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A Hybrid System Based on an Electronic Nose Coupled with an Electronic Tongue for the Characterization of Moroccan Waters

[1,2] Z. Haddi, [1] M. Bougrini, [1] K. Tahri, [3] Y. Braham, [4] M. Souiri, [5] N. El Bari, [3] A. Maaref, [4] A. Othmane, [2] N. Jaffrezic-Renault, [1,*] B. Bouchikhi

[1] Sensor Electronic & Instrumentation Group, Faculty of Sciences, Department of Physics, Moulay Ismaïl University, B.P. 11201, Zitoune, Meknes, Morocco
[2] University of Lyon, Institute of Analytical Sciences, UMR 5280, Claude Bernard Lyon 1 University, 5 rue de la Doua, 69100 Villeurbanne, France
[3] Laboratoire des Interfaces et des Matériaux Avancés, Faculté des Sciences de Monastir 5000, Tunisia
[4] Laboratoire de Biophysique, Faculté de Médecine de Monastir, 5019 Monastir, Tunisia
[5] Biotechnology Agroalimentary and Biomedical Analysis Group, Department of Biology, Faculty of sciences, Moulay Ismaïl University, B.P. 11201, Zitoune, Meknes, Morocco
[*] E-mail: benachir.bouchikhi@gmail.com

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Abstract: A hybrid multisensor system combined with multivariate analysis was applied to the characterization of different kinds of Moroccan waters. The proposed hybrid system based on an electronic nose coupled with an electronic tongue consisted of metal oxide semiconductors and potentiometric sensors respectively. Five Taguchi Gas Sensors were implemented in the electronic nose for the discrimination between mineral, natural, sparkling, river and tap waters. Afterwards, the electronic tongue, based on series of Ion-Selective-Electrodes was applied to the analysis of the same waters. Multisensor responses obtained from the waters were processed by two chemometrics: Principal Component Analysis (PCA) and Linear Discriminant Analysis (LDA). PCA results using electronic nose data depict all of the potable water samples in a separate group from the samples that were originated from river. Furthermore, PCA and LDA analysis on electronic tongue data permitted clear and rapid recognizing of the different waters due to the concentration changes of the chemical parameters from source to another. *Copyright © 2014 IFSA Publishing, S. L.*

Keywords: Electronic nose, Electronic tongue, Loading analysis, Multivariate analysis, Water characterization.

1. Introduction

Quality monitoring of water is one of the worldwide challenge that never will go out of sight. Determination of its quality and control of compliance to standard is often an urgent matter revealed by agencies of consumer protection and food security organizations. To this end, studies are carried out with the help of sensorial profiling panels, or of High-Performance Liquid Chromatography or

Gas Chromatography (GC) and other spectroscopic and instrumental techniques. These different methods determine the chemical composition of a sample and some describe the taste and the flavour of a food product. They can be highly selective and reliable, but they are laborious and require expensive instrumentation, sample preparation, and also they must be operated by skilled panelists. For these reasons there is a strong need for the development of rapid and low-cost methods for product analysis, control and classification. Over the past twenty years, electronic sensing systems, namely electronic nose and tongue are and still endlessly fascinating.

According to the recent International Union of Pure and Applied Chemistry (IUPAC) definition [1], the electronic tongue is defined as a multisensor system, which consists of an array of low-selective liquid sensors and uses advanced mathematical procedures for signal processing based on pattern recognition and/or multivariate analysis, while an array of reduced selectivity of gas sensors is referred to as electronic nose [2]. These devices have known a great success in a variety of applications related to many areas such as food and beverage industry [3, 4], environment [5, 6], pharmacy [7, 8], space [9], etc. Moreover, they showed potential applications within the fields of bioprocess monitoring and medicine [10–12]. Sensor systems are now one of the most promising tools dedicated to foodstuff quality control.

The electronic nose and tongue are capable of both qualitative recognition and quantitative determination of odour and taste. Many categories of sensor array have been involved in the development of electronic noses, e.g., tin oxide sensors, conducting polymer sensors, acoustic wave sensors, MOSFET technology [13]. However, for the electronic tongue systems, various sensors were employed [14]: potentiometric, voltammetric, impedimetric, amperometric, piezoelectric, optical, etc., which were measured in stationary [15] or flow condition [16]. Metal Oxide Semiconductor (MOS) sensors are the most common commercially used chemical sensors in electronic nose systems, while, Ion-Selective Electrodes (ISEs) are the most used sensors in the case of potentiometric electronic tongues. Despite the name, ISEs are often non-selective devices as they are based on ionophores designed for complexing, interacting, holding the analyte in a molecular cavity, etc. This type of interactions is actually non-specific so that interference from other species with similar size and charge commonly appear [17]. Therefore in the current study, and as a first attempt, an electronic nose based on MOS sensors was home-fabricated in order to check its ability in recognizing potable and non-potable waters and then, a potentiometric electronic tongue based on ISEs electrodes was designed to characterize eight waters from different sources (i.e., mineral, natural, sparkling, river and tap waters) using potentiometric measurements combined with Principal Component Analysis (PCA) and Linear Discriminant Analysis (LDA) algorithms. A discussion about the more suitable combination of electrodes is also included.

