E-Sensing Techniques In Food Quality Analysis

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1. Sensory - Profiling
2. Need for e –sensing techniques
3. Biological smelling and Tasting
4. E-Sensors and Technology
   • MOS- Metal oxide sensors
   • CP – Conductive polymers
   • QCM-Quartz crystal micro balance
5. E-sensing techniques
   • e-Nose
   • e-Tongue
   • e-Eye (IRIS)
6. Data analysis - Pattern matching systems
   • PCA
   • DFA
   • SIMCA
   • PLS
7. Applications - in Food with case studies
8. Limitations, errors, advantages
9. Conclusion
SENSORY ANALYSIS is a scientific discipline used to evoke, measure, analyse and interpret reactions to those characteristics of food as they are perceived by the senses

- *sight, smell, taste, touch and hearing.*

- *IFT*

- Discriminative
- Descriptive
- Consumer

- Human beings - Instruments - Calibration
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<th>Sensory Properties measured</th>
<th>Analytical techniques</th>
<th>Parameters</th>
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<td>Colour, Shape, Size</td>
<td>Hunter, E-eye SEM, Particle size analyzers</td>
<td>L, a, b and L*, a*, b* Dimensions</td>
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<td>Feel</td>
<td>Texture</td>
<td>Texture analyzers</td>
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<td>E-nose/GC/GC-O</td>
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<td>E-tongue/LC</td>
<td>Sensor responses</td>
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<td>Hearing</td>
<td>Structure/texture</td>
<td>Acoustic devices</td>
<td>Sound</td>
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Need for – E-sensing devices

- Requirement for fast, reliable - Online QC
  - For continuous monitoring to ensure consistent quality
- Numerical (Numbers)
- International standards
- Lack of facility for sensory analysis
- Non-availability of trained panel
- Time and Cost - Constraints
Electronic Nose
Biological smelling

Odourants ------ Nasal Cavity ----- Olfactory mucosa ------ Olfactory Receptor Neurons (activation of receptors) ------ CNS
Commercial E-nose diagram
Comparison of biological smelling and e-nose

Biological systems often serve as models for new technology. The electronic nose - called "Enose" - got its name because it operates like a human nose by containing a large number of sensors.
ELECTRONIC NOSE

With auto sampler
Alpha MOS Model: α-FOX 4000
Inside The E-Nose

Sensor matrix is composed of...

• 16 MOS (Metal-oxide semiconductor) sensors
• Specially designed stainless steel measurement chamber
• Air sample pump
• Cooling system

Key concepts of MOS sensor:
• Wide spectrum of responses (non-specific)
• Sensitive
• Durable
• Easy to replace
• Inexpensive
Sensor is a device that is able to provide a signal - proportional to the physical or chemical property to which the device responds.
METAL OXIDE SENSORS

- Metal oxides are semi-conducting materials (e.g., ZnO) which are gas sensitive.

- Sensors comprise of a thin layer of an oxide film deposited on a ceramic tube or plate and heated to temp. 175° to 450°C.

- Selectivity depends on catalytic amounts of a doping metal (Palladium for tin oxide sensors) introduced as a trace impurity on the sensor surface.
The resistance of the sensor thus decreases in the presence of an odor with size of the response depending on the

1. Nature of the odour molecules and
2. Types of metal oxide

Response time depends
- Reaction kinetics
- Head space nature
- Volume of measured headspace and
- Flow rate of the carrier gas.

Very sensitive and fast response

\[ R + O^- \xrightarrow{400^\circ C} RO + e^- \]
(odor molecule) (oxygen from metal oxide)
## Different sensor types and their working principles

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<th>Working Principles</th>
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<td>Conducting polymer sensors</td>
<td>Resistance and impedance</td>
</tr>
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<td>Electrochemical sensors</td>
<td>Conductance, intensity and voltage</td>
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<tr>
<td>Acoustic sensors (SAW, BAW, QMB, Cantilever)</td>
<td>Mass and frequency shift</td>
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<tr>
<td>Calorimetric sensors</td>
<td>Temperature</td>
</tr>
<tr>
<td>Optical sensors</td>
<td>Optical properties</td>
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</table>
Conducting polymer sensors

- Fabricated by deposition of very thin film of electrically conducting polymer which are electropolymerised (polypyrrole or polyandrine) with various counter ions in a solvent between two electrodes.
- Different types of electrochemically deposited sensors on silicon substrate.
- The basic co-polymers used are pyrrole, their derivatives, aniline derivatives, indole, and thiophene.
Quartz Crystal Microbalance (QCM)

The gas which is soluble in the coating will increase the added mass on the crystal and decrease the frequency of the oscillation according to

A coating (silicons, (poly-) glycols- which is gas sensitive) is deposited on a quartz support.

