Analytical Methods

Discrimination of mineral waters by electronic tongue, sensory evaluation and chemical analysis

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Mineral, spring and tap water samples of different geographical origins (7 classes) were distinguished by various methods, such as sensory evaluation, electronic tongue measurement, inductively coupled plasma atomic emission spectroscopy and ion chromatography. Samples from the same geographical origin were correctly classified by chemical analysis and electronic tongue (100%), but it was found that only 80% classification rate can be achieved by sensory evaluation. Different water brands (different brand names) from the same geographical origin did not show definite differences, as expected. Forward step-wise algorithm selected three chemical parameters namely, chloride (Cl⁻), sulphate (SO₄²⁻) and magnesium (Mg) content and two electronic tongue sensor signals (ZZ and HA) to discriminate according to the geographical origins.

1. Introduction

In the food industry quality control relies on description of flavour and aroma by sensory panels. Analysis of sensory attributes is generally useful for quality assessment and it is often necessary for quality control (Bleibaum et al., 2002). Sensory evaluation is time consuming, expensive and it is a fact that even trained panelists are influenced by several physiological, economic and personal issues. Moreover, human perception is not constant in time or across people as mentioned by Lawless (1995). In industrial practice necessary sensory tests are generally performed by company employees which means extra load for them. However, a good and robust QC system has to take the sensory properties of the product into account. Therefore, determination of sensory attributes by instrumental measurements is a major trend.

Analytical methods can fit these requirements but they are selective to individual components. It may be necessary to substitute these methods with a generally new concept available in the sensor development; using less selective sensors simultaneously.

This idea is already available to analyse not only for odour but for taste attributes of the samples (Rudnitskaya & Legin, 2008).

Electronic tongues (ET) or taste sensors are one of the most researched tools in this field. Rudnitskaya et al. (2009) analysed eight different types of Belgian and Dutch beers, that were characterised with respect to the sensory properties and were measured using a potentiometric electronic tongue. They observed that the electronic tongue was capable to determine main differences between beer samples, according to sensory data.

Prediction of preference of six different apple juice samples and three modified products (containing added sucrose and citric acid) was performed by Bleibaum et al. (2002) using electronic tongue and electronic nose. Their results demonstrate that the electronic tongue and electronic nose combined can be used to predict sensory characteristics of apple juices and their relationship to the quality of apple juices measured by consumers. The authors conclude that once the electronic sensors are ‘trained’, they can be integrated into the production quality monitoring system, but sensory, physical/chemical, and electronic tongue and nose research must be conducted initially to identify key measures used to define quality for the target consumer.
Another study shows the recognition of apricot storage changes testing three different apricot varieties applying four different controlled atmosphere conditions (Kantor, Hitka, Fekete, & Balla, 2008). Classification of apricot varieties and determination of correlations between electronic tongue, chemical properties and sensory analysis were successful. Electronic tongue was more sensitive to differences than sensory analysis.

Five black teas from plantations of different geographical origins were analysed as a purpose of origin identification with a trained sensory panel and electronic nose and tongue by Kovács et al. (2010). They evaluated the relationship between sensory and instrumental results and found relationship between the growing region of tea samples and the electronic nose and tongue data.

Samples of five brands of milk produced in Poland were tested with the use of a miniaturised potentiometric electronic tongue for the classification of milk’s origins by Ciosek and Wróblewski (2008). The system showed good classification abilities towards milks originating from various dairies.

A real-time system for monitoring water quality was introduced by Taniguchi, Naito, Maeda, Sato, and Ikezaki (1999). Their results showed that taste sensor can distinguish between normal river water and river water contaminated by cyanide and cyano complex and it is also able to distinguish between normal tap water and tap water with an unusual taste due to poly aluminum chloride. In addition to the results obtained by their taste sensors showed a good correlation with the sensory tests results.

The potential application of the electronic tongue has been introduced in the bibliography also in the field of water analyses. Kamiwaki, Terasawa, Masuda, and Uzawa (1992) analysed eight major inorganic ions in commercial mineral water products, city waters and well waters by ion chromatography. The relationship between inorganic components and flavour was examined. Results showed that factor governing the flavour of the drinking waters was the smell and residual chlorine rather than the inorganic components.

Martinez-Manez et al. (2005) successfully employed an electronic tongue including unspecific sensors for the differentiation (identification success rate was higher than 93%) of nine water samples containing six natural mineral waters, two tap waters and an osmotised water.

Sghaier, Barhoumi, Maaref, Siadat, and Jaffrezi-Renaud (2009) used a sensor array consisting of a series of ion-selective electrodes to perform the analysis of six different Tunisian tap water samples from three different geographic regions and seven different brands of bottled mineral water. Principal Component Analysis (PCA) and Discriminant Factorial Analysis (DFA) were applied for the data processing and were employed for qualitative analysis of water. The methods allowed clear representation of differences between the classes of Tunisian water samples. PCA showed that the discrimination was made according to the ionic content in water.

Kundu, Panchariya, and Kundu (2011) introduced a development of water sample classification and authentication based on an electronic tongue application with pulse voltammetry method. The results of the system, implemented in a laboratory environment for six different certified water samples, were used to build classification and regression models. The method can be suitable for water classification/authentication in water quality assessment and monitoring.

Garcia-Brejo et al. (2011) developed an electronic tongue using an array of eighteen thick-film electrodes for drinking water discrimination based on Neural Networks algorithms. Their algorithm was initially trained using seven classes of waters (still, sparkling and tap) to obtain optimum architecture of the networks. After the system was trained it could correctly classify unknown water samples.

The comparison of electronic tongue, concentration of elements and sensory evaluation for waters has not been investigated yet. Our objective was to develop a method with the combination of electronic tongue and concentration of elements. It could provide a simple, cheap and robust method for quality control. Second objective of the paper was to distinguish mineral, spring and tap waters of different geographical origin by different methods. An interesting question was what kind of differences are detectable among the natural bottled water samples from a definite geographical origin, but bottled by different companies with different brand names.

2. Materials and methods

2.1. Samples

Water samples were distributed into eight classes: six commercially available bottled mineral waters, one spring water (non-carbonated, still), and the tap water from Budapest city water supply. The mineral and spring water samples (indicated together as natural water in the text) were originated from the following three geographical regions of Hungary: Albertirs, Visegrad and Miskolc. Throughout the paper water samples are indicated with source region (i.e.: “Albertirs”) and name of well or spring (i.e.: “K-131”). Also, water samples from same regions with different brand names are indicated by “A” or “B”.

Directive 2009/54/EC deals with the marketing and exploitation of natural mineral waters in the European Union. The two main recognised types of bottled water are mineral water and spring water. The Hungarian legislation concerning mineral water and spring water complies with the European Union analogous directives. The tap water samples were taken from the city water supply of Budapest, at Corvinus University Campus.

2.2. Sensory evaluation

Profile analysis was implemented in accordance with relevant international standards (ISO11035:1994; ISO13299:2003). Panelists were trained about the principles of sensory analysis and the procedure of profile analysis. Evaluation was performed at the sensory laboratory of Corvinus University of Budapest. The testing room was designed in accordance with relevant international standards (ISO8589:2007). Panelists worked in independent testing booths, to provide a standard environment, and to avoid disturbance and influence among panelists. Special purpose software (ProfiSens, a joint development of Corvinus and Budapest Technical University) was used during the test sessions to create the experimental design, to collect and to analyse panelists’ data. Presentation order of the samples was randomised and balanced.

All water samples were evaluated by the sensory panel immediately after the samples were poured into glasses. In the first phase of the test sessions panelists received the coded samples. They were asked to record all sensory attributes which they perceived.

During the consensus group session the panelists worked together in order to reach a consensus about the final list of sensory attributes. As the samples were coded, panelists were able to discuss the samples if something was not clear. For each attribute the panelists created an unstructured line scale with descriptor labels at either ends. In case of some attributes a text box was provided for additional comments. One of the samples (“Visegrad K-7/A”) was chosen as a reference sample prior to the test session. The panel was asked to define the intensity value of the reference sample relative to every sensory attribute. This technique is called anchoring in literature, because panelists are provided a fixed
sample during testing. This result in reduced variation among panelists’ data. Once the test sheet was ready, a new set of samples was coded and presented to each panelist. Scale responses of the assessors were converted to numeric values ranging from 0 to 100 percent.

