Samples of volatile compounds from each of the thirteen rice types were analyzed by GC-O using 3 trained evaluators including the experimenters in triplicate. Identification for each odorant was reported when its mass spectra, Kovats' retention indices (RI), an odor character matched those of authentic standards while quantification was determined using standard curves for each odorant (Yang 2007).

Statistical analysis

Results derived from warm-up samples were compared with those previously reported (Limpawattana and Shewfelt 2007) by a paired t-test using PROC TTEST (SAS version 9.13, SAS Institute, Inc., Cary, N.C., U.S.A.) since both results were taken from the same panel as to indicate panel consistency. Prediction models for sensory descriptors as functions of volatiles were developed by Multiple linear regression (MLR) using stepwise procedure (SAS 2007). Correlations between sensory descriptors and volatiles were also determined.

Results and Discussion

Sensory terms detected by the panel in the warm-up rice samples were similar to those found in the same types of rice that have been previously assessed (Limpawattana and Shewfelt 2007). Mean scores of sensory terms for all warm-up samples were not significantly different from those previously reported ($p < 0.05$, data not shown) indicating the panel consistency. Thus, the sensory evaluation of thirteen rice samples in this study was useful with merely one replication. The different flavor profiles of each type of rice are shown in Figure 5.1. Compared with the other rice type except the premium rice, aromatic rice revealed to have high scores in popcorn aroma and nutty aroma. Hay-like and barny aromas were rated highly in all three black rice samples. The highest scores for starchy, cooked grain, waxy, woody, smoky and barny were found among premium rice especially in one sample (GT1). The intensities of sweet, salty, bitter and metallic were highest among premium rice samples compared to other rice types. Perhaps this was the reason why they are described as “good tasting” rice in the market. Astringent was strongest note in one sample of glutinous rice (KG2).

GC-O analysis on the basis of the odor activity values (OAVs) as reported by Yang (2007) indicated 1-pentanol, hexanal, (*E*)-2-hexenal, p-xylene, 2-heptanone, heptanal, 2-acetyl-1-pyrroline, (*E*)-2-heptenal, benzaldehyde, 1-octen-3-ol, 2-pentylfuran, octanal, 3-octen-2-one, (*E*)-2-octenal, guaiacol, 2-nonanone, nonanal, p-menthane-3-one, (*E*)-2-nonenal, naphthalene, dodecane, decanal, (*E,E*)-2,4-nonadienal, (*E*)-2-decenal, and (*E,E*)-2,4-decadienal were the most potent odorants in the 13 specialty rice samples. Thus, they served as the potential predictor variables in the regression model (Table 5.2). GC-O separates odor-active compounds from the odorless compounds. Hexanal with a characteristic of green tomato aroma, had the highest concentration in glutinous rice samples followed by the black rice samples (data not shown).

Hexanal was previously reported as a potent odorant in tomato (Buttery and others 1987; Tandon and others 2003).

Thirteen significant prediction models were generated for aromatics terms based on the coefficient of determination (r^2) over 0.6 without multicollinearity present in the models (Table 5.2). The quadratic terms for all variables were not included in the predictor matrix to prevent the models from being overly cumbersome. Data was not transformed, even though previous report showed that it could improve the r^2 (Togari and others 1995), since GC-O is a direct approach to assessing aroma potency. A strong popcorn aroma was primarily detected in the aromatic rice samples and one sample of glutinous rice (GT 3) as indicated in Figure 5.1. The concentration of 2-acetyl-1-pyrroline (Table 5.2), a significant contributor to the popcorn aroma in scented rice (Buttery and others 1982), appeared to negatively influence the perception of cooked grain and sweet aromatics. The popcorn descriptor obtained in the descriptive analysis was associated with guaiacol and (*E,E*)-2,4-decadienal. Unexpectedly, 2-acetyl-1-pyrroline was not selected for the model for popcorn descriptor. Considering these two volatile compounds, only (*E,E*)-2,4-decadienal was naturally present in popcorn aroma (Buttery and others 1997). The negative contribution of guaiacol and (*E,E*)-2,4-decadienal which have characteristic smoky and fatty notes respectively, indicated that the heating process during preparation of reference standard for popcorn effected the perception of the popcorn aroma.

