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E-nose and E-tongue: an Analytical Tool for Quality Control and Management in the Pet Food Industry

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Abstract: In pet food production, the development of new products must take into account both nutritional and palatability aspects. Pet food palatability is related to the pet food sensory properties, such as aroma, flavour, texture shape, and particle size. The pet food industry may take advantage of a sensorial analysis as a powerful tool for quality control and management. The objective of this idea is to set up an electronic nose (e-nose) and tongue (e-tongue) as rapid quality control and research & development tools for the pet food industry. The final goal is to integrate e-nose and e-tongue with other sensing and imaging devices to 1) Ensure high pet food standards in terms of nutritional properties, palatability and acceptability; 2) Set up an instrumental protocol with good correlation to animal sensory properties in order to replace animal preference test, chemical and texture analysis.

Keywords: Pet food, Palatability, Electronic nose, Electronic tongue.

1. Introduction

The pet food industry offers a wide range of products to satisfy pet's and owner's requirements. Maintaining the health of dogs and cats by feeding wholesome nutritional diets is becoming an important component of responsible pet ownership [1]. In pet food production, the development of new products must take into account both nutritional and palatability aspects. Pet food palatability is related to the pet food sensory properties, such as aroma, flavour, texture shape, and particle size [2]. Pet food formulation is one of the factors affecting its aromatic profile that is strictly associated with palatability and acceptability. Sensorial analysis of pet food may be conducted by using several hedonic and analytical methods (Fig. 1) [3].

Although taste and olfactory perceptions are not completely similar, dogs and cats use both taste and smell in food detection and selection [2, 4]. Besides ingredient composition, pet food palatability may be affected by the use of palatability enhancers and food processing.

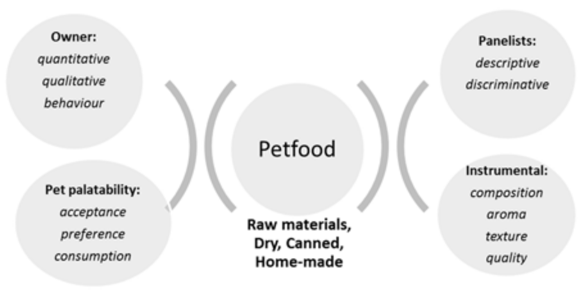

Fig. 1. Pet food sensory analysis research methods.

Microbial growth, oxidation, and the presence of undesirable compounds and contaminants represent risk factors responsible of changes in aroma, flavours, and loss of palatability [5]. At the industry level, adoption of a rapid, low-cost, high-throughput and on-line analytical approach is needed at all stages of pet food production and processing in order to guarantee and standardize the quality of the production.

The objective of this idea is to set up an integrated e-nose and e-tongue technology as a rapid quality control and research & development tool for the pet food industry, focusing on protein source characterization in pet food. To develop the idea, a step by step procedure must be designed: knowledge of e-nose and e-tongue characteristics and applications in the pet food industry, proper selection of an appropriate e-nose and e-tongue system for the specific application, and analysis of critical points for the use of the e-nose/e-tongue in an integrated system for quality control and research & development.

2. Pet Food Industry in the Society

The global market of the pet food industry has been a US$75 billion dollars business in 2016, with North America dominating the market [6]. Of that market, dog food accounts for over half and cat food 35 %. An overview of pet food industry and pet animals in Europe is reported in Table 1 [7].

Table 1. Europe: pet animals and pet food industry in society.

Main topics	Values
Dog population	80 million
Cat population	100 million
Estimated number of European households owning at least one pet animal	75 million
Number of pet food producing companies	650 plants
Employment	Direct employment pet food industry: 80.000 Indirect employment: 700.000
Annual sales of pet food products	Volume: 9 million tons Turnover: € 15 billion
Annual value of pet related products and services	€ 6.5 billion accessories € 8.5 billion services Total: € 15 billion
Annual growth rate of the pet food industry (average value over the past 3 years)	1.8 %

All these data support the need of analytical methods for a rapid analysis of pet food characteristics and properties. As many characteristics that directly determine the effective quality and/or safety of a pet food are often aspects of or described by its odour/aroma/taste, and we believe that electronic nose and tongue may be able to meet the requirements of the pet food industry.

