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INSTRUMENTAL TECHNIQUES USED FOR ASSESSMENT OF FOOD QUALITY

INSTRUMENTALNE TECHNIKI OCENY JAKOŚCI ŻYWNOŚCI

Abstract: Two instrumental techniques applied for food quality assessment such as gas chromatography--olfactometry (GC-O) and electronic nose (e-nose) are characterized. The principle of each technique and typical analytical approaches in odour determination are briefly described. Furthermore, the examples of applications, performed also in the author's laboratory, are given.

Keywords: gas chromatography-olfactometry, electronic nose, volatile compounds, aroma, quality

Introduction

Growing interest and care about safe food products fulfilling high quality criteria is becoming one of the major priorities in food technology sector. One of the signs of this care is development and optimization of monitoring and control methods of both, food materials and their processing. Valuable source of information about quality of the particular product is the analysis of volatile compounds based on either classic sensor analysis or application of instrumental methods.

Food products flavour is mostly caused by many volatile compounds which appear in such product. Volatile compounds are mainly responsible for shaping the organoleptic quality of many kinds of food, even though they are found in relatively small quantities in such products [1]. For consumers, an organoleptic quality is equally important and often decisive in the purchase. From chemical point of view, the aroma of most food products is a complicated mixture, sometimes consisting of several hundred compounds. The presence of volatile compounds, their concentration and composition in food products can give valuable information about health quality of food.

In many companies a classical approach in evaluation of organoleptic quality of food products is quite often the only method used on this purpose. Classical methods are mainly based on the sensory analysis, carried out by a group of trained panelists. This analysis is a perfect tool in carrying out marketing tests of consumers but because of many limitations is not sufficient enough for quality control in industrial laboratories. Because of many disadvantages a good complement in the evaluation of organoleptic food properties is instrumental analysis. Detailed and complex qualitative and quantitative analysis of volatile components with the use of appropriate instrumental methods allow for identification of volatile compounds which influence on the flavour composition of food products [2]. The methods employed most often, allowing the creation and recognition of aromagrams are chromatographic techniques, in particular gas chromatography and so called electronic

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nose. Instrumental analysis used for characterization of the food aroma is dominated by two approaches.

First applies gas chromatography-olfactometry (GC-O) to examine single compounds responsible for the aroma of the product. In recent years, intensive studies have been carried out regarding sensory activity of the individual volatile components of various food products and the dependence between the odour and the chemical composition of the volatile fraction of these products, using gas chromatography with olfactometric detection (GC-O) [3, 4].

Second approach is the analysis of the aroma as a whole, without dividing it into individual aromatic compounds. This approach involves analytical methods applying sensors known as 'electronic noses' or 'artificial noses', which because of their simple construction and short time of analysis have become very popular lately.

Gas chromatography-olfactometry (GC-O)

GC-O is a technique based on sensory evaluation of the eluate from the chromatographic column by trained assessor or group of assessors. Quantitative and qualitative odour evaluation is possible thanks to the presence of a specially constructed attachment, called olfactometric port (Fig. 1).

Olfactogram appearance is highly dependent on the analyte isolation procedure and the quantitative method used. Identification of aroma active compounds is possible on the basis of simultaneous use of second detector. Mostly, second detector function performs mass spectrometer (MS) or flame-ionization detector (FID).

