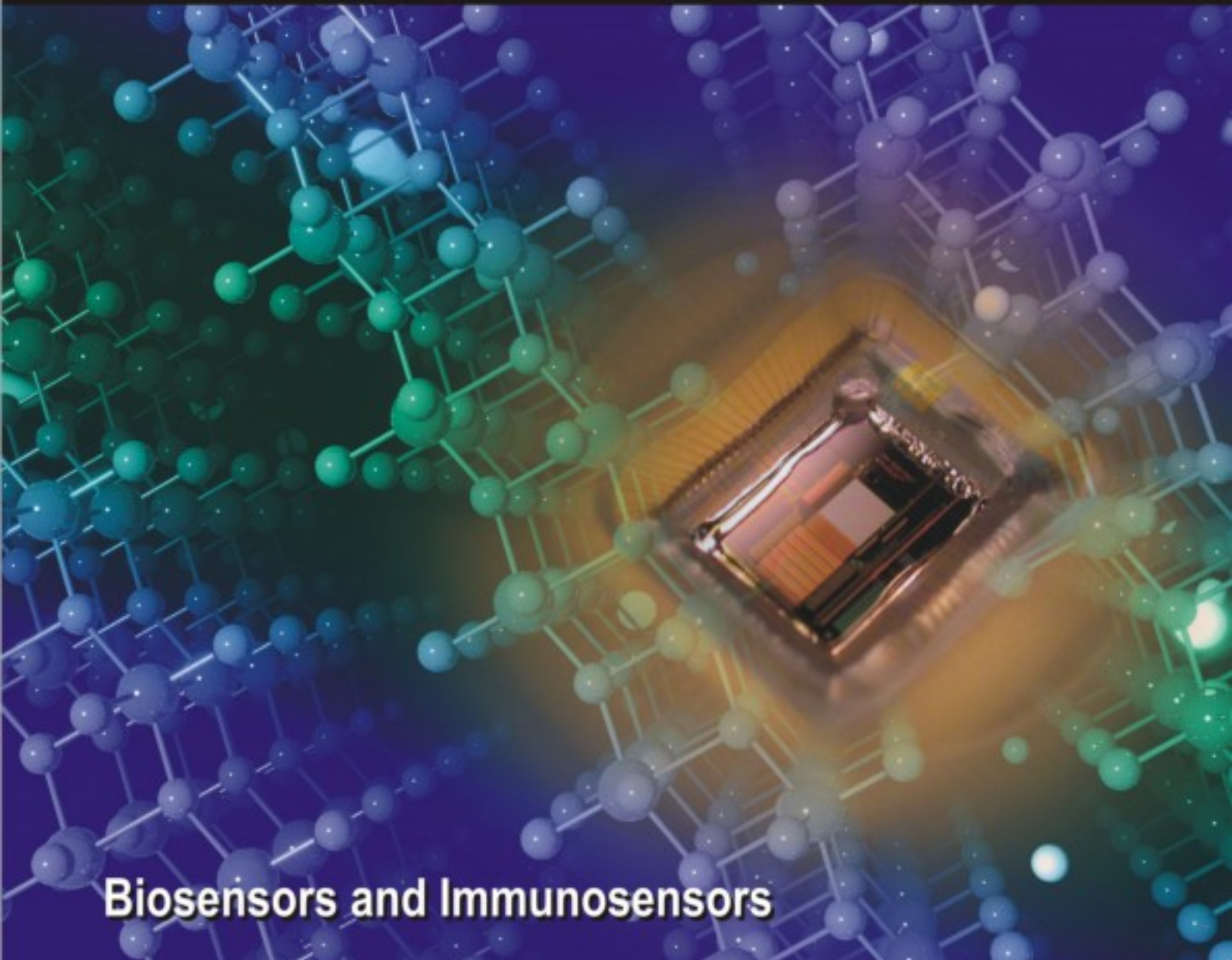


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Odour Profile of Beef Using an Electronic Nose Based on MOS-Sensor

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Abstract: The development of the aroma and flavour of cooked meat is a very complex process in which different components react to produce chemical intermediates or final flavour volatile compounds. The aim of the present research was to study the odour profile of beef produced under different feeding regimes and cooking conditions using an electronic nose based on MOS sensors to monitor the odour characteristics. Odour profiles of different grilled-cooked muscles were evaluated. A clear discrimination between groups corresponding to muscle was observed. Grilled-cooked samples of *Longissimus dorsi* muscle from animals fed to other cooking process. under different diets showed a clear discrimination between groups. Finally, Striploins samples cooked by moist-heat method tend to be different when compared. *Copyright* © 2013 IFSA.