The cooked grain descriptor was detected in all rice samples. The negative contribution of 2-heptanone (floral), 2-acetyl-1-pyrroline (popcorn), (*E,E*)-2,4-decadienal (fatty) and positive contribution of naphthalene was understandable because the cooked grain term is referred to general aromatics not being tied to any specific grain. The starchy descriptor was enhanced when the content of (*E*)-2-hexenal and (*E*)-2-heptenal which possess the green notes decreased.

Although guaiacol was prominent in smoke flavorings (Kostyra and Barylko-Pikielna 2006), only naphthalene, also present in smoke flavorings, contributed in a positive manner to the smoky descriptor and negatively to woody notes. It should be noted that (*E,E*)-2,4-decadienal negatively influenced the perception of grain, corn, earthy and sweet aromatics but positively influenced in smoky perception. Cooked grain, woody, smoky, grain, barny, earthy and sweet aromatics were influenced by the presence of naphthalene. The compounds possessing floral aroma detected by GC-O (data not shown) such as heptanal, 2-pentylfuran, 3-octen-2-one, 2-nonanone did not appear in the model for the floral descriptor. The same phenomenon was found for the buttery, nutty, sulfury, dairy and green descriptors. Hexanal, which contributed negatively to the barny notes, was present in high concentrations in the glutinous rice (data not shown). This observation is consistent with scores given to both glutinous rice samples in terms of barny perception.

Correlation analysis between sensory descriptors and volatile compounds is shown in Table 5.3. Most of correlation coefficient were low (< 0.70) but significant. Interestingly, most of aromatic descriptors were negatively correlated with volatile compounds. Volatile compounds from cooked rice were described by GC-O as individual compounds at the lowest threshold level, but each aroma attribute was obtained from descriptive analysis as an odor mixture. When odors are mixed, mixture suppression or masking of aromas at suprathreshold levels is not an unusual phenomenon, making identification of the components difficult especially when there are more than three compounds (Keast and others 2004). This suppression indicates that the quality of the aroma of the product does not resemble the sum of the component aromas. Flavor compounds have been shown to follow a sigmoidal psychophysical curve with enhancement at threshold, very low intensity, additivity at low/moderate intensity and

suppression at higher intensity (Keast and Breslin 2003). In our study, however, food references were used for each sensory descriptor to exhibit a sufficient demonstration for that particular term as to clarify panelists' perception of the attributes (Rainey 1986; Munoz and Civille 1998). A few terms were positively correlated with volatile. Smoky was positively correlated with (*E,E*)-2,4,-decadienal. Grain was positively correlated with naphthalene. The corn attribute was positively correlated with benzaldehyde, guaiacol and nonanal. The dairy note was correlated in a positive manner with heptanal. Rancid and sweet aromatics were positively correlated with 1-octen-3-ol and guaiacol respectively. A negative correlation between sensory data and chemical data was previously reported in tomato (Tandon and others 2003).

Conclusions

The use of multivariate linear regression to relate aroma quality obtained from descriptive analysis to aroma compounds obtained from GC-O allowed the satisfactory prediction of thirteen aromatics attributes of cooked specialty rice even though aroma perception is a complex process. It is clear that difference in flavor among aromatic rice types is far more complex than simply the concentration of 2-acetyl-1-pyrroline. These models were generated to aid in prescreening rice selections for flavor traits, circumventing the need to conduct sensory panels on hundreds of samples during the progeny selection process in rice breeding programs. Systematic methods of flavor quality evaluation are needed if rice breeders are going effectively to select lines on the basis of flavor traits. Models generated in this study provide a first step in achieving this goal.

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Table 5.1- Rice sample information and rice codes

Code	Cultivar	Type	Pedigree/line
HM1	Hyangmibyeo-1	Aromatic	Suwon 393
HM2	Hyangmibyeo-2	Aromatic	Suwon 413
KG1	Hwasunchalbyeo	Glutinous	Suwon 384
KG2	Hangangchalbyeo	Glutinous	Milyang 167
KK1	Heukjinjubyeo	Black	Suwon 477
KK2	Heuknambyeo	Black	Suwon 415
KK3	Heukkwangbyeo	Black	Iksan 427
GT1	Ilpumbyeo	Premium	Suwon 355
GT2	Taebongbyeo	Premium	Cheolwon 59
GT3	Hwasangbyeo	Premium	Suwon 330
GT4	Gopumbyeo	Premium	Suwon 479
GT5	Samkwangbyeo	Premium	Suwon 474
GT6	Choochungbyeo	Premium	Akkibari

Table 5.2 - Prediction models developed for aromatic descriptors of rice flavor as functions of the volatile components (ppb)

Popcorn	= 28.35 - 4.39[guaiacol] - 15.99[(<i>E,E</i>)-2,4-decadienal]	($r^2=0.64$)
Cookedgrain	= 21.73 - 0.92[2-heptanone] - 0.76[2-acetyl-1-pyrroline] + 3.76[naphthalene] - 15.32[(<i>E,E</i>)-2,4-decadienal]	($r^2=0.91$)
Starchy	= 17.38 - 13.53[(<i>E</i>)-2-hexenal] - 0.45[(<i>E</i>)-2-heptenal]	($r^2=0.64$)
Woody	= 25.95 - 2.33[benzaldehyde] - 3.24[p-menthan-3-one] - 3.34[naphthalene]	($r^2=0.68$)
Smoky	= 1.45 - 4.14[1-pentanol] + 3.08[naphthalene] + 14.56[(<i>E,E</i>)-2,4-decadienal]	($r^2=0.67$)
Grain	= 9.86 + 1.00[p-xylene] - 2.22[guaiacol] + 4.78[naphthalene] - 8.04[(<i>E,E</i>)-2,4-decadienal]	($r^2=0.79$)
Corn	= 6.27 - 0.57[2-heptanone] + 1.29[nonanal] - 11.73[(<i>E,E</i>)-2,4-decadienal]	($r^2=0.74$)
Hay-like	= 17.00 + 23.12[(<i>E,E</i>)-2,4-nonadienal] - 44.91[(<i>E</i>)-2-decenal]	($r^2=0.57$)
Barney	= 8.12 - 0.05[hexanal] + 31.50[2-nonanone] + 5.42[naphthalene]	($r^2=0.77$)
Rancid	= 3.98 + 1.49[heptanal] - 2.06[guaiacol] - 5.76[decanal]	($r^2=0.64$)
Waxy	= 7.52 - 7.63[(<i>E</i>)-2-hexenal] - 3.03[guaiacol]	($r^2=0.85$)
Earthy	= 12.04 + 1.25[2-heptanone] - 16.26[2-nonanone] - 1.91[naphthalene] - 9.62[(<i>E,E</i>)-2,4-decadienal]	($r^2=0.71$)
Sweet-aromatics	= 14.17 + 11.71[(<i>E</i>)-2-hexenal] - 0.41[2-acetyl-1-pyrroline] + 4.52[naphthalene] - 23.73[(<i>E,E</i>)-2,4-decadienal]	($r^2=0.95$)

Table 5.3 -Correlation analysis between aromatic descriptors and volatile components

Aromatic descriptor	Volatile compound	Correlation coefficient (<i>r</i>)*
Popcorn	p-xylene	-0.51
	decanal	-0.52
Starchy	(<i>E</i>)-2-hexenal	-0.73
	2-heptanone	-0.57
	heptanal	-0.66
	1-octen-3-ol	-0.50
	2-pentylfuran	-0.50
	3-octen-2-one	-0.66
	2-nonanone	-0.59
	(<i>E</i>)-2-nonenal	-0.55
	decanal	-0.49
Woody	p-xylene	-0.68
	benzaldehyde	-0.75
	guaiacol	-0.67
	decanal	-0.61
Cooked grain	hexanal	-0.74
	(<i>E</i>)-2-hexenal	-0.67
	2-heptanone	-0.77
	heptanal	-0.58
	(<i>E</i>)-2-heptenal	-0.64
	1-octen-3-ol	-0.74
	2-pentylfuran	-0.78
	octanal	-0.55
	3-octen-2-one	-0.67
	(<i>E</i>)-2-octenal	-0.65
	2-nonanone	-0.55
	p-menthan-3-one	-0.50
	(<i>E</i>)-2-nonenal	-0.78
	dodecane	-0.68
	1-pentanol	-0.58
Smoky	(<i>E,E</i>)-2,4-decadienal	0.51
Grain	heptanal	-0.54
	naphthalene	0.60
Sulfury	naphthalene	-0.68
	(<i>E,E</i>)-2,4-nonadienal	-0.63
Corn	benzaldehyde	0.52
	guaiacol	0.54
	nonanal	0.56
	(<i>E,E</i>)-2,4-decadienal	-0.56
Nutty	(<i>E,E</i>)-2,4-nonadienal	-0.50
Dairy	heptanal	0.56

*Data in bold letter are significant at $P < 0.1$, all others are significant at $P < 0.05$

Table 5.3 -Correlation analysis between aromatic descriptors and volatile components (continued)

Aromatic descriptor	Volatile compound	Correlation coefficient (<i>r</i>)*
Hay-like	(<i>E</i>)-2-decenal	-0.62
Barny	hexanal	-0.77
	(<i>E</i>)-2-hexenal	-0.59
	2-heptanone	-0.57
	heptanal	-0.59
	(<i>E</i>)-2-heptenal	-0.72
	1-octen-3-ol	-0.71
	2-pentylfuran	-0.72
	octanal	-0.66
	3-octen-2-one	-0.62
	(<i>E</i>)-2-octenal	-0.59
	p-menthan-3-one	-0.64
	(<i>E</i>)-2-nonenal	-0.68
	dodecane	-0.81
Rancid	1-octen-3-ol	0.49
	guaiacol	-0.55
	naphthalene	-0.53
Waxy	(<i>E</i>)-2-hexenal	-0.70
	p-xylene	-0.67
	2-heptanone	-0.62
	heptanal	-0.57
	2-acetyl-1-pyrroline	-0.54
	benzaldehyde	-0.63
	3-octen-2-one	-0.64
	guaiacol	-0.74
	nonanal	-0.71
	(<i>E</i>)-2-nonenal	-0.58
	decanal	-0.66
Earthy	(<i>E,E</i>)-2,4-decadienal	-0.49
Sweet aromatics	guaiacol	0.50
	(<i>E,E</i>)-2,4-decadienal	-0.61

*Data in bold letter are significant at $P < 0.1$, all others are significant at $P < 0.05$

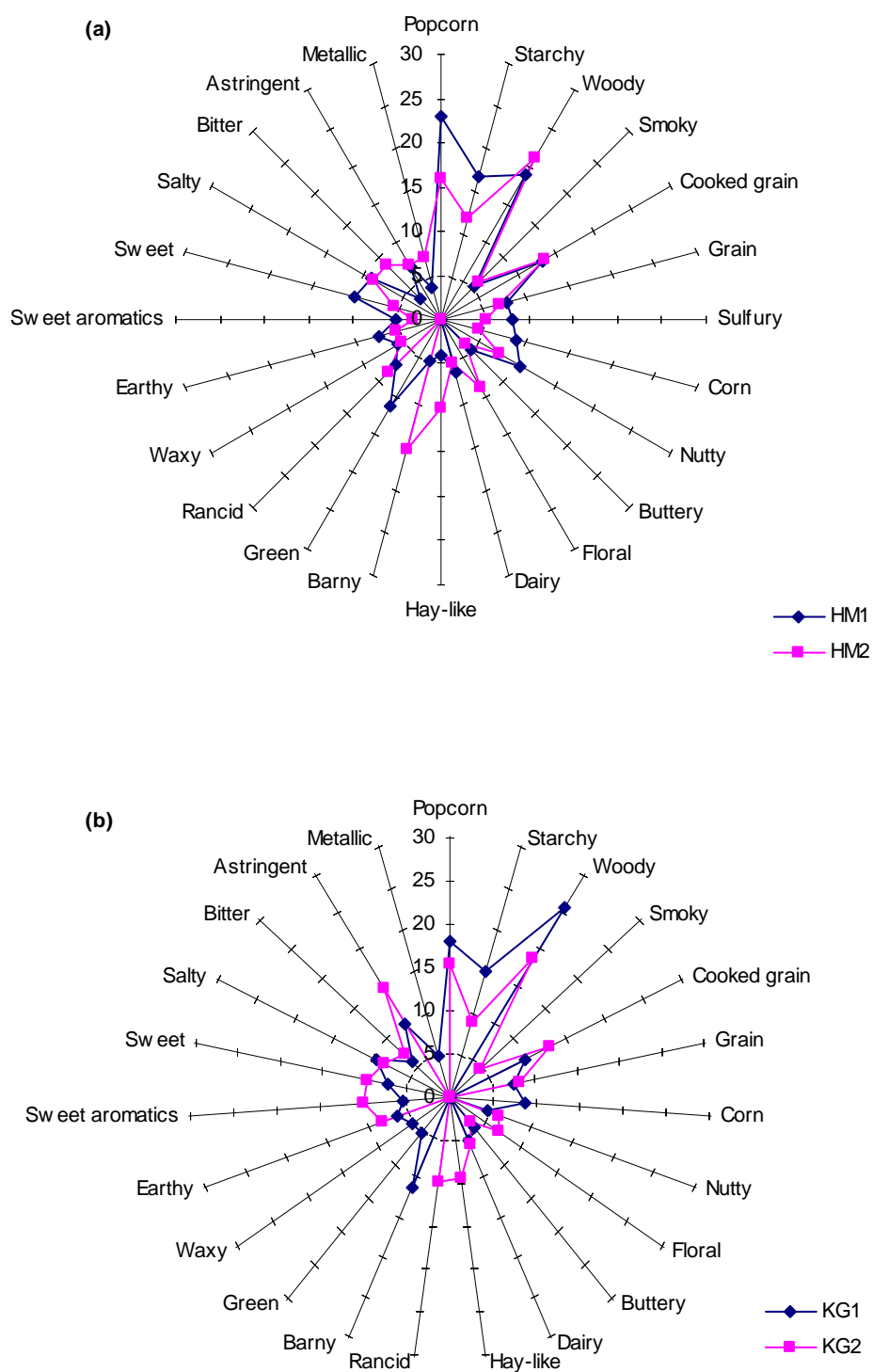


Figure 5.1- Spider plot of the mean intensity of descriptors found in (a) aromatic rice (b) glutinous rice (c) black rice (d-e) premium rice. See sample codes in Table 5.1

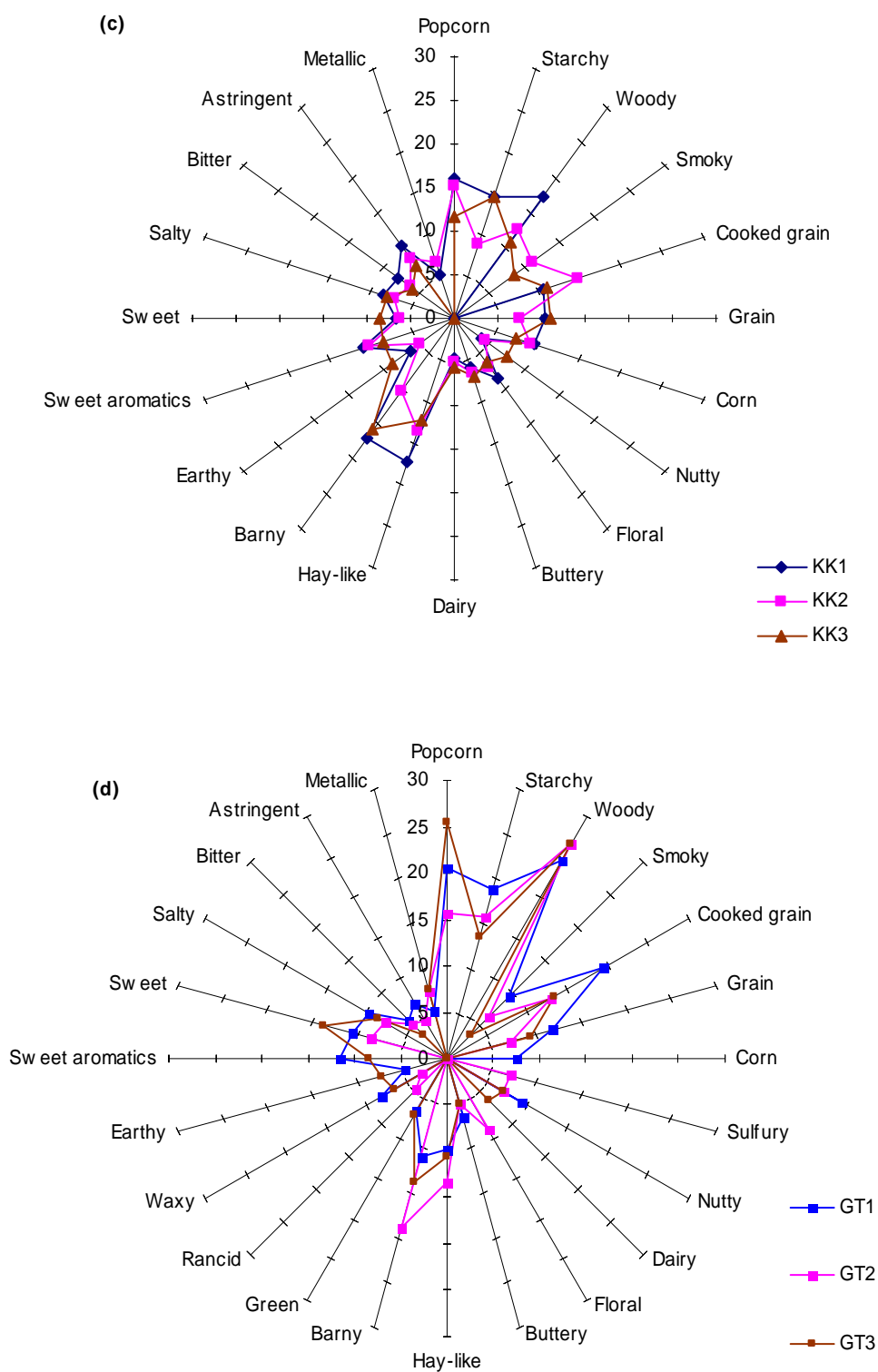


Figure 5.1- Spider plot of the mean intensity of descriptors found in (a) aromatic rice (b) glutinous rice (c) black rice (d-e) premium rice. See sample codes in Table 5.1

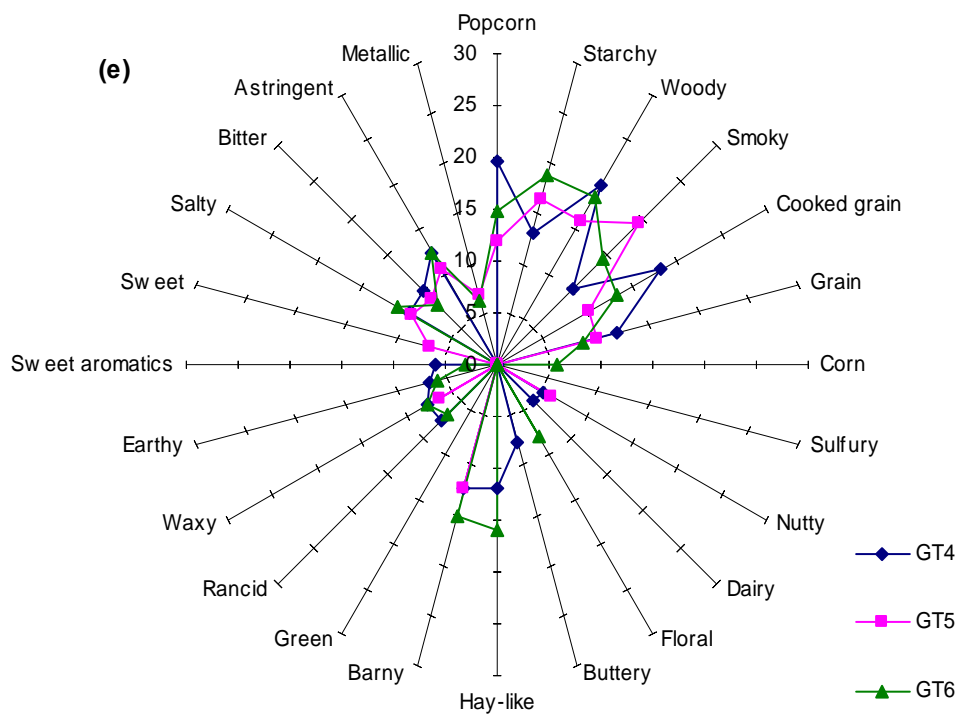


Figure 5.1- Spider plot of the mean intensity of descriptors found in (a) aromatic rice (b) glutinous rice (c) black rice (d-e) premium rice. See sample codes in Table 5.1

CHAPTER 6

SUMMARY AND CONCLUSION

Rice (*Oryza sativa* Linn.) is the predominant dietary energy supply for more than half of the world population. Global rice trade has progressed over the past years in response to the increase of rice consumption, partly due to a rising population in Asia and a widespread of ethnic cuisine popularity in Western countries. In the United States, high-quality imported aromatic rice plays an integral part of domestic rice disappearance. Providing consumers with desirable flavored rice provides an incentive for systematic investigation of flavor quality evaluation necessary for the U.S rice breeders. This study was conducted in three parts.

The first study (Chapter 3) developed a lexicon for flavor profiling of rice samples employing the descriptive Spectrum™ methodology. Seventeen rice samples out of a wide array of 36 domestic and imported rice samples were used for lexicon generation whereas the other 16 samples were used for validation. Overall, 33 cooked rice samples were fully characterized for flavors using 24 established descriptors by eight trained panelists. Of these 24 descriptors, 18 descriptors were significantly present in most rice samples. Rather weak but significant correlations were observed among these 18 descriptors. Principal component analysis (PCA) with Varimax rotation applied to significant descriptive terms was not effectively able to reduce attributes that could adequately described the rice samples since six components were required to explain 74.7 % of the total variation. Three components accounted for only 54.2 % of the variation in the data. Some attributes necessary to fully describe unique characteristics of a

particular type of rice would be lost if there was further elimination of descriptors in order to get a smaller set of components. The Spectrum™ technique with universal scale basis facilitates the addition of new terms for better characterization of new rice types in the future.

The second study (Chapter 4) determined the orthonasal detection thresholds of selected important volatile compounds found in cooked aromatic rice by eight trained panelists using the ascending methods of limits. Since food is a multi-component medium, partially deodorized rice was developed and used as a medium in comparison with water and corn starch. Best estimate threshold (BET) values of hexanal, heptanal, octanal, nonanal, (*E*)-2-nonenal and 2-acetyl-2-thiazoline measured in water were the lowest among the three matrices, followed by partially deodorized rice and corn starch media respectively. However, threshold values determined in water by this panel were mostly lower than published values of the same compound indicating the increased sensitivity of the panelists. Partially deodorized rice was identified and quantified for the selected volatile compounds compared to the control using GC-MS. All selected compound concentrations except (*E*)-2-nonenal were lower than in the control. Odor activity values of each compound (its concentration divided by its odor threshold) in partially deodorized rice, did not surpass the common value of one suggesting the matrix effect between rice medium and the spiked volatile compounds. Determination of threshold in the same matrix as the sample under investigation is yet necessary and realistic.

The third study (Chapter 5) linked the developed sensory descriptors to the odor-active compounds characterized by GC-O using stepwise multiple linear regression. Thirteen specialty rice samples were evaluated by a trained panel employing the Spectrum™ descriptive analysis with a lexicon developed in the first study. Nineteen aromatic descriptors in cooked samples were regressed against 25 important volatile compounds derived from the same set of rice by

GC-O. Significant prediction models as indicated by the coefficient of determination (r^2) were developed for most aromatic descriptors including popcorn, cooked grain, starchy, woody, smoky, grain, corn, hay-like, barny, rancid, waxy, earthy, and sweet aromatics. (*E,E*)-2,4-decadienal, naphthalene, guaiacol, (*E*)-2-hexenal, 2-acetyl-1-pyrroline, and 2-heptanone contributed most to these sensory descriptors. These generated models could serve as prescreening tools for rice breeders in targeting the important flavor traits which in turn provide a first step in achieving the goal for systematic methods of flavor quality evaluation.

This project identified 18 key descriptors that differentiate flavor among types of rice and related sensory aromatic descriptors to specific volatile compounds in the rice selections. This information coupled with the chemistry of flavor preference will be useful as a database in developing a rapid analytical method for use by rice breeders to screen progeny with the desired traits for superior flavor quality.