NEWER METHODS IN SENSORY ANALYSIS

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Background.

Food aroma is complex with literally hundreds of chemical compounds having the possibility of being significant to the aroma component of food flavor. Often, the compounds of greatest interest to flavor have threshold detection levels in the parts per trillion (ppt). Determining the actual combination of compounds of real importance to food aroma has been and remain a very real challenge to the sensory scientist.

This paper will look selectively at some of the newer tools for flavor research, with emphasis on aroma methodologies that have become particularly useful in the last 5-10 years. Marsili (1997) reviewed advances in the techniques for analysis of food aroma compounds. Off particular significance to this discussion are:

- Chemometrics
- Olfactometry dilution GC analysis
- Advances in head space analysis
- Electronic Noses

Major attention will be given to the electronic nose and its applications.

Chemometrics

Chemometrics includes a wide variety of multivarient analytical techniques that have become powerful tools for differentiating complex, multivarient sets of information. This information may come from a wide variety of sources that include

- Descriptive sensory analysis
- Chemical analysis
- Electronic nose data analysis

Multivarient analysis has been divided in two broad categories: (a) untrained and (b) trained. Untrained techniques include cluster analyses, principal components analyses (PCA) and utilizes data without indicating the relationships between data points.

Chemometrics has been a powerful tool for the interpretations of a wide range of multivarient sets of information. These include

- Solid phase microextraction (Bicchi et al, 1997; Nakai et al, 1999)
- Gas chromatographic data
- Infra-red data (Al-Jowder et al, 1999)
- Spectrophotometric data
- Electronic nose data Bartlet et al (1997)

Chemometrics has proven to be essential to the interpretation of electronic nose data,

Solid Phase Microextraction (SPME)

Solid phase micro-extraction (SPM has been shown to be a very useful method for the evaluation of head space aroma compound by gas chromatography and olfactometry. A number of investigators have used SPME for meat research (Arnold and Senter, 1998; Brunto et al, 2000; Nakai et al, 1999 and Ruiz et al, 1998).

Olfactometry

Gas Chromatography (GC) and GC/MS have become standard methods for the separation of identification of volatile aroma compounds. Olfactometry is the evaluation of the aroma of compounds that eluted from the gas chromatographic (GC) analysis of aroma compounds. Olactometric dilution analysis has become as useful tool in flavor research. This has been frequently combined with SPME to confine the analyses to headspace aromatic compounds. With GC olfactometry dilution analyses, the Odor activity value (OAV) is a measure of the ratio of the concentration of the compounds in the sample to the lowest dilution at which the compound can be detected by sensory evaluation of the compound coming off a GC column. This value at is utilized to determine the relative significance of the compound to the aroma of the food. A number of investigators have proposed the higher the OVA, the more significant in the compound. However, since most food aromas depend upon the balance of 4 or more compounds to provide a specific attribute, interpretation of OAV values needs to be made with care.

Olfactometry has been applied to meat by a number of investigators, including Baek and Cadawallander, 1997; Braggins, 1996; Chung and Cadwallander, 1995; Farkas et al, 1997; Guillard, 1996; Kerscher and Grosch, 1997 and Siegmund and Pfannhauser, 1999; Stanke (1995).

Generally, GC dilution olfactometry has been considered to be more useful that just GC-olfactometry alone. Siegmund and Phannhuaser (1999) showed that the results of GC dilution olfactometry could be correlated with those obtained by an electronic nose.

Electronic Analysis

The electronic nose was introduced commercially in the U.S.A. in 1994, and has gained wide use in quality control of food aromas. Recently development work has been presented on the development of the electronic tongue. Livigne at al (1999) discussed instruments based either on electrodes that measures and expresses taste quantitatively by measuring the potential across a series of lipid/polymer membranes as they react with analytes in solution, or on beads made from blends of polymers (representing electronic taste buds) that show changes in optical properties in response to analytes, resulting in colorimetric/fluorescent patterns that are captured by semiconductor arrays and interpreted using multi-variant analysis.

The Electronic Nose.

Arrays of electronic sensors, capable of detecting and differentiating complex mixtures of volatile compound, have been utilized to differentiate aromas of food and related materials. These sensor arrays have been dubbed "Electronic Noses" and have been commercially available in the USA for the past 5-6 years.