The sensing element is the coated quartz resonator.
## Comparison of sensors

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<tr>
<th></th>
<th><strong>MOS</strong></th>
<th><strong>CP</strong></th>
<th><strong>QCM</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sensitivity</strong></td>
<td>ppb-ppm</td>
<td>ppm</td>
<td>ppb-ppm</td>
</tr>
<tr>
<td><strong>Life time</strong></td>
<td>18-36 months</td>
<td>6-9 months</td>
<td>9-12 months</td>
</tr>
<tr>
<td><strong>Humidity sensitivity</strong></td>
<td>low – medium</td>
<td>high</td>
<td>high</td>
</tr>
<tr>
<td><strong>Desorption time</strong></td>
<td>Fast</td>
<td>Medium</td>
<td>medium</td>
</tr>
<tr>
<td><strong>Sensor drift</strong></td>
<td>Nil</td>
<td>More</td>
<td>Medium</td>
</tr>
</tbody>
</table>
Comparison of Sensor Characteristics

The selectivity is the capacity of a sensor to be sensitive to a specific compound.
**Important volatile compounds influencing flavor**

<table>
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<tr>
<th>Aromatic compound group</th>
<th>Example compound</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydroxy compounds</td>
<td>geosmin</td>
<td>Earthy</td>
</tr>
<tr>
<td>Aldehydes</td>
<td>hexenal</td>
<td>Apples</td>
</tr>
<tr>
<td>Ketones</td>
<td>2,3 butanedione</td>
<td>Celery</td>
</tr>
<tr>
<td>Acids</td>
<td>acetic acid</td>
<td>Vinegar</td>
</tr>
<tr>
<td>Esters</td>
<td>methyl anthranilate</td>
<td>Concord grape</td>
</tr>
<tr>
<td>Sulfur compounds</td>
<td>dimethyl sulfide</td>
<td>Asparagus</td>
</tr>
<tr>
<td>Oxygen hetrocycles</td>
<td>furaneol</td>
<td>Pineapple</td>
</tr>
<tr>
<td>Nitrogen hetrocycles</td>
<td>Pyroles</td>
<td>Peppers</td>
</tr>
<tr>
<td>Sulfur heterocycles</td>
<td>Thiophenes</td>
<td>Fried onion</td>
</tr>
<tr>
<td>Other compounds</td>
<td>iodine</td>
<td>Edible seaweed</td>
</tr>
</tbody>
</table>
Sensor output

Change in Resistance (ohms)

Time (s)
DISCRIMINATION OF ODOR BY E-NOSE - CORIANDER

R1-R5 Spicy Herbal
R6 Earthy Spicy
R7 & R8 Pleasant Cooling Floral Green

C1: 89.55%
C2: 10.45%
Aroma finger printing of three coffee beans from different origins
### Electronic Noses for Laboratory Analysis

<table>
<thead>
<tr>
<th>Odor analyzer Instrument</th>
<th>Sampling</th>
<th>Detection technology</th>
<th>Odor Analysis Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>FOX Electronic Nose</strong></td>
<td>Manual or</td>
<td>Metal Oxide</td>
<td>R&amp;D, product development, Quality Control</td>
</tr>
<tr>
<td><strong>FOX E- Nose brochure</strong></td>
<td>autosampler</td>
<td>Sensors</td>
<td></td>
</tr>
<tr>
<td><strong>GEMINI Electronic Nose</strong></td>
<td>Manual or</td>
<td>Metal Oxide</td>
<td>Quality Control</td>
</tr>
<tr>
<td><strong>GEMINI E-Nose Brochure</strong></td>
<td>autosampler</td>
<td>Sensors</td>
<td></td>
</tr>
<tr>
<td><strong>HERACLES Electronic Nose</strong></td>
<td>Manual or</td>
<td>Ultra Fast GC</td>
<td>R&amp;D, product development, Quality Control, process monitoring</td>
</tr>
<tr>
<td><strong>lab or on-field configurations</strong></td>
<td>autosampler</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>HERACLES E-Nose brochure</strong></td>
<td></td>
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</tr>
</tbody>
</table>

### High-End Electronic Nose for On-line Analysis

<table>
<thead>
<tr>
<th>AIRSENSE Electronic Nose</th>
<th>Sampling</th>
<th>Detection technology</th>
<th>Odor Analysis Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>AIRSENSE E-Nose brochure</strong></td>
<td>Online or</td>
<td>Soft Ionized Mass</td>
<td>Quality control, chemical identification, product development, process development and monitoring</td>
</tr>
<tr>
<td></td>
<td>off-line</td>
<td>Spectrometry</td>
<td></td>
</tr>
</tbody>
</table>
Many variants of e-noses

GEMINI Electronic Nose
HERACLES Electronic Nose

Lab configuration

On-field configuration
Electronic Tongue
TASTE Perception

**BASIC TASTES:**
Sweet, Sour, Salty, Bitter, Umami, Kokumi

**Tongue, Taste bud:** Receptors, basal and supporting cells.

- 4 types of papillae.
  - Foliate
  - Filiform
  - Fungiform
  - Circumvallate

~2000 taste buds.
• Flavor molecules fit into receptors on the microvilli at the top of the taste sensory cell, causing electrical changes that release transmitter onto the nerve ending at the bottom of the cell.
• The nerve carries taste messages to the brain by different ion channels.
Taste is related to chemical composition

1. Bitter- Compounds tend to have multiple nitrogen atoms
Taste is related to chemical composition

2. Sour compounds are acidic in nature

The sourness of substances is rated relative to dilute hydrochloric acid, which has a sourness index of 1.

By comparison, Tartaric acid has a sourness index of 0.70
Citric acid an index of 0.46
Carbonic acid an index of 0.06
Taste is related to chemical composition

3. Salty – Simply simple salts

\[ \text{NaCl}^{(s)} \rightarrow \text{Na}^{+}(aq) + \text{Cl}^{-}(aq) \]

Salts are formed between groups 1, 2 and 3

<table>
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<tr>
<th>Alkali metals</th>
<th>Group 1 – Li, Na, K, Rb, Cs, Fr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkali earth metals</td>
<td>Group 2 – Be, Mg, Ca, Sr, Ba, Ra</td>
</tr>
<tr>
<td>Halogens</td>
<td>Group 3 – F, Cl, Br, I, At</td>
</tr>
</tbody>
</table>

Salts made from group 1 and 3 taste salty to us

Salts made from group 2 and 3 do not
4. Sweet – Sweetness is often connected to aldehydes and ketones - which contain a carbonyl group (C=O).

Glucose

Aspartame

Sucralose

Saccharine
Taste is related to chemical composition

5. Umami – Savory
Associated with the amino acid Mono Sodium Glutamate (MSG)

Glutamic Acid

Mono sodium glutamate

• Mushrooms
• Tomato
6. **Kokumi - the sixth taste (?)**

It is sometimes translated as “heartiness” or “mouthfulness” and describes compounds in food that don’t have their own flavor, but enhance the flavors with which they’re combined.

These compounds include
- Calcium
- **Protamine** (found in milt, or fish sperm, which is eaten in Japan and Russia),
- **L-histidine** (an amino acid) and
- **Glutathione** (found in yeast extract).
• Thus a molecule is perceived by the receptors on our tongues is dependent on the chemical make-up of the molecule.

• Monell Chemical Center – Mechanisms and functions of taste and smell and define the broad significance of these senses in human health and disease
Electronic Tongue
Electronic tongue

Sensors

In the presence of dissolved compounds, a potentiometric difference is measured between 7 sensors and the reference electrode.

Each sensor has a specific organic membrane with interacts with ionic, neutral and chemical compounds present in the liquid sample in a specific manner.

Any interaction at the membrane interface is detected by the sensor and converted into electronic signal.

Chemical Modified Field Effect Transistor (ChemFET)

ChemFET sensor technology (Chemical modified Field Effect Transistor) using potentiometric measurement: 7 cross-selective liquid sensors sensitive to ionic, neutral & chemical compounds responsible for taste.
## Electronic Tongue Analyzers

<table>
<thead>
<tr>
<th>Taste Analyzer Instrument</th>
<th>Sampling</th>
<th>Detection technology</th>
<th>Taste Analysis Applications</th>
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<tbody>
<tr>
<td><strong>ASTREE Electronic Tongue</strong></td>
<td>Autosampler with 16 or 48 positions</td>
<td>ChemFET sensors</td>
<td>R&amp;D, formulation / product development, benchmarking</td>
</tr>
<tr>
<td><strong>ASTREE E-Tongue brochure &gt;&gt;</strong></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td><strong>Solid Form Dynamic Analyzer (SFDA)</strong></td>
<td>Manual</td>
<td>ChemFET sensors</td>
<td>R&amp;D, formulation / product development, galenic, benchmarking</td>
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### DATA PROCESSING SYSTEMS

Chemometric techniques, a type of multivariate statistics used in the analytical field, provide data processing, which consist of recognition, classification, and identification and multivariate calibration.

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<th>Use</th>
<th>Application</th>
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<td>Qualification, exploration and discrimination</td>
<td>Initial formulation studies</td>
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<td>Discrimination factorial analysis (DFA)</td>
<td>Discrimination and identification</td>
<td>Recognition of unknown sample</td>
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<tr>
<td>Soft independent modeling of class analogy (SIMCA)</td>
<td>Good/bad modeling</td>
<td>Quality control against reference good product</td>
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<tr>
<td>Partial least square (PLS)</td>
<td>Quantification</td>
<td>Quantification of bitterness against sensory panel</td>
</tr>
</tbody>
</table>
Pattern Recognition

Unsupervised
- to clustering of variables or samples into groups that are mutually related
  - PCA, FA, Cluster analysis, MDS

Supervisory
- Variable or samples are classified into known groups
  - MRA, CA, PLS, LDA, KNN
Asecsulfame-K
(1, 2, 3, 4, 5, 6, 7, 8%)
Sucralose
(1%, 2%, 3%, 4%, 5%, 6%, 7%, 8%)
Aspartame
(1%, 2%, 3%, 4%, 5%, 6%, 7%, 8%)
Sugar
(2%, 4%, 8%, 16%, 32%, 64%)
Electronic Eye
Electronic eye - IRIS

Camera imaging
- 16 million colors imaging
- Integrated zoom
- Automated monitoring by software

Light cabin
- Reproducible lighting conditions, D65 compliant, 6700°K color temperature
- Top and bottom lighting (backlighting to avoid shadow effects)
- Large measurement surface (420 x 560mm)

E-Eye Alphasoft software
- Data acquisition
- Automated color calibration
- Data processing (color and shape analysis)
- Multivariate Statistics (Principal Components Analysis, Statistical Quality Control, etc)
Perform an overall visual analysis

Visual Analyzer for advanced color analysis of complex surfaces of food, beverage and packaging products. This instrument performs an overall visual analysis of the product as seen by the consumer, whatever the size, shape, texture, packaging. It also allows a focused visual analysis of selected portions.

The IRIS Visual Analyzer consists of:

> a closable measurement chamber of large dimensions (420 x 560 mm) that guarantees controlled light conditions, with no influence of external light for visual analysis

> a CCD camera (16 Million colors) for high resolution imaging

> Visual palatability of food products & packaging
> Visual monitoring of product stability and shelf life
> Visual benchmarking of competitive products
> Exogenous colors / materials identification
> Surface analysis
> Analysis of consumer vis

<table>
<thead>
<tr>
<th></th>
<th>% of red color</th>
<th>% of pink color</th>
<th>% of white color</th>
</tr>
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<tbody>
<tr>
<td>New recipe</td>
<td>22.8</td>
<td>76.8</td>
<td>0.4</td>
</tr>
<tr>
<td>Current recipe</td>
<td>19.6</td>
<td>79.2</td>
<td>0.4</td>
</tr>
<tr>
<td>Reduced salt recipe</td>
<td>60.3</td>
<td>37.4</td>
<td>0.4</td>
</tr>
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</table>
Potential application areas

Raw material

- Quality of raw materials
- True to type
- Taints pickup
- Storage life
- Deterioration in transport

Ensure processes are operating correctly

End product

- True to type
- Taints pickup
- Packaging odors

Storage

- Deterioration in aroma
- Taste quality
Limitations

- Qualitative, identification of compounds – not possible
- Lack of getting quantitative data for aroma differences.
- Type of sensors, Operating conditions (Sampling protocol, Air flow, Temperature, Humidity)
- Appropriate sensor type - volatile compounds
- **Loss of sensitivity** - in the presence of water vapor or high concentrations of a single component like alcohol;
- **Sensor drift** and the inability to provide absolute calibration;
- Relatively **shorter life** of some sensors
- Method development work - for each specific application and
- High sensitivity - human nose -correlation problem
- **Lack of sensitivity** to odors of interest
- Interference from non-odorous molecules
- **Non –linearity** of sensor response
Advantages

- No reagents
- No pre-treatment of samples
- Sensitivity
- Selectivity
- Rapid
- Non destructive – On line QC – manufacturing
Conclusion

• Very effective tools for Odor/taste/colour analysis
• Fast result - Online Quality control (Yes/No)
• Potential replacer for human panel – (Partially?)
• Knowledge of Multivariate analysis - projection techniques

• As on today, the sensor technology, it is still far from the sensitivity and selectivity of a mammalian nose.

• A sensory panel is necessary to define the desired product quality which can then be used to train the system.

• It could occasionally replace sensory analysis and even perform better than a sensory panel in routine work, or in cases where non-odorous or irritant gases need to be detected.
Some facts on smell/Taste

- Taste is mostly (~75%) smell
- All smells are small molecules (less than 350 molecular mass)
- Insect antennae attached to electronic circuits are being used as odor sensors
- Dogs can distinguish non-identical twins by smell - but not identical twins!
- Sniffer rats” have been used to detect explosives
- Everyone has a unique smell (except identical twins)
- Nobel prize for medicine in 2004. Richard Axel and Linda B. Buck (Columbia university USA) for their discoveries on “Odorant receptors and the organization of the olfactory system"
Thank you

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