2. Experimental

2.1. Electronic Nose Set-up

The electronic nose is mainly composed of three parts: sensor array, sampling vessel with system of measurement, and data acquisition system [18]. Fig. 1 depicts the prototype of the electronic nose. The array comprised of five metal oxide Taguchi Gas Sensors: TGS 8xx (with xx=15, 22, 24, 25 and 42) obtained from FIGARO® Engineering, Inc. (Osaka, Japan), a temperature sensor (LM335Z) and a relative humidity sensor (HIH4000-01) from National Semiconductor (Santa Clara, CA, USA). The sensors present in the array are listed in Table 1 together with their target gases. The TGS 8xx sensors require two voltage inputs to operate: a 5 V heater voltage (V_H) to stabilize the sensing circuit inside the sensor, and a 10 V circuit voltage (V_C) for measuring the sensor output. These sensors were placed in a half-bridge circuit according to Figaro Engineering operating data sheets for measuring the sensor conductance variation. More details about the electronic nose system can be found elsewhere [18].

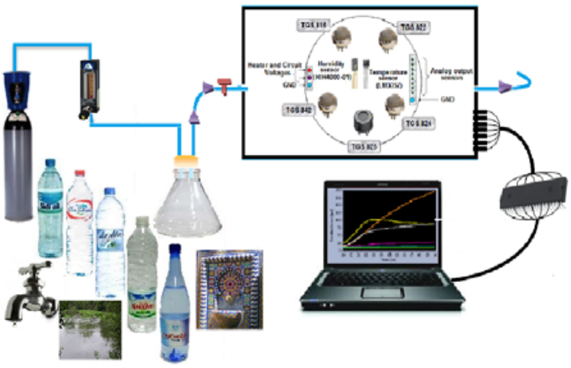

Fig. 1. Schematic representation of the experimental set-up of the electronic nose used in water measurements.

Table 1. Gas sensors used in the electronic nose.

Sensors	Target Gas
TGS 815	CH_4 and combustibles gases
TGS 822	Alcohols, Xylene and Toluene
TGS 824	NH_3
TGS 825	Hydrogen Sulfide
TGS 842	Methane

2.2. Electronic Tongue Set-up

The electronic tongue setup used for the analysis of waters was designed using a sensor array of seven potentiometric chemical sensors which were allsolid-state ion-selective electrodes with poly(vinyl chloride) (PVC) membranes (Calcium, Potassium, Cadmium, Fluoride, Chloride, Nitrate, Sodium) (ELIT electrodes from NICO 2000 Ltd and Metrohm electrodes) and one pH glass electrode [19]. The scheme of the experimental device used in this study is shown in Fig. 2. Sensor potential values were measured versus a conventional Ag/AgCl reference electrode. The sensors and their attributes are shown in Table 2. Ion-Selective-Electrodes allowed to determine then the concentration of cations and anions such as Ca^{2+}, K^{+}, Cd^{2+}, F^{-}, Na^{+}, Cl^{-}, and NO_3^{-} in the studied waters.

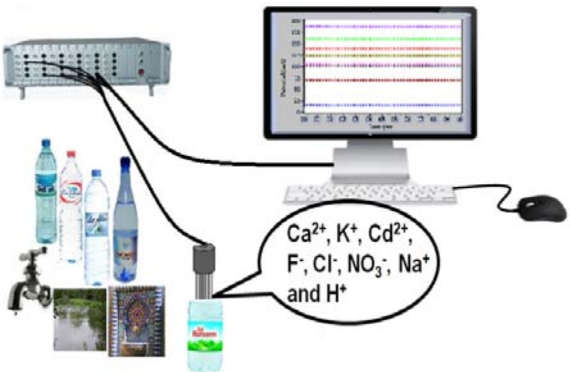

Fig. 2. Scheme of the potentiometric electronic tongue set-up.

Table 2. ISEs employed in the electronic tongue.

Electrode	Membrane	Concentration Range
Calcium (Elit 8041)	PVC	0.02 to 4,000 ppm
Potassium (Elit 8031)	PVC	0.4 to 3,900 ppm
Cadmium (Elit 8241)	Crystal	0.1 to 11,000 ppm
Fluoride (Elit 8221)	Crystal	0.06 to 1,900 ppm
Sodium (Elit 8230)	PVC	0.05 to 2,300 ppm
Chloride (Metrohm)	Crystal	5×10^{-5} to 1 Molar
Nitrate (Elit 8021)	PVC	0.3 to 6,200 ppm
Ag/AgCl	-	-

2.3. Waters under Study

A total of 80 water samples, from different sources (mineral, natural, sparkling, river and tap waters) were analyzed by means of the electronic nose and tongue. Four waters from different brands (Sidi Ali, Sidi Harazem, Aïn Soltane, Aïn Atlas) were mineral waters, one sparkling water (Oulmès), one water from a natural source located between Meknes and Sidi Kacem (Aïn Zouawak), one tap water (RADEM) and finally one river water from Oued Bourouh (wastewater of Meknes). The mineral and sparkling waters were collected from a local supermarket in Meknes.

2.4. Electronic Nose and Electronic Tongue Measuring Set-up

Each water source was introduced into a refrigerