Subject: Food Technology under Graduate course

Core course 14 : Food quality and Sensory Evaluation

Unit 3: Olfaction (part - 2)

e- Content Topic: Odour measurement techniques.

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Introduction

Human olfaction is a protective sense as it allows the detection of odours which helps for food acceptance, recognizing danger and disliking the unpleasant. Odours are mixture of light and small molecules that, coming in contact with various human sensory system, also at very low concentrations of inhaled air, are able to stimulate an anatomical response. The experienced perception is called odour . Odour assessment is a key point in food product processes to arraive at a quality product, likable and well accepted product too. Although the human olfactory system is still regarded as the most important and effective analytical instrument for odour evalution, the demand for objective methods still exist. Many a time, the comparison of subjective and objective methods of odour evaluation has been researched to arraive at a more confident, realistic picture of the odours and the compounds responsible for the sensing of odours. The analysical methods extension has led to the development of sensor based machines, olfaction potentially imitating the biological system.

The odour measurement is essential for odour regulation and control. Odour emission often consists of a complex mixture of many odourous compounds. Analysis of individual chemical compounds present is usually not practical. More often, complexity of odour involves psychological and physiological response, perceives the odour as pungent, ethereal, aromatic, pepperminty, musky, camphorous, putrid and so on referring to the source. Therefore, the analytical methods can reflect chemical compound responsible for these perceptions. As a result, odour sensory methods, instead of instrumental methods are normally used to measure such odours. Broadly, odour measurement can be carried out by two methods.

1. Sensory methods
2. Instrumental methods

Repeatability, reproducibility and accuracy have to be accomplished with any method. The sampling procedures, sample containers, olfactometer construction & operation, olfactometer & assessor interface, the odour testing room, methods of data processing, training of panel and consistency in their performance are of important concern for analysis.

1. Sensory methods:

Sensory measurements employ the human nose as the odour detector and human odour is directly experienced which is related to properties. Sensory methods are available to monitor odour both from source emissions and in the ambient air. These two diverse circumstances require different approaches for measuring odour. The sensation of odour has four properties related to threshold and tolerance. They are odour concentration, odour intensity, odour quality and hedonic tone.

Thus sensory measurements techniques comprise of two categories.

1. Quantitative measurements which couple the nose with some instrumentation.

2. Parametric measurements in which the nose is used without any other device

Instrumental sensory measurements employ the human nose in conjunction with the instrument, called olfactometer. This dilutes the odour sample with odour-free-air according to precise ratios, to determine odour concentrations. Odour concentration is an odour's pervasiveness. To measure odour sensation, an odour is diluted to certain amount to reach a **detection or recognition threshold**. The detection threshold is the concentration of an odour in air when 50% of a population can distinguish between the odour sample and odour free blank. The recognition threshold is the concentration of an odour in air in which 50% of a population can discern from an odourous sample and odour free blank. The recognition threshold is usually a factor of a 2 to 5 times higher than the detection threshold. To establish the odour concentration, an olfactometer is used which employs a group of panelists. The olfactometric measurement is affected by the olfactometer design, test procedure, differing sensitivity of observers and data quality. The materials used in olfactometer construction should not cause sample contamination. Low absorbency materials such as stainless steel, teflon or glass are used and internal surface areas are minimized. The **test procedure** followed should have a proper choice of samples presentation to the panel, considering the olfactory adaptation in panelists. An ascending order of strong samples is preferred so that the panelists can detect higher dilutor samples followed by lower dilution that means to say stronger samples.

Among the two standardised methods for the presentation of odour sample to the panel, single port used method is simpler than the forced choice multiple ports method. Sampling odour mixtures at different dilutions are presented to a group of selected panelists for sniffing and their responses are recorded. Generally, the first mixture presented to a group of an odour panel is diluted with a very large volume o air in order to be undetectable by the human nose. In subsequent sample presentation, the volume of diluent is decreased by a predetermined and

constant factor. After having set the factor, it is possible to create a geometric progression of dilutions useful to describe the logarithmic relation between odour intensity and concentration. The process continues until each panelist positively detects an odour in the diluted mixture. At this stage the panelist responses has reached the detection threshold for that odour. This threshold is expressed as threshold odour numbers and used to calculate the concentration of the odour in terms of European odour units (ou_E/m^3).

Panelists are qualified examiners used as sensors in olfactometric analyses and their olfactive response (odour threshold) is the measured parameter for calculating odour concentrations. These panelists are selected according to standardized procedure to minimize the difference among their detection. The panelists should be the individuals with average olfactive sensitivity, who constitute a representative sample of human population. The screening is usually performed using reference gases and normally n-butanol odour threshold in a range of 20-80ppb is the accepted limit with 40ppb+2.3. Panelists must be continuously screened and trained. Panelists with illness, cough, cold, allergy etc. are eliminated.

In order to present the odour sample to the panelists, samples must be collected using specialized sample bags. Sampling needs particular attention to avoid sample losses due to sorption on the container. The materials used for odour containers should be odourless, stable to physical and chemical reactions and has low permeability. Stainless steel, polytetrafluoroethylene (PTFE), tetrofluoroethylene hexafluoropropylene copolymer (Teflon), Polyvinylfluoride, Polyterythalic ester copolymer. Nalophan and glass are considered suitable materials for odour sampling.

The most accepted technique for collecting odour samples is the **lung technique**. The sample bag is placed in a sealed drum and vacuum is placed on the drum, which fills the sample bag. As the bag expands, it draws the sample from the source into the bag. Critically, all components which touch the odour sample must be odour free.

The **data quality** is achieved when standard sampling techniques are followed, representative sample of human population is used, quality control protocols are followed during the conduct of experiments. The statistical analysis of the data specifically ANOVA which reflects the variance between group and within group has to be adopted.

The other instrumental based quantification of odour samples is studied using Gas chromography-olfactometer (Gc-o).The Gc – olfactometer consists of a tradional Gc where a split column distributes the eluate between a conventional detector, such as flame ionization detector (FID) or a mass spectrometer (MS) and a sniffing port where a properly trained person or panel could detect the active odour species. The commercially available olfactory ports are glass or PTFE cones, fitting the nose shape. The eluate is delivered through a dedicated transfer line which is heated to avoid the condensation of semivolatile analytes. In order to present the

nasal mucous membrane drying, during long time analysis, auxillary gas (humid air) is added to the eluate. The sensory responses are recorded in an olfactogram. The eluate splitting occurs allowing the analytes to reach both human and instrumental detectors simultaneously,in order to compare the chromatogram with the olfactogram. The Gc-o technique is widely used for the evalution of food aromas.

The second approach for odour evalution by **parametric sensory measurements** has the advantage of being quick to obtain at relatively low cost, as no particular equipment is required. The subjective measurement of parameters of odour perception by the well trained panel. The parameters include **odour character, odour intensity and hedonic tone.**

Odour character, often called odour quality, is a nominal scale of measurement. Odours can be characterized using a reference vocabulary with a standard list of descriptor terms.

Odour intensity is the relative strength of the odour above the recognition threshold (suprathreshold). Odour intensity is measured using several methods including descriptive category scales, magnitude estimation and reference scales. There are several scales that usually employ 3-10 categories and panelists must assess the intensity of the sample according to the specified scale. It is a verbal description of an odour sensation to which a numerical value is assigned. For example, the odour intensity is described as

No odour - 0

Very weak - 1 (odour threshold)

Weak - 2

Distinct -3

Strong – 4

Very strong – 5

Intolerable–6

With a numerical value of 0– 6: This test is followed in the laboratories and the odour intensity is done by a series of suitably trained panelists.

Hedonic tone is the process of scaling odours on a scale ranging from extremely unpleasant via neutral up-to extremely pleasant. The method is followed by serving the different dilution sample through an olfactometer to the panelists. If the panelists detects an odour, the hedonic odour tone of the perceived concentration must be evaluated according to the category scale. It is described as

Extremely unpleasant - 4

Very unpleasant – 3

Fairly unpleasant – 2

Unpleasant – 1

Neither pleasant or unpleasant 0

Pleasant +1

Fairly pleasant +2

Very pleasant +3

Extremely pleasant +4

Moreover, it is important to note that perception of an odour may change from pleasant to unpleasant with increasing concentration, intensity, time, frequency and previous experience with a specific odour. All these factors are determining the response.

The overall sets of qualities are sometimes called as the FIDAL factors, (Frequency, Intensity, Duration, offensiveness and Location).

2. Instrumental methods.

Though human perception of odour is desirable and regarded as most important and effective analytical instrument for odour evaluation, still there are problems in comparing different persons experience in quantifying the odour. As a first step, olfactometer called the dilution instrument has been coupled with the sensory method to provide to the panel of human assessors. Later, in 1988, **electronic sensing** technologies have undergone development for the technicalities as well as for commercial applications. Electronic sensing equipment called e -nose or electronic - nose is capable of reproducing human senses using sensor arrays and pattern recognition systems. **E-noses** can detect and recognize odours and flavours. The stages of recognition process are similar to human olfaction and are performed for identification including data storage and retrieval. Further, the development of a new olfaction system, called **Electronic mucosa** is based on advanced pattern recognition algorithms for space and time classification of odourants (Che Harun etal, 2009). E-Nose has been applied in the space shettle to monitor air quality in the cabin (Willers etal, 2004).

E-Nose: The electronic nose consists of head space sampling, sensor array and pattern recognition modules to generate signal pattern that are used for characterizing odours in terms of perception as a global fingerprint. The major parts of E-nose are a sample delivery system, a detection system and a computing system. The **sample delivery system** enables the generation of the headspace ie.volatile compounds of a sample. The system then injects this headspace into

the detection system. The sample delivery system is essential to guarantee constant operating conditions. **The detecting system** consists of a sensor set, a reactive component of the instrument. When volatile compounds come in contact, the sensors react through the interfaced transducer electrical signal. A specific response is recorded. A specific response is recorded by the electronic interface transforming the signal into digital value. Recorded data are then computed based on statistical models. In most E-noses, each sensor is sensitive to all volatile molecules, but each in their specific way. However in bio-E-nose receptor proteins cloned from biological organisms eg. humans respond to specific odour molecules through binding. The more commonly used sensors are metal-oxide semiconductors devices. Beside this, the sensors are conducting polymers, polymer composites, quartz crystal resonators, and surface acoustic wave. Some devices combine multiple sensor types in a single device. In recent years, other types of E-noses have been developed that utilize mass spectrometry or ultra gas chromatography as detection system. Several studies concerning the use of nanomaterials as gas sensor materials have been reported.

The Computing System combines the responses of all of the sensors which represent the input data. This instrument performs global fingerprint analysis and provide results that can be easily interpreted. E-nose results can be correlated to those obtained from sensory panel, GC, GC/MS results.

The analyses with the E-nose as a first step, needs to be trained with qualified samples so as to build a database of reference. Then the instrument can recognize new sample by comparing volatile compounds fingerprint to those contained in the database. The limitation is the analysis of odours made up of multiple different molecules which is normally detected by the device as different compounds. E-noses are mainly used in R&D labs and process and production departments. E-noses can be used in quality control mechanisms of conformity of raw materials, origin of vendor selection, and detection of contamination spoilage, adulteration and monitoring of storage conditions. In the processing, it can be applied for managing raw material variability, comparison with a reference product to narrow down the batch variation, scaling-up of process and monitoring the place safety.

Considering the different techniques of odour evaluation, no one of the described techniques alone is able to exhaustive information about the odours emissions from different food systems as well from different kinds of human activities that cause olfactory picture. Therefore, a comparison or an integration of the olfactory methods with the methods of sensorial analysis makes the tasks complete to get a realistic picture of odours.

Besides these, clinical test approach for detecting the odour identification ability of the panelists can be followed. However, the clinical testing can be time consuming and difficult to perform precisely. The major goal of sensory testing is to assess the chemosensory property. In this direction, simplified and standardized commercial kits are available. These can provide a reliable measure of olfactory ability. Tests of olfactory function can evaluate the threshold of

odour detection, identification and quantification. These tests include butanol threshold test, the university of Penssylvania smell identification test (UPSIT) and the sniffin stick test.

The butanol threshold test involves a forced-choice test using an aqueous concentration of butyl alcohol in one sniff bottle and water in the other. The person is asked to identify the bottle containing the odourant, with each nostril tested separately. After each incorrect response, the concentration of butanol is increased by a factor of 3 until the person either achieves 5 correct responses or fails to correctly identifies the bottle with 4% butanol. The detection threshold is recorded as the concentration at which the person correctly identifies the butanol on 5 consecutive trials.

The UPSIT involves 40 encapsulated odours in a scratch and sniff format, with 4 response alternatives accompanying each odour. The person takes the test alone, with instructions to identify, if not, atleast to guess the odour. The scores are compared against sex age related norms and the results are analysed. This test has excellent test- retest reliability.

A varint of the UPSIT called cross cultural smell identification test (CC-SIT) can be given in 5 minutes. This test gives a quick measure of olfactory function.The12-item CC-SIT has been developed using input on the familiarity of odours in several countries including China, Colombia, France, Germany, Italy, Japan, Russia, and Sweden.The odourants chosen include banana, chocolate, cinnamon, gasoline, lemon,onion, paint thinner,pineapple, rose, soap, smoke and turpentine.The odour identification was most consistant.The test is an excellent alternative in clinical setting. It is rapid and reliable But the disadvantage is that its brevity limits its sensitivity in detecting the subtle changes in olfactory function.

The sniff stick method use a series of reusable pen-like odour dispensing devices and tests odour threshold by a single staircase method. The overall evaluation of olfactory function is done by odour discrimination with forced choice among 3 of 16 different common odourants. For research studies, olfactory-evoked response method is normally used.

In **conclusion,** both subjective and objective standardised methods of analysis are described for odour evalution. The sensory analysis with the nose as the detector with device proves to be simple to conduct but critical in analysing the odour compounds. The combination of human with devices may be more fruitful with the single port olfactometer and qualified taste panelists. The instrumental analysis of odours is achieved using GC, GCMS, GC-O which is widely used for the evaluation of food aromas. However, olfactometry is time consuming and quite expensive. On the other hand, E-nose which analyses odours similar to human olfaction remain as a promising method of odour evalution. E-nose analysis present lower analysis costs and quick results. But the limitation is the large data base required for identification. However, no single technique satisfies the totality of odour evaluation and often a combination techniques are used for good end results. The clinical tests can be adopted for quick analysis and for patients.

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