Gas sensors were first reported for evaluating the production of off odors in fish in 1984 (Storey, et al. 1984), and have become widely used in the past 5 years for quality control. The electronic nose ca be defined as "an instrument which comprises an array of electronic chemical sensors with partial specificity and an appropriate pattern recognition system capable of recognizing simple or complex odors" (Bartlet et al., 1993). The similarities between the biological olfactory pathway and the 'electronic nose' have been described by Pearce (1997a, b).

The nature and potential use of these instruments has been reviewed by several authors (Bartlett, et al. 1997; Hodkins, 1996; Hurst, 1999; Lavigne et al, 1999 Marsili, 1997; Prosser at al., 1999, Shaller and Bosset, 1998).

Sensors.

Conducting organic polymers and metal oxide sensors have been used most commonly in Electronic Noses. Of all of these detectors, only the mass detector provides any indication of the chemical basis upon which the Electronic Nose differentiates aromas. Recently, there has been a tendency to use a combination of sensor types. Common sensors include:

- Conducting organic polymers
- Metal oxide (doped or undoped semiconductors)
- Quartz crystal microbalance
- Surface acoustic wave
- Optical glass fibers
- Electro-chemical
- Mass detector
- Combination of sensors

Advantages and disadvantages of the most common sensors:

The sensors currently most commonly used are the organic polymers and metal oxides. In the past two years there has been an increase in the use of mass detectors.

Organic polymers

Advantages of organic polymer sensors include:

- Sensors are more selective
- Easier to miniaturize and allow for more sensors in an array
- Low power consumption
- Can use inert carrier gas

Limitations of organic polymer sensors include:

- High cost of sensor arrays
- Slow response time
- Sensors not always reproducible - batch to batch
- Highest degree of drift over time
- Most sensitive to water

Metal oxide sensors.

Advantages of metal oxide sensors include:

- Least sensitive to moisture
- Least amount of 'drift' over time
- Electrical response = log of the intensity of the stimulus
- Quick recovery time

Limitations of metal oxide sensors include:

- Poor selectivity - requiring arrays of sensors to aroma differentiation
- High power consumption
- Loss of sensitivity in presence of a high concentration of a polar compound

Mass Detectors.

Advantages for mass sensors include:

- Can provide a basis for the chemical basis of aroma differentiation
- Can evaluate on the basis of selected mass units, thus eliminating interference with water or alcohol
- Essentially no drift in signal
- Can be used in different selective ion modes

Disadvantages of mass sensors include:

- High cost
- Lower sensitivity for some instruments
- Higher maintenance costs

Commercial electronic noses currently in use include:

- Alpha MOS - Fox - metal oxide 6-18 sensors*
- Aroma Scan - polymer 32 sensor
- Bloodhound - polymer 6-12 sensors
- Moses ii - multiple sensors*
- Hewlett Packard - head space with mass detector
- Neotronics - polymer 12 sensors*
- Perkin-Elmer - Quartz Crystal sensors

*Combinations of detectors available

Strengths of Electronic Noses.

The many similarities between organoleptic olfaction and the electronic nose have been reviewed in detail by Pearce (1997a, b). Electronic noses are highly sensitive, with threshold detection levels generally in ppm and ppb ranges. The sensitivity of the electronic nose appears to be similar to that of the human nose for 30 food aroma compounds with different chemical and aroma characteristics (Harper and Kleinhenz, 1998). Table 1 shows some of these results comparing orthonasal threshold values and electronic nose detection threshold level for 6 of the 30 compounds in water. A compound with a human threshold level of ppm also had an Electronic Nose (using 12metal oxide sensors - Fox 3000) in the ppm levels, and compounds with reported human orthonasal threshold values in the ppb also had ppb threshold levels when tested with the electronic nose. Another major strength of the electronic nose is its ability to relate to human sensory panel evaluation of food products (Hurst, 1999).

In addition the electronic nose provides an objective evaluation of the aroma of a product - not subject to human variability and is capable of running a large number of samples at one time without having the problem of fatigue that affects human sensory analysis.

Current Weaknesses of the Electronic Nose.

Electronic Nose technology is a technology in development, with changes to both hardware and software occurring at a rapid rate. Current weakness of the most commonly used instruments include:

- Sensor drift
- Limited sensor sensitivity
- Sensor poisoning
- Lack of relationship between odor quality and intensity
- High sensitivity for some systems to water and other polar compounds that may be present in high concentrations (such as alcohol)
- Sensor life limitations and high cost for some sensor systems
- No absolute calibration currently available
- Gives only part of the flavor picture

Some of the limitations of the electronic nose are also limitations for biological olfactory systems (Pearce, 1997b). including sensor poisoning and providing only part of the flavor.

The use of mass detectors as sensors have minimized drift, eliminated interference with water and have permitted an understanding of the chemistry involved in the differentiation of aromas. Uses of olfactometry and electronic noses is expect to increase in the future.

Applications.

Electronic Noses are being widely used by some companies as a quality control instrument (Barlett, et. al., 1997; 1997; Koopal, et al, 1999; Marsilli, 1997a; Porretta, et al, 1997; Prosser et al. 1999). There is some evidence that the sensors differentiate aromas on the basis of relatively few compounds and in the future a relationship between specific chemicals and a single flavor attribute may be achievable through the use of mass detectors combined with GC/MS.

Pihksgard et al (1998) compared GS/MS, GC-olfactometry, sensory analysis and an electronic nose for the aroma profiles of sugar samples from a refinery. The samples were analyzed by means of headspace GC-MS/FID, GC-olfactometry, sensory analysis, and an electronic nose. They found no direct correlation between odor intensity of compounds identified by GC/MS and headspace concentration. The electronic nose was able to differentiate between the samples and was also able to rank them according to the amount of volatile substances in them and gave some differentiation of the basis of sensory analysis

Food Industry Applications.

There are a number of reports of application in the food industry, especially in respect to quality control. Broad applications include: (a) Quality assurance - raw materials, (b) Monitoring of cooking processes, (c) Monitoring fermentations, (d) Process monitoring and (e) Study of effects of food storage. Review of various applications to food have been presented by Bartlett et al 1997; Hurst, 1999; Schaller et al 1998,)

Applications related to meat and related products include:

- Meat inspection (Hall, 1997)
- Monitoring sausage fermentation ((Barlett, et al, 1997)
- Freshness of fish (Ashima, 1991; Bradley, 1996; Storey et al, 1984)
- Spoilage in vacuum packed meat (Blixt and Borch, 1999)
- Spoilage in poultry (Arnold and Senter (1999)
- Ham curing (Abas, et al, 1999)
- Boar taint in pork (Annor-Frempong et al, 1998)
- Fish quality (Newman et al, 1999; Luzuriaga and Balaban, 1999a, b)

Other food applications include:

- Apple maturity (Young et al, 1999)
- Evaluation of beer and wine (Tomlison, 1996; Tomlinson, et al, 1998
- Classification of grains (Borgesson, et al; 1996; Johnson, et al., 1997)
- Sugar quality (Kaipainen, et al. 1997)
- Coffee classification (Bartlett, et al, 1993; Springett, et al.; 1990; Tan, et al, (1995)
- Differentiating aromatic rice (Hyung, et al. 1998)
- Classification of blueberry ripeness (Simon, et al 1991)
- Freshness of orange and other citrus juices (Hodgkins, 1995; Poretta, 1996; Tamera, et al. 1994)
- Differentiation of cheese and following cheese ripening (Harper, et al., 1996; Jin and Harper, 1996; Muir, et al, 1997; Zannoni, 1995)

Reported applications continue to grow and can be expected to expand in the future.

Non-food applications, which are growing also, include:

- Evaluation of bioprocesses (Mulville, 1997;Ping, et al, 1997; Namdev, et al., 1998)
- Monitoring environmental quality (Alexander, 1997)
- Tobacco industry (Su, 1997)
- Chemical industry (Moy and Collins, 1996)

Other applications that have been mentioned in general reviews include the automotive industry, paper industry, and in the medical field both in microbial classification and medical diagnosis.

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Meat – EN

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Table 1: Comparison of detection threshold levels as determined by a metal oxide electronic nose (Fox 3000) and reported orthonasal detection threshold level in water.

Compound	Electronic Nose Threshold	Orthonasal reported threshold