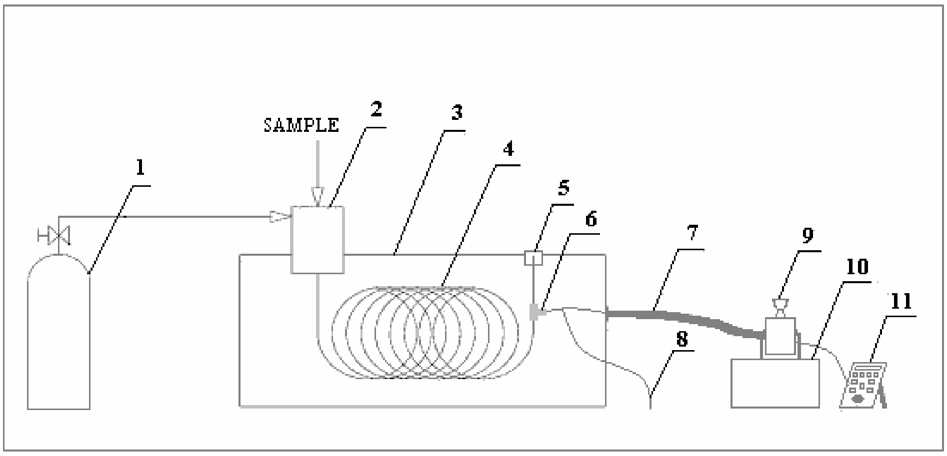

Fig. 1. Scheme of gas chromatograph coupled with olfactometric detector: 1 - carrier gas, 2 - injector, 3 - gas chromatograph, 4 - capillary column, 5 - detector, 6 - splitter of column flow, 7- heated transfer line, 8 - humified air, 9 - olfactometric port, 10 - controller of temperature and gas pressure, 11 - signal generator

Gas chromatography-olfactometry is mostly utilize to intensity evaluation, duration and description of odorous stimulus as well as determination which compounds are responsible for shaping unique aroma or taints of many food products [5]. Development in

GC-O technique results in devising few quantity methods. Each of those methods is based on different principles of the analysis. Three principles can be distinguished: aroma intensity perception, proportion of aroma concentration to detection threshold and the amount of the assessors who detected an odour [6]. Figure 2 presents three categories of GC-O quantitative methods.

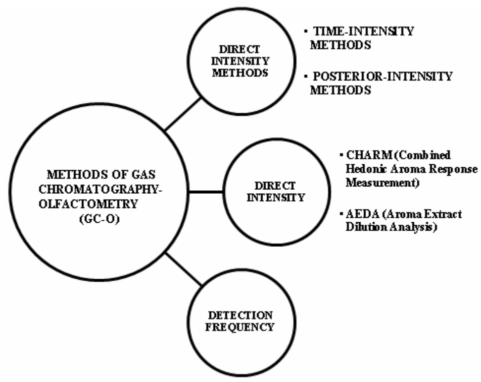

Fig. 2. Quantitative methods in gas chromatography-olfactometry

Although gas chromatography-olfactometry exists over 40 years, the new applications still emerge (Tab. 1). Even samples with complicated matrix can be analyzed by this technique.

Table 1

The examples of GC-O application to food products analysis

Food product	Purpose of the analysis	References
raw spirits	discrimination of different organoleptic quality raw spirits	[7]
honey	identification of distinctive aroma compound in different types of honey	[8]
cognac	quality control of the product on the basic of discrepancies in its volatile fraction	[9]
tarhana	influence of drying methods on active aroma compounds	[10]
olive oil	effect of extraction conditions on virgin oil sensory quality	[11]
champagne	effect of antioxidants on champagne flavour characteristics	[12]
banana	changes in aromatic compounds of banana during ripening	[13]
ham	characterizing of most aroma active compounds	[14]
beer	identification the hop aromatic compounds	[15]
chocolate	identification of key odorant compounds	[16]
Liquor Yanghe Daqu	identification of aroma compounds in young and aged liquors	[17]

Identification of volatile compounds in GC-O coupled with GC-MS analysis can be conducted as follows. As an example can be raw spirits and honeys analysis. In those cases identification of spirits and honey volatiles was made by comparison mass spectra with data in NIST Mass Spectral Database. Additionally, flavour compounds detected by sensory-panel were registered in the form of olfactograms by fingerspan method. Volatile flavour compounds were identified after comparison of their retention times from olfactogram with mass spectra. The predicted aroma of compound was additionally confirmed by literature data and standard substances [18].

Electronic nose (e-nose)

Electronic nose is a device consisting of a set (matrix) of electrochemical sensors, selective for certain volatile compounds. The instrument is equipped with appropriate system of pattern recognition capable of identification of simple and complex odours (Fig. 3) [19].

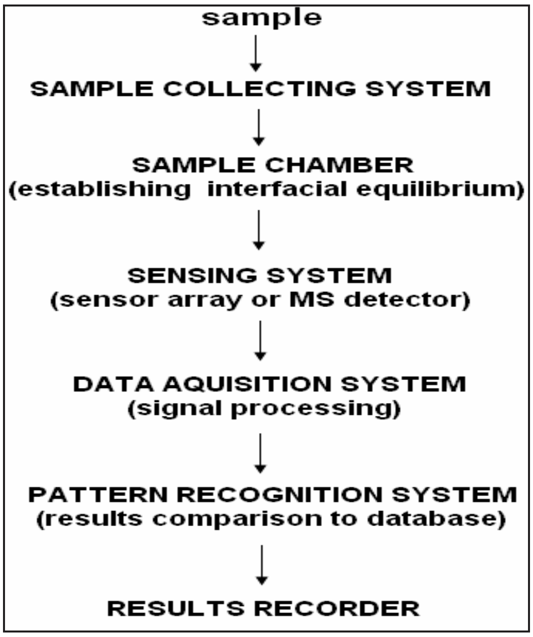

Fig. 3. Electronic nose construction scheme

Since ‘artificial noses’ [19, 20] are cheap and the time of analysis is short, they have become an alternative for relatively expensive methods of food quality assessment [20] such as gas chromatography-olfactometry, infrared spectroscopy or classic sensor analysis. The latter is still the mostly used technique in food analytics and the decisive factor in quality assessment of food products and semi products. However, sensor analysis is characterized by many limitations caused by presence of human factor (eg. olfactory sensitivity of the assessor, their self-well-being, physical and mental state, tiredness or certain anosmia), low repeatability and reproducibility of results, lack of possibilities to identify compounds which influence the aroma and taste or to perform quantitative analysis of them.

Quantitative analysis of odour may be an additional source of valuable information about quality of examined sample. Electronic nose, which creates such possibilities, can be treated as a new type sensor converting chemical signal (odour) into electrical signal. Odour, as well as colour is an analytical signal. However, the quantitative analysis of odour for many years could not have been conducted because of a lack of suitable equipment - sensors.

For the odour of a tested product responsible are not only single aromatic compounds (eg benzaldehyde is responsible for the aroma of bitter almonds), but also their complex mixtures. They give a resultant aromatic effect which is present eg in perfumes. On that account, it is necessary to employ a matrix of gas sensors (the examples of the most frequently utilized are presented in Table 1) [21]. Sensor matrix is built of at least few sensors, each of which is selective towards one different substance and generates appropriate signal, which does not overlap with other signals. The sensors are analogues of protein receptors in nasal epithelium. The electronic system which transfers signal from a sensor works like sensory neurons and Artificial Neural Networks (ANN) constitute its main part [22]. Microprocessor or computer play a role of a brain. There is a resultant signal from all the sensors collected in the computer and an electronic pattern of the odour is created. Similarly to human brain, the pattern is compared with computer database of odours, or more precisely, their chemical structure [19, 22]. Thanks to the pattern recognition system, the analysis can be conducted [23].

To sum up, electronic and human noses function similarly in spite of the differences in the way that signals about odorant are received. Functioning of biological olfactory cells is based on the phenomenon of depolarization of their epithelium (principle of the sodium-potassium pump). Sensors employed in e-nose mainly use the changes in resistance in the active material layer and other physical and chemo-physical phenomena [19, 21].

Electronic nose systems were designed to be used in many branches of industry, where aroma play significant role. Especially, food industry is the biggest and most promising market for these systems [19, 20, 24]. Applications of e-nose in the food analysis include mainly: quality control of raw and manufactured products, monitoring of process, freshness and maturity, shelf-life investigations, authenticity assessment of products and checking packaging for poisonous compounds containment (Fig. 4) [19, 24-26].

Table 2

Characterization of e-nose sensors [27]

Type of sensor	Principle of operation	Sensitivity	Advantages	Disadvantages
MO	Conductivity	5÷500 ppm	Inexpensive, microfabricated, fast response and recovery times	Operates at high temperature, sulphur poisoning, not many coatings available
CP	Conductivity	0.1÷100 ppm	Operates at room temp., microfabricated	Very sensitive to humidity
QCM	Piezoelectricity	1.0 ng mass change	Good reproducibility	MEMS fabricated, complex circuitry, poor signal-to-noise ratio
SAW	Piezoelectricity	1.0 ng mass change	Diverse range of coatings, high sensitivity	Interface electronics, difficult to reproduce
MOSFET	Capacitive charge coupling	ppm	Small, low cost sensors, integratable	Baseline drift
Optical	Fluorescence, Chemiluminescence	Low ppb	High electrical noise immunity	Restricted availability of light sources
MS	Atomic mass spectrum	Low ppb	Potential analytical accuracy	Sample concentration required
MO - Metal Oxide, CP - Conductive Polymer, QCM - Quartz Crystal Microbalance, SAW - Surface Acoustic Wave, MOSFET - Metal-Oxide Semiconductor Field-Effect Transistor, MS - Mass Spectrometry				

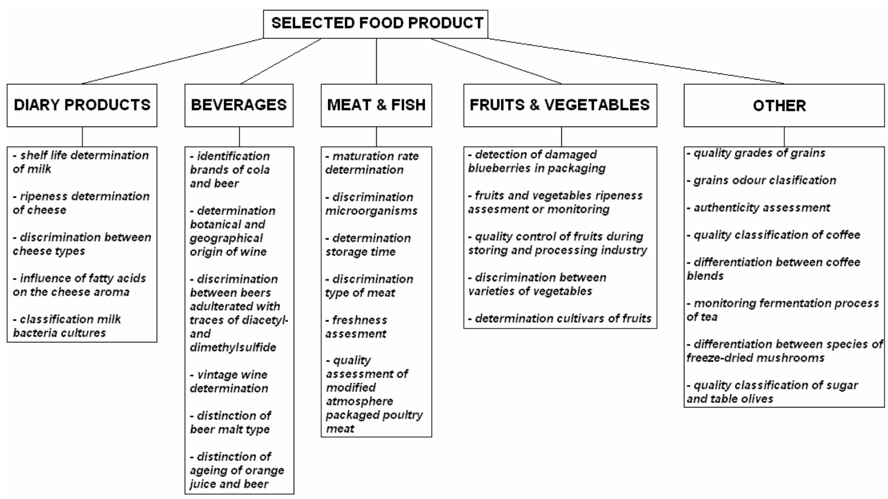

Fig. 4. Practical applications of electronic nose in food industry

Conclusions

Both presented techniques are better than classical organoleptic analyses which are widely applied in the industry. However, more promising seems to be e-nose. It is due to the following features: short time of analysis, high sensitivity and reproducibility, objective identification of odours, possible in situ analysis (portable devise). E-nose system has also a few disadvantages, like ageing material of sensors, possibility of sensors poisoning, in some cases humidity influence to sensor responses.

Nowadays, GC-O and e-nose are additionally applied or even replace methods so far used in the industrial laboratories. Essential feature of those techniques is the fact that such analysis give more detailed information about food products quality (human nose is even more sensitive than conventional detectors). The modern approaches of such type of analysis can point out key aroma compounds shaping flavour of the product and may help in technology improvement. The use of these methods also allow for discrimination raw material used in the production.

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INSTRUMENTALNE TECHNIKI OCENY JAKOŚCI ŻYWNOŚCI

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Abstrakt: Scharakteryzowano dwie techniki instrumentalne stosowane w ocenie jakości produktów spożywczych, tj. chromatografię gazową w połączeniu z detekcją olfaktometryczną oraz nos elektroniczny. Opisano zasady działania obu technik oraz typowe procedury analityczne stosowane w ocenie zapachu. Ponadto podano przykłady praktycznego zastosowania obu technik, włącznie z tymi, które wykorzystano w pracowni autorów.

Słowa kluczowe: chromatografia gazowa-olfaktometria, nos elektroniczny, związki lotne, zapach, jakość