Water samples were tested in two sessions because there is a general recommendation that in descriptive tests the number of samples should not be more than six (ISO 6658:2005). In the first test session there were ten panelists, and in the second session twelve. Two different data sheets were created by two sensory test sessions. Two sets of samples were overlapped for later comparison of results of the two sessions.

The following samples were tested in the first session:


The following samples were tested in the second session:


The attributes (such as metallic taste, bubbles, chlorinate odour, chlorinate taste, salty taste, sweet taste, carbonation, mineralization and fresh taste) were accepted to be the variables for statistical evaluation.

2.3. Measurement by electronic tongue

Water samples were analysed by Alpha ASTREE II (Alpha MOS, Toulouse, France) potentiometric electronic tongue that was designed to analyse, recognise and identify complex dissolved compounds in liquid foods. The instrument is connected to an LS 16 auto sampler unit. The system’s principle is to simulate the human tasting procedure. The electronic tongue has seven ISFET (ion sensitive field effect transistor) potentiometric chemical sensors for food application (ZZ3401, BA3401, BB3401, CA3401, GA3401, HA3401 and JB3401). These chemical sensors are potentiometric sensors with an organic membrane coating that gives each sensor specific sensitivity and selectivity (AlphaM.O.S., 2003). They measure dissolved organic and inorganic compounds in liquids including taste and flavour compounds. The sensors have specific sensitivity for different flavour components. Sensor potential was measured versus a standard Ag/AgCl 3 M KCl reference electrode (Metrohm AG). The sensor signals in equilibrium state were considered as variables for the statistical evaluation (termed as ZZ, BA, BB, CA, GA, and JB). Sensors were cleaned with distilled water between subsequent measurements until stable potential was obtained. Measurements were made at room temperature. The measurement sequence (of the samples) was randomised. The number of samples in classes of geographic origin was as follows: Albertírska K-131 = 7, Albertírska K-125 = 14, Visegrád K7 = 14, Miskolc B-108 = 14. The conditions were following: 100 ml sample volume, 180 s time per analysis, 30 s time per cleaning. The samples were put into the sample holder glasses one hour before the measurement to release their CO2 content, in order to provide reliable analysis.

2.4. Analytical measurements

Concentrations of dissolved elements were measured by inductively coupled plasma atomic emission spectrometer (Thermo Jarrell-Ash ICP-AES 9000, Babington nebulizer) and anions were measured by ion chromatography.

The following parameters were used to determine concentration of Mg, Na and Ca. The power supplied to the plasma was 1.05 kW, the nebulizing gas flow rate (also argon) was 0.55 L.min⁻¹. Experimental conditions were optimised to obtain maximum yield from simultaneous determination of a number of elements. Monoelemental ICP standards from Merck (Darmstadt, Germany) were used for calibration. The calibration was checked after every fourth determination. The pH of the samples was adjusted with nitric acid (10 ml sample + 10 μl of 98% nitric acid).

All anions (F⁻, Cl⁻, NO₃⁻, SO₄²⁻) were analysed with the same ion chromatograph. The separator column used were an IonPac AS11-HC (250 x 4 mm, 9 μm) and an IonPac AG11-HC (50 x 4 mm, 13 μm). Temperature of the columns was 35 °C. Post-column eluent suppression was achieved using a 4 mm Anion Self-Regenerating Suppressor (ASRS-ULTRA II, Dionex (Sunnyvale, CA, USA)), suppressor current was 140 mA. An eluent-generator cartridge (EGC-KOH, Dionex (Sunnyvale, CA, USA)) was controlled by a Reagent Free Controller (RFC-30). Eluent flow rate was 1 mL/min. A Merck Hitachi L-7470 conductivity detector was used. Data were collected and processed by Agilent Chemstation software. The samples were filtered through a SLHV103NL 0.2 μm filter (Milllex-HV Millipore Co., Billerica, MA). Each water sampling was repeated seven times. Concentrations of the measured metals, such as Sodium (Na), Magnesium (Mg), Calcium (Ca) and anions: Fluoride (F⁻), Chloride (Cl⁻), Nitrate (NO₃⁻), Sulphate (SO₄²⁻) were accepted to be the variables for statistical evaluation.

2.5. Statistical analysis

Two unsupervised methods were applied: principal component analysis (PCA) and hierarchical cluster analysis (HCA); as well as one supervised technique: linear discriminant analysis (LDA). These multivariate methods have already been summarized in detail in a bibliography (Berrueta, Alonso-Salces, & Héberger, 2007).

PCA is often the first step of data analysis. Reduction of data dimensionality allows detecting patterns and allows pattern visualisation retaining as much important information present in the original data as possible. Each principal component (PC) is uncorrelated (orthogonal) and is a linear combination of original measured variables. The first principal component (PC1) accounts for the maximum of total variance, the second (PC2) is uncorrelated with the first and accounts for the maximum of residual variance, and so on, until the total variance is accounted for. In practice, it is sufficient to retain only components that account for a large percentage of total variance. The linear coefficients of inverse relation of linear combinations are called component loadings, i.e. correlation coefficients between original variables and principal components. The values that represent samples in space defined by the principal components are the component scores.

Other unsupervised technique for preliminary evaluation is the cluster analysis (HCA), which was used for outlier identification. In HCA, samples are grouped on the basis of distances without taking into account the information about the class membership. HCA calculates the distances between all samples using a defined metric such as Euclidean distance, Manhattan distance, etc. Grouping of samples can be performed by different clustering algorithms, depending on criteria to define the distance between two groups (linkage rule): single (nearest neighbour), complete (farthest neighbour) or average linkages, centroid method, Ward’s method, etc. In this study cluster analysis was done by Ward’s method using Euclidean distances.

Linear discriminant analysis (LDA) is probably the most frequently used supervised pattern recognition method, which is based on the determination of linear discriminant functions. LDA maximises the ratio of between-class variance and minimizes the ratio of within-class variance. LDA utilises a priori knowledge of the group membership for each sample. At the first step most discriminative variables are selected for classifying previously
Results of the first session are shown in Fig. 1. First three principle components and second sensory sessions contained 42 cases with 6 groups to get the final data tables. Both data tables (first sensory session) were randomly selected and removed from each sample class in order to build a uniform data matrix. Cluster analysis of sensory evaluations had to be reduced to 7 data points per sample. Test sessions involved 12 and 10 panelists. Therefore, the number of repetitions; altogether 56 samples were measured, while sensory with bubbles (−0.659) and carbonation (−0.924), and positively with chlorine odour (0.981), chlorine taste (0.967), sweet taste (0.715) and aftertaste (0.848). However, PC2 correlates negatively with metallic taste (−0.756), mineralization (−0.872) and salty taste (−0.691), while PC3 correlates negatively with fresh taste (−0.864).

Three principle components for second sensory analysis explain 70.8% of total variance. PC1 correlates negatively with bubbles (−0.656), sweet taste (−0.712) and carbonation (−0.636), Chlorine odour (0.834) and chlorine taste (0.827) correlates positively with PC1, while PC2 correlates negatively with metallic taste (0.783) and mineralization (0.626). PC3 correlates positively with salty taste (0.822). Results are very similar to that of the first sensory session. Observed differences can be explained by different ability of sensory groups and different compositions of natural water samples.

Results of PCA for both sensory analyses showed that the tap water samples are too different to be analysed together with natural waters. Principal component 1 is mainly determined by tap water attributes. Tap water samples would make the PCA analysis results for other water samples inefficient; therefore we did the PCA analysis separately for the natural water samples. NO3 concentration was a marker compound for tap water since only tap water had significant amount of NO3 content. Chlorine odour was a marker to tap water samples in sensory analysis. The dataset had to be analysed without these markers to avoid trivial results and to achieve a separation of groups. Moreover, selection of marker compounds for natural waters is also an important goal. In further analysis each of the two sensory data tables contained five groups (waters) with 35 cases (samples), eight sensory attributes, six ion concentration data and seven electronic tongue sensor sign.

3. Results and discussion

There are two data sets due to the design of the sensory analysis, one for each session (called: first sensory and second sensory sessions in the text). Standardization was needed because the scales were not equal to each other in case of the different data sets (sensory, electronic tongue and chemical analysis).

The variables were standardized to zero mean and unit standard deviation: variable means were subtracted from each matrix entry and then each entry was divided by the standard deviation of variables.

3.1. Preliminary data treatment and analysis

Since “Visegrád K7/A” samples were used as reference values there were no errors in case of sensory evaluation data. Therefore, standard deviation of “Visegrád K7/B” samples was assigned to “Visegrád K7/A” samples, as well.

Both analytical and electronic tongue tests were performed in repetitions; altogether 56 samples were measured, while sensory test sessions involved 12 and 10 panelists. Therefore, the number of sensory evaluations had to be reduced to 7 data points per sample class in order to build a uniform data matrix. Cluster analysis was done to check for outliers. In case of the first sensory session three panelists can be considered as outlier panelists, while only one panelist in the second session. After eliminating the outliers two panelists were randomly selected and removed from each session to get the final data tables. Both data tables (first sensory session and second sensory session) contained 42 cases with 6 groups and 23 variables; the two data sets overlapped, i.e. 56 samples were measured, not 84.

PCA was applied as data visualisation tool for sensory analysis. Results of the first session are shown in Fig. 1. First three principle components explain 80.1% of total variance; hence loadings of these components were investigated. The loadings show how PCs correlate with original sensory attributes. PC1 correlates negatively with bubbles (−0.659) and carbonation (−0.924), and positively with chlorine odour (0.981), chlorine taste (0.967), sweet taste (0.715) and aftertaste (0.848). However, PC2 correlates negatively with metallic taste (−0.756), mineralization (−0.872) and salty taste (−0.691), while PC3 correlates negatively with fresh taste (−0.864).

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3.2. Sensory evaluation

Stepwise LDA was used to distinguish natural waters according to the sensory evaluation. Model building was done by two different ways. Classes for model building were defined either according to regions of the source (three groups) or according to the commercial brands (five groups).

When samples were defined according to the three regions with the first sensory session the stepwise LDA variable selection resulted in three sensory attributes, namely mineralization, salty taste and metallic taste (ordered in decreasing significance). The three attributes have the highest discrimination power at a 5%
significance level for distinguishing the water samples taken from different regions (Fig. 2).

The first root separated three groups quite well. The group of “Albertirs K-125” sample is closer to the “Albertirs K-131” group than to the “Visegrad K7” group. There was only one misclassification case in the validation set (Table 1). A sample from “Albertirs K-131” group was assigned to “Albertirs K-125” group. However, the source of “Albertirs K-131” spring water is located only a few hundred meters from the “Albertirs K-125” well. All samples of “Visegrad K7” group were correctly classified.

When the analysis was performed to classify the natural water brands, similar to groupings for geographical origin, “mineralization” and “salty taste” variables had the highest discrimination power for differentiation of the natural water samples at 5% significance level. Next to the above mentioned ones, the variable “bubbles” had the third highest discrimination power. LDA loading plots and classification matrix show similar pattern to the groupings according to the geographical origin (figures are not shown).

Models were built also in two different ways for the second sensory analysis. When the samples were classified according to the three regions similar to the first sensory analysis, “mineralization”, “salty taste” and “metallic taste” (ordering in decreasing
significance) had the highest discrimination power for the second sensory analysis. The results of discriminant analysis are shown on Fig. 3.

The group of “Miskolc B-108” samples shows an acceptable separation from the two other groups (“Albertirsa K-125”A and “Visegrádi K7”). The latter overlaps the other based on the ellipses of 80% probability. The classification matrix shows that the LDA model recognizes 90.7% of the “Miskolc B-108” well water, correctly predicts 78.6% of “Miskolc B-108” well water, and misclassifies 21.4% as belonging to “Visegrádi K7” (Table 2).

When the analysis was performed to classify different natural water brands same variables were selected by stepwise LDA as in case of the first sensory analysis. So, “mineralization”, “salty taste” and “bubbles” (ordering in decreasing significance), have the highest discrimination power for differentiation of the natural water samples at 5% error level. The groups of the “Miskolc B-108”A and “Miskolc B-108”B show an acceptable separation from other sample groups, but overlap each other. The sample, “Albertirsa K-125” is separated from the groups of “Visegrádi K7”A well. LDA loading plots and the classification matrix show similar patterns to the groupings of geographical origin (figures are not shown).

3.3. Electronic tongue and analytical analysis

The results are presented in a single dataset that contained 7 different water groups with 7 repeats resulting in 49 cases, with 6 chemical variables and 7 electronic tongue variables. The ranges of the measured chemical variables in case of 7 different water groups were following: Na between 10.54 mg/l and 57.77 mg/l, Mg between 12.47 mg/l and 46.31 mg/l, Ca between 33.75 mg/l and 141.10 mg/l, F between 0.11 mg/l and 1.94 mg/l, Cl between 3.00 mg/l and 52.77 mg/l, and SO₄²⁻ between 1.5 mg/l and 99.7 mg/l.

According to the forward stepwise method three chemical parameters namely, chloride (Cl⁻), sulphate (SO₄²⁻) and magnesium (Mg) and two electronic tongue sensors (ZZ and HA) were selected for the best model of discrimination according to geographical origin. The first two LDA canonical variables for the combined results of electronic tongue and concentrations of elements are shown in Fig. 4.

The three groups of “Albertirsa K-131” and “Albertirsa K-125”, “Visegrádi K7” and “Miskolc B-108” were distributed properly. The results “Albertirsa K-131” and “Albertirsa K-125” samples were overlapping as expected, because these wells are located close to each other. The percentage of correctly classified waters achieved 90.8% that was better than classification with the sensory evaluation. “Misclassifications” were observed only between the samples from the same geographical origin. However, all tested samples from the same geographical origin were correctly classified (100%). The natural variation could have been quite large among samples considering some major elements. A factor analysis study confirmed that the distribution of certain groups of elements mainly reflects the geological patterns (Bertoldi et al., 2011). Similar results were found by Bityukova and Petersell (2010) when analysing five commercial bottled water brands of Estonia: the difference in chemical composition of the bottled waters is linked to host rocks’ geology in the studied area. These experiments did not tell anything about the sensory characteristics of the tested waters. So the integration of sensory and analytical methods provides a fuller understanding of this topic.

4. Conclusions

The tap water samples were more different from the natural water groups than the natural water groups from each other. NO₃⁻ and chlorine odour was major marker of discrimination. To be able to make accurate prediction about mineral water groups the outlying results of the panelists were not taken into account for data processing.

The selection was performed by cluster analysis. We were able to distinguish samples by first sensory test according to their geographical origin with a 90% success rate.

Three chemical parameters namely, chloride (Cl⁻), sulphate (SO₄²⁻) and magnesium (Mg) and signals of two electronic tongue sensors (ZZ and HA) were selected for the best model of discrimination according to the geographical origin. Results of electronic tongue show considerable taste differences between samples from different geographical origins. Chloride, sulphate and magnesium concentration confirmed the discrimination found with the electronic tongue.

Different water brands from the same geographical origin did not show definite differences with the above mentioned experiments. The two brands bottled at the same district are rooted in identical layers. So these waters sold under different brand names but originated from the same geographical region cannot be distinguished. In those cases when the water samples were collected from different regions the discrimination was successful.

Parallel application of analytical, e-sense and sensory technologies supports the modeling of data correlations between these matrices. For a food product like water with rather neutral flavour character and high sensitivity to taste faults, joint application of these technologies is recommended for quality assurance, research, development, and origin identification.

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