Keywords: Electronic nose, Volatile compounds, Meat, MOS-sensors.

1. Introduction

During the last decade, the demand of meat consumption has increased due to the fact that the preferences of meat consumers are associated to their high nutritional value and sensory characteristics, primarily tenderness, juiciness, and flavour.

The complex flavour, combination of odour and taste, is mainly produced by chemical compounds. Meat flavour from lean or fat tissues can be divided into two categories: at first, those related to common flavour to all species of animals and in second place from specific flavour of each species [1]. Mottram et

al. [2], reported that sugars, amino acids, organic and inorganic salts are responsible for the sweet, sour, salty, and bitter flavour, typical of meat.

The development of the aroma and flavour of cooked meat is a very complex process in which different components react to produce chemical intermediates or final volatile flavour [3]. Several authors have stated [2, 3] the role of fatty acids in meat flavour formation, since thermal lipid degradation is a major contributor to aroma volatiles. Hundreds of volatile compounds have been identified in cooked meat, including aliphatic hydrocarbons, aldehydes, ketones, alcohols, carboxylic acids and esters [4].

Sensory and instrumental studies are the most common methods for measuring flavour. In sensory analysis, the taste and aroma aspects of food products are evaluated by panels, specially trained people. Consumer studies, on the other hand, provide unique information about the acceptance levels of a food and it is also widely used for the determination of overall quality. The most important problem affecting sensory analysis includes standardization of measurements, correctness of training, stability, accuracy and reliability.

Sensory analysis is one of the most important and straightforward research methods in food analysis and provides information about the overall quality of food products. Even though, the relatively low sensitivity and discrimination capabilities of the human nose, coupled with olfactory fatigue, has led to the need for electronic instruments with sensors capable of performing repeated discriminations with high precision to eliminate human fatigue [5]. It should be noticed that odour derived from organic sources may be included hundreds of different compounds all of which contribute to the unique qualities and characteristics of the typical aroma.

Different artificial-nose devices have been developed to discriminate complex vapor mixtures containing many different types of volatile organic compounds. These devices comprise several sensor types including metal-oxide, semi conductive polymers, conductive electroactive polymers, optical, surface acoustic waves, and electrochemical gas sensors. Metal-oxide sensors exhibit very high sensitivity, sub-ppm levels for some gases. The sensing reaction is based on an oxygen exchange between the volatile gas molecules and the metal coating material: electrons are attracted to the loaded oxygen and result in decreases in sensor conductivity [5].

An electronic nose system typically consists of a multisensors array, an information-processing unit such as an artificial neural network, software with digital pattern-recognition algorithms, and reference-library databases.

The sensor array in an electronic nose performs very similar functions to the olfactory nerves in the human olfactory system. The sensors present in the array are chosen to respond to a wide range of chemical classes and discriminate diverse mixtures of possible analytcs. The output from individual sensors is integrated to produce a distinct response pattern that represents the odour profile of the sample [5].

The effectiveness of the electronic nose in the quality control of red meat has been stated by several authors. According to Barbri et al. [6], the electronic nose system can be used as an alternative tool, for shelf-life determination (i.e. quality assessment) and spoilage classification (safety assessment) in red meats.

The aim of the study is to comprise different assays in order to address the ability of a MOS-based electronic nose to monitor beef odour characteristics.

2. Material and Methods

2.1. Samples

Different types of meat were used according to the diet conditions of the animal. The study was divided in three parts, under different conditions.

2.1.1. Experiment 1

The study was carried out at the Agricultural Experiment Station of INTA Anguil, La Pampa, Argentina. Angus steers of similar age and weight were fed with different treatment: 1) Finished on pure stands of cereal rye (Rye); 2) Triticale (Trit); 3) Tricepiro; 4) Oats.

After slaughter, carcasses were individually graded and chilled at 2 °C. Forty eight hours after slaughter, Longissimus dorsi, Gluteus medius, Psoas major and Semitendinosus muscles were removed from the left side of each carcass, vacuum packaged and stored at (-20 ± 1) °C.

2.1.2. Experiment 2

The study was carried out at the Agricultural Experiment Station of INTA Villegas, Buenos Aires, Argentina. Steers from the Angus breed received a grain-based diet. Thereafter, they changed to the following diets. Grain diet: 39 % corn silage (37 % grain), 59 % whole corn and 2 % mineral premix with monensin. Pasture diet: Triticale, Triticosecale Wittmack in vegetative growth stage with a daily forage allowance equivalent to 2.5 % of live weight [8]. Forty eight hours after slaughter, Triceps brachii muscle was removed from the left side of each carcass, vacuum packaged and stored at (-20 ± 1) °C.

2.1.3. Experiment 3

Fresh beef striploins were bought at a commercial slaughterhouse in Buenos Aires, Argentina. Samples were vacuum packed following commercial practice, transported to the Lab and stored at (-20 ± 1) °C until analysis.

Three different types of samples were considered. Samples from Angus breed steers exclusively fed through pasture grazing since their weaning, Angus breed steers from feed-lot, and selected crossbred steers with less than 50 % of breeds of the zebu type fed with forage and supplemented with finely chopped maize grain and sorghum.

2.2. Meat Sample Preparation

Samples were kept at (-20 ± 1) °C before analysis. After thawing at (4 ± 1) °C for 24 h, steaks

were deboned and trimmed of subcutaneous fat and epimysium.

For electronic nose measurement, samples were minced and an aliquot of (2.5 ± 0.01) g of each sample was placed in a 10 mL glass vial equipped with a screw cap and silicon septum. The following cooking methods were considered in the experiments.

2.2.1. Grill

Samples were grilled on a pre-heated Philips electric grill until internal temperature reached $71\text{ }^{\circ}\text{C}$ (AMSA, 1995). Cooked steaks were cooled to less than $10\text{ }^{\circ}\text{C}$ overnight. Internal temperature was monitored with a T-type thermocouple inserted in the geometric centre of each sample.

2.2.2. Dry Heat

Samples were cooked in a pre-heating oven at $165 \pm 0.5\text{ }^{\circ}\text{C}$. The temperature was kept constant until the steak reached at $(71 \pm 0.5)\text{ }^{\circ}\text{C}$, controlled with a T-type thermocouple inserted in the geometric centre of the sample. Then, the sample was removed from the oven and maintaining at room temperature during 30 min. After that, it was stored at $(4 \pm 0.5)\text{ }^{\circ}\text{C}$ up to its measurement, protected from desiccation.

2.2.3. Moist Heat

Sample was placed in a plastic bag, sealed without air inside and then put in a water bath at $(70 \pm 0.5)\text{ }^{\circ}\text{C}$ for 1 h. Then, it was cooled in water at room temperature during 30 min and maintaining at $(4 \pm 0.5)\text{ }^{\circ}\text{C}$ until analysis, protected from desiccation.

2.3. Electronic Nose Protocol

An electronic nose comprising 18 semi-conductor oxide metallic sensors pure and doped semiconductor (MOS), coupled with a mass spectrometer system (NE-MS, Alpha Prometheus, Alpha MOS) was used.

The used device is equipped with two types of sensors: P and T sensors and LY ones. P and T are metal oxide sensors based on tin dioxide SnO_2 (n-type semiconductor), the difference between them resides in the geometry of the sensors. The LY sensors are metal oxide ones based on chromium titanium oxide (p-type semiconductor) and on tungstene oxide (n-type semiconductor). In the presence of a reducing gas, there is absorption with an electronic exchange of gas towards the sensors: the conductance of the n-type increase while for the p-type the resistance will increase [9].

Doping with different elements increases SnO_2 selectivity for different gases. The adopted

configuration results are very flexible for general purposes and convenient for a wide range of applications. Sensors are relatively non-specific and can combine the response of all the sensors in a unique signal. In Fig. 1, each curve represents a different sensor. The curves represent the sensor conductivity (Y-axis) over time (X-axis) when the volatiles from the meat reach the measurement chamber, respect to the value measured when the carrier gas reaches the sensor. As it was shown in Fig. 1, the response of the sensors gradually changed and, finally they reached a stable equilibrium.

An electronic nose system must satisfy reproducibility, long term stability, identification capability and model robustness. In order to monitor these requirements, standardized chemicals aqueous solutions were analyzed. The solutions used were propanol (Aldrich) 0.1 %v/v, ketone (Aldrich) 0.1 %v/v and isopropanol (Aldrich) 0.05 %v/v; all solutions were prepared with HPLC degree water. Measurements were performed regularly at intervals of 15 days.

The analysis was defined as follows: during the acquisition process, samples were kept at $90\text{ }^{\circ}\text{C}$ for 40 min while shaken at 500 rpm to reach the equilibrium in the headspace. The device was continuously purged with dry air (synthetic air N35, Air Liquid) set at 150 mL/min. The acquisition time was set at 120 s and the delay time (time elapsed between subsequent analyses) was 18 min. These experimental conditions ensured that each step during data acquisition was enough to establish a correct baseline to collect volatile compounds and to allow the recovery up of sensors between sample analyses. The maximal amplitude for sensor response curve was considered for analysis.

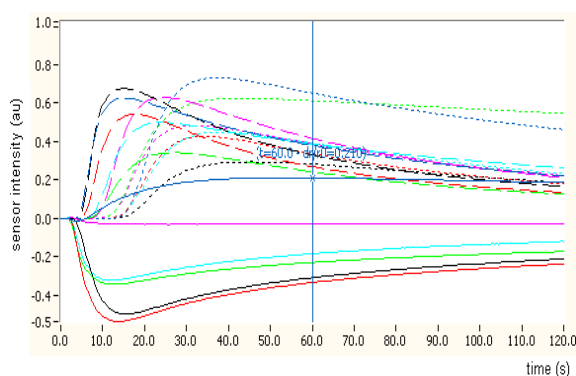


Fig. 1. Signals of the 18 semi-conductor metallic oxide sensors with pure and doped semiconductor.

2.4. Statistical Analysis

Statistical analysis was applied to the maximal intensity response of each sensor. Principal Component Analysis (PCA) is an extraction method used to reduce dimensions and to analyze the inherent structure of the data. Linear Discriminant

Analysis (LDA) estimates a linear combination of the original variable to build a discriminant function and allows visualizing the distribution of points in the same group and the distance between them.

Leave-one-out cross-validation technique is often used to evaluate the classification performance. During cross-validation, all sample data of each class are used for training except one, which is left for testing. That is, for a given dataset with “n” observations in each class, one observation is randomly removed and the rest (n-1) of the observations is used for training. This process of data separation and subsequent validation of the created model is continued “n” time, and the average classification accuracy for all these times is computed. This procedure gives an almost unbiased estimate of the expected generalization error [10]. Data processing methods were performed by SPSS software (version 12.0, Illinois, USA).

3. Results and Discussion

Electronic nose data was analyzed applying PCA and LDA methods. LDA was chosen due that it considers the relation of data points for the specified classes, taken into account the distribution within classes and the distances between them. Therefore, it allows us to collect information from all sensors in order to improve the resolution of classes [10].

3.1. Experiment 1

Odour profile of different muscles (Longissimus dorsi; Gluteus medius; Psoas major and Semitendinosus) were compared from steers finished on pure stands of cereal rye (Rye). All samples were submitted to grill cooking process.

Fig. 2 shows the results obtained for grilled samples according to different muscles. Three discriminant functions (LDA) were found, accounting for 84.3 %, 11.4 % and 4.3 % of the total variation respectively, with a success rate of correct classification of each sample in their respective group (i.e.: muscle) of 96.9 % and 89.1 % of the original cases and after cross validation. A clear discrimination between groups corresponding to muscle can be observed.

Fig. 3 shows the results as obtained with electronic nose data corresponding to grilled samples of Longissimus dorsi muscle from animals fed under different treatment (Tricepiro; Triticale; Rye and Oats). Two discriminant functions (LDA) were found, accounting 98.6 % of the total variation, with a success rate of correct classification of each sample in their respective group (i.e.: feeding) of 98.2 % and 91.1 % of the original cases and after cross validation. A clear discrimination between groups corresponding to different feeding systems was observed.

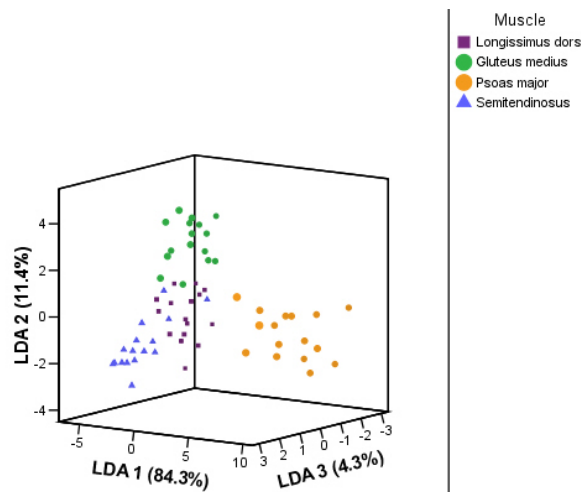


Fig. 2. Discriminant function analysis of electronic data for different types of muscle Longissimus dorsi (■); Gluteus medius (●); Psoas major (●) and Semitendinosus (▲) from steers finished on pure stands of cereal rye (Rye).

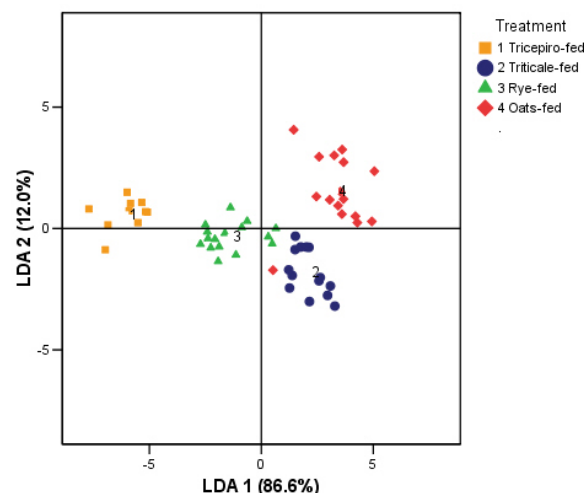


Fig. 3. Discriminant function analysis of electronic data for grilled samples of Longissimus dorsi muscle of animals fed by different treatments. Tricepiro (■); Triticale (●); Rye (▲) and Oats (◆).

3.2. Experiment 2

In this experiment different cooking methods were compared. Meat samples belonging to animals fed under the treatment of pasture (P) and grain diets (G) were analyzed. Two discriminant functions (LDA) were found, accounting 64.4 % of the total variation (Fig. 4). No clear discrimination between groups was achieved; even though, samples were first differentiated according to the base diet of the animals pasture (P) and grain diets (G). Odour profiles of samples cooked by moist-heat method tend to be different if it was compared with other cooking processes.

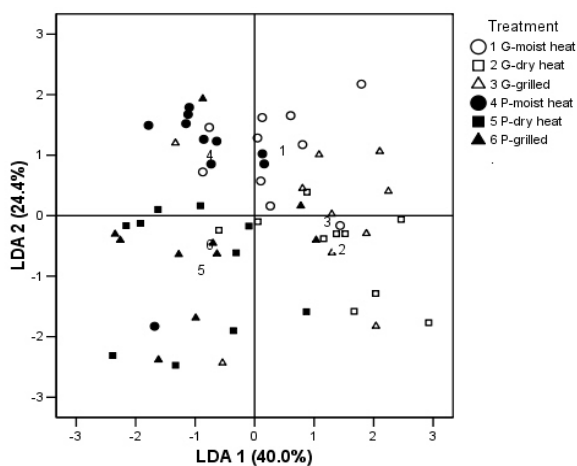


Fig. 4. Discriminant function analysis of electronic data for samples of two types of diet, pasture (P) and grain (G), compared to different treatment (cooking process: moist heat; dry heat and grilled). (Grain (G) moist heat (○); Grain (G) dry heat (□); Grain (G) grilled (Δ); Pasture (P) moist heat (●); Pasture (P) dry heat (■); Pasture (P) grilled (▲)).

3.3. Experiment 3

Odour profile of grill-cooked striploins samples were compared, results are shown in Fig. 5. Two discriminant functions (LDA) were found, accounting for 88.6 % and 11.4 % of the total variation respectively, with a success rate of correct classification of each sample in their respective group of 99.0 % and 92.4 % of the original cases and after cross validation. A clear discrimination between groups can be observed.

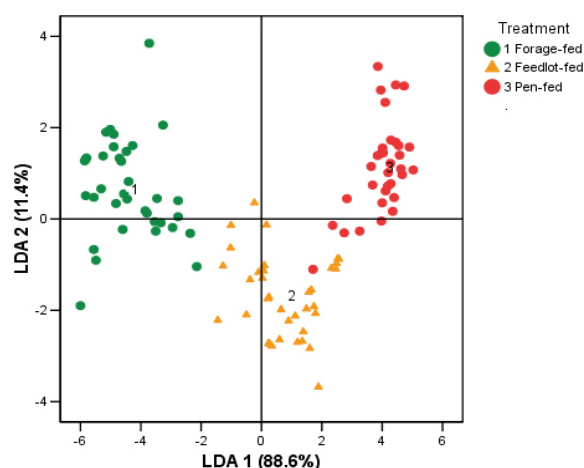


Fig. 5. Discriminant function analysis of electronic data for grill-cooked samples belonging to different types of diet (Forage (●); Feedlot (▲); Pen (●)).

According to previous results, differences in beef odour from animals under different feeding regimes were observed. Even more, it was possible to distinct odour profiles from different muscles. Unexpectedly,

no clear discrimination was achieved when comparing cooking methods.

The volatile compounds in simulated beef flavour and compared flavour compounds in roasted and boiled beef analyzed under the same conditions [11]. The simulated beef flavour was provided by 2-methyl-3-furanthiol with various pyrazines contributing roasted notes, while other aroma compounds including terpenoids, along with the absence of various aldehydes and ketones, resulted in the subtle differences between the simulated beef flavour and cooked beef.

Wisner et al. [12] stated that difference in genetics, feeding and management practices in cattle confer different flavour attributes. These characteristic should be associated to differences in fat deposition, which in turn have been attributed to differences in fatty acid profiles of beef due to differences in diet, breed type and sex. In this context, Elmore et. al. [14] compared the volatile and fatty acid compositions of grilled beef from animals fed either concentrates or silage, belonging to Aberdeen Angus and Holstein-Friesian breed. The stated author mentioned that it is still necessary to understand how the observed differences in both profiles impact on to the characteristic notes of beef odour and flavour.

Descalzo et. al [14] studied how feeding influences overall antioxidant power in meat and its possible relation with meat odour characteristics assessed by electronic nose. Authors found a linear correlation between a set of MOS-sensor and antioxidant activity, expressed by FRAP (total ferric reducing antioxidant power) levels, of fresh meat. Electronic nose methodology successfully discriminated the odour characteristics of samples corresponding to pasture- and grain-based diet.

4. Conclusions

Electronic nose analysis was able to discriminate odour profiles from different muscle and from the same muscle of animals under different feeding treatment. These results can be attributed to differences in the volatile fraction composition impacting on their odour profiles. The observed differences between different feeding treatment, muscle and cooking process must be considered when odour profile in meat is analyzed.

Nowadays, the development of the electronic nose methodology, including a chemical sensory array, provides a powerful tool to analyze odour as a set of odorants present in a given sample.

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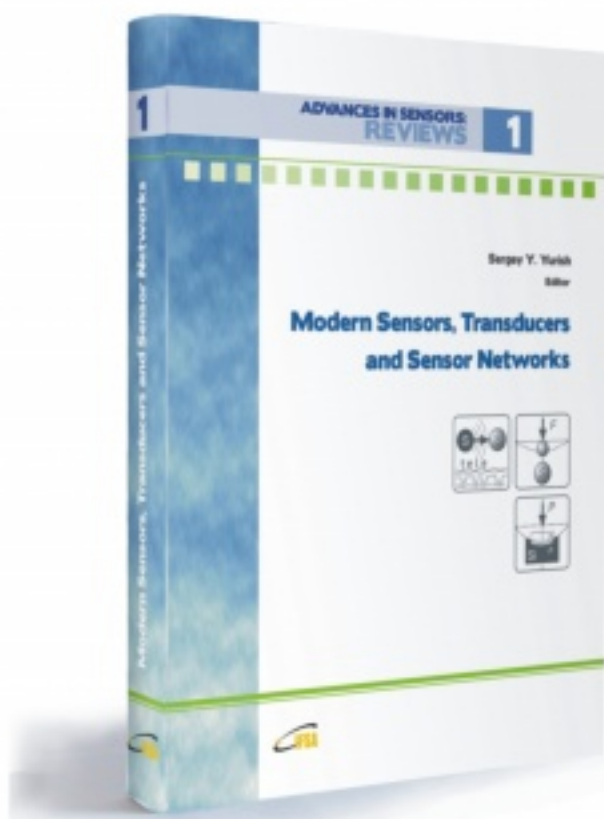
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