3. The Electronic Nose and Electronic Tongue

E-nose and e-tongue have been increasingly used in the food industry as rapid and reliable tools for quality assessment [8]. The e-nose is an instrument that comprises an array of electronic chemical sensors, with partial specificity and an appropriate pattern recognition system, capable of recognizing simple or complex volatile organic compounds' (VOCs) patterns associated to a product odour [9]. The conventional aroma analysis by gas chromatography–mass spectrometry (GC–MS) is too time-consuming, complex and labour-intensive for routine quality application. Compared to GC-MS, e-nose presents several advantages for manufacturers and processors (portable, ease to use, rapid response and low costs), which make it a powerful tool for screening analysis to address the needs for routine quality testing in the food industry. Therefore, the major differences between e-nose and standard analytical chemistry equipment are that e-nose

1) Produces a qualitative output;
2) Can often be easier to automate;
3) Can be used in real-time analysis;
4) Can be easily integrated in current production processes.

The electronic tongue (e-tongue) has been developed in the last decade to evaluate the taste of liquid media. The common principle of the different e-tongue technologies is the application of an array of non-specific chemical sensors with partial specificity (cross-selectivity), coupled with chemometric processing, for recognizing the qualitative and quantitative composition of multispecies solutions [10]. To analyse results, similar pattern recognition techniques are needed for both the e-nose and e-tongue.

In literature, the applications of the e-nose and e-tongue in pet food analysis are very scarce. This could be attributed to the need to tune either the hardware and/or software to a specific application, or because data are kept confidentially by the product developers. E-nose associated or not with e-tongue has been used in studies for the standardization of a product development process, and in the quality control of the finished product [11-15]. In a study carried out in our department on commercial dry complete and dietetic dog and cat pet food, the e-nose was able to discriminate samples, although not completely, according to the species (dog *vs* cat), to the pet food formula (complete and balanced vs dietetic). Interestingly, e-nose was able to discriminate complete pet food for puppies or adult dogs (Fig. 2) [15].

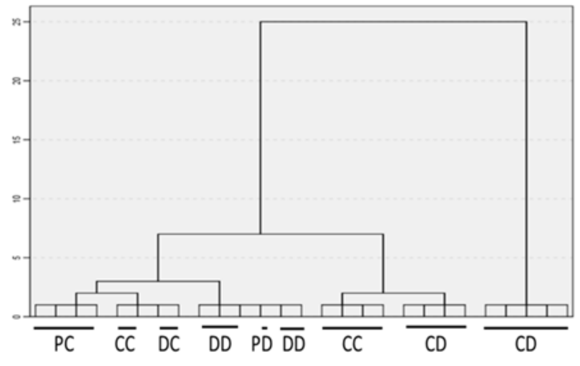

Fig. 2. E-nose for dry pet food analysis (PC: puppy complete; DD: dog complete; CC: cat complete; PD: puppy dietetic; DD: dog dietetic CD: cat dietetic) (adapted from [9]).

In a study on commercial canned dog and cat pet food, similar results have been reported, with no complete discrimination obtained with e-nose analysis. A combination of e-nose and e-tongue determined a better discrimination between samples [13].

Although inconsistent results have been reported, e-nose and e-tongue may represent rapid and sensitive instrumental techniques for pet food evaluation [16]. However, to represent an effective tool for a rapid quality control and research & development in the pet food industry, the analytical platform still needs several improvements, such as the definition of the best sensors' array, the development of data fusion analysis systems, and a better understanding of the industrial needs. The final aim of this idea is to develop an analytical sensory platform able to ensure high pet food standards in terms of nutritional properties, palatability and acceptability. The development of an instrumental protocol with good correlation to animal sensory properties could replace animal preference test, and chemical and texture analysis.

4. The Idea: e-nose and e-tongue for Protein Source Characterization in Pet Food

4.1. Set the e-nose and e-tongue for the Specific Application

The pet food industry traditionally utilizes a wide range of animal protein sources including meat and bone meals, poultry meals, poultry by-products meals, and fish by-products [17]. While traditional options such as beef, lamb, chicken, turkey, salmon and white fish continue to make up the bulk of pet food proteins, exotic sources are increasingly in evidence. Selection of high quality protein ingredients, consumption rates and digestibility are critical points affecting pet health and also the nutritional sustainability of pet food production. Moreover, a successful pet food must taste good. The most nutritious food in the world is useless if it will not be eaten. Ingredients and pet food formulation affect its aromatic profile that is strictly associated with palatability and acceptability.

A proper selection of an appropriate e-nose and e-tongue system for a particular application must involve an evaluation on a case-by-case basis. E-nose and e-tongue selection for a particular application must necessarily include: the assessment of the selectivity and the sensitivity range of individual sensor arrays for a particular target VOCs' profile (i.e., related to pet food components and target organoleptic properties of the products) and chemical substances showing the basic taste qualities, unnecessary (redundancy) sensors with similar sensitivities, as well as sensor accuracy, reproducibility, response speed, recovery rate, robustness, and overall performance. Most of these steps are common points in a validation procedure.

In order to configure an e-nose and e-tongue for protein source characterisation, the different steps of the analytical workflow must be must be considered and set-up (Fig. 3).

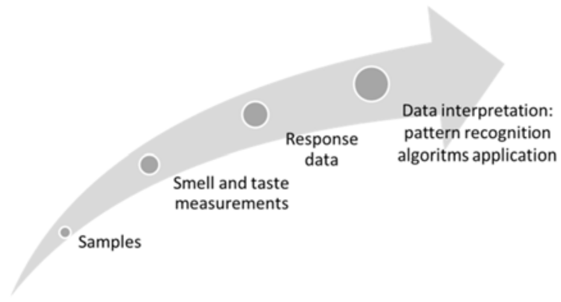

Fig. 3. E-nose and e-tongue analytical workflow.

The first step is the sample collection. Dry pet food containing different protein sources (poultry, mammalian, fish and no animal proteins) will be collected and divided into two subsets. One of the two subsets, training set, will be used to calibrate the model, and the other one, validation set, will be used to verify the robustness of the established model.

The second step is the set-up of a protocol for the analysis of the VOCs'profile and taste. VOCs analysis will be performed on a PEN3 model EN operating with an EDU2 enrichment and desorption unit (EDU) from Airsense Analytics GmbH (Schwerin, Germany) and equipped with a HSS 32 headspace autosampler (Perichrom Sarl, Saulx-Les-Chartreux, France). The sensor array consists of ten metal-oxide-semiconductors (MOS), which characteristics are listed in Table 2.

Samples will be analysed either with thermal desorption pre-treatments or without thermal desorption. All parameters involved in the headspace sampling and analysis will be optimized to obtain the best compromise between sensor responses and measurement time. The ratio G/G0 (where G and G0 are the resistance of a sensor in a detecting gas and in clean air, respectively) will be recorded by the e-nose dedicated software. For experimental purposes, three aliquots of each sample will be singularly analysed, and the mean value of the sensor signals from each aliquot will be calculated and recorded as a single odour profile.

Table 2. MOS Sensor Array of PEN 3.

Sensor name	Description	Reference
W1A - aromatic	Aromatic compound	Toluene, 10 mg/kg
W5B - broadrange	Broad range sensitivity reacts to nitrogen oxides and ozone very sensitive with negative signal	NO_2, 10 mg/kg
W3A - aromatic	Ammonia, used as sensor for aromatic compounds	Benzene, 10 mg/kg
W6B - hydrogen	Mainly hydrogen, selective (breath gases)	H_2, 100 mg/kg
W5A - arom-aliph	Alkanes, aromatic compounds, less polar compounds	Propane, 1 mg/kg
W1B - broad-methane	Sensitive to methane (environment) ca. 10 mg/kg. Broad range, similar to No. 8	CH_4, 100 mg/kg
W1C - sulphur-organic	Reacts on sulphur compounds H_2S 0.1 mg/kg	Sensitive to many terpenes and sulphur organic compounds, which are important for smell, limonene, pyrazine
W2B - broad-alcohol	Detects alcohols, partially aromatic compounds, broad range	CO, 100 mg/kg
W2C - sulphur-chlor	Aromatics compounds, sulphur organic compounds	H_2S, 1 mg/kg
W3B - methane-aliph	Reacts on high concentration > 100 mg/kg, sometimes very selective (methane)	H_4, 10 mg/kg

An Astree e-tongue (Alpha-MOS, Toulouse, France) with a liquid auto sampler unit will be used to measure the taste characteristics of the liquid samples. The sensor array consists of one reference electrode (Ag/AgCl) and 7 liquid cross selective sensors. All parameters involved in sample preparation and analysis will be optimized to obtain the best compromise between sensor responses and measurement time. For experimental purposes, for each sample three parallel measurements will be performed. The raw e-tongue sensor values will be saved in the form of relative resistance changes.

The last step is the feature extraction and data processing. Pattern recognition systems (principal component analysis - PCA; linear discriminant analysis - LDA) will be employed to select variables and build a model to improve the sample discrimination analysis and create models to be used as quality control process tools. Moreover, e-nose and e-tongue data will be dragged into a unique model. The results will be given as percentage of correctly classified samples.

Results from the e-nose and e-tongue will allow to identify the best analytical protocols for protein source in pet food, and to evaluate the capability of e-nose and e-tongue to classify pet food samples into different clusters based on different protein source components. Besides pet food composition, the set up and validation protocol could be used for enhancing the performance of the sensor system for a “total quality evaluation” extending the range of applications of e-noses in the food industry.

4.2. Pet Food Industry: Requirement and Critical Points for e-nose and e-tongue Analysis

When the pet food industry studies and designs new products, it must consider that there are two “consumers”: the pet and the owner. A pet owner buying prepared pet food has two prime concerns: the product must provide a healthy diet and the pet must enjoy eating it. The second critical topic is the nutritional objective: a high quality pet food must be formulated and designed according to the type of pet, its size, age, activity and nutritional status. Pet food safety is another important topic, but safety is now a prerequisite for the pet food industry. Therefore, according to The European Pet Food Industry Federation, quality and safety, nutritional balance and palatability, variety and value for money, pet owner demands, and convenience are the important elements for pets and their owners [18]. Feed material selection, processing, production techniques for canned or dry products, and final product quality and palatability control represent the critical processing points, which need and may take advantage from a real-time monitoring by the use of e-nose and/or e-tongue. Besides quality control, e-nose and e-tongue may be used for research & development purposes in the pet food. Regarding this point, several hot topics may be suggested (Table 3).

4.3. E-nose and e-tongue in an Integrated System for Pet Food Evaluation

Domestic dogs and cats have different nutritional requirements and feeding behaviours, are sensitive to numerous palatability drivers, and differ in the food characteristics that they find desirable. Cats are strongly affected by the aroma of a food, and carefully smell a new food before tasting it. Dogs often prefer foods that are high in fat and include protein from animal sources. For both dogs and cats, the texture, size, and shape of food pieces are important aspects; scientists who study palatability refer to this as *"mouth feel"*. Therefore, e-nose and e-tongue should be integrated in an instrumental platform to develop the full potential of an electronic sense analysis of pet food. Multi-sensor data fusion is an available technology capable of combining information from several sources in order to form a unified picture that can be used as a "finger print" of a product. A practical and general data fusion system model capable of handling data from various applications must be established on the basis of feature extraction. The final goal is to create a high-level fusion, namely decision-making fusion, able to analyse the features from each analytical system first, and then to associate these features to produce a fused result.

Table 3. Pet food industry: e-nose and e-tongue for research & development.

Main topics	Application areas
Composition	Evaluation of protein sources (animal vs vegetal), lipid sources, …
Safety	Analysis of off-odours as markers of contamination (toxins, …), degradation, oxidation, … Measurement of product stability and shelf life
Palatants	Flavour profile of new palatants for pets and identifications of key aromas and taste attributes Development of new pet food palatants
Palatability	Replace animal preference test, chemical and texture analysis Development of highly palatable pet food for: puppies, senior pet, dietetic pet food, etc. Enhance palatability of pet food: dry, semi-dry, wet
Packaging	Effects on aroma and taste, evaluation of the shelf life
Brand	Characterisation of a quality brand

5. Conclusions

The pet food industry may take advantage of an appropriate e-nose and e-tongue system as a powerful tool for both quality control and research & development purposes. Future work is needed on the materials' side (sensors' array), on the data analysis side (better modelling, development of data fusion analysis for the process control system for a continuous quality assurance), and on the industrial side (better understanding of the industrial needs related to quality control and monitoring of food processing). Moreover, to develop the full potential of an electronic sense analysis of pet food, e-nose and e-tongue could be integrated in an instrumental platform including electronic sensors for colour, texture, size, and shape evaluation. Once properly set, this platform could replace animal preference test, chemical and texture analysis to assess pet food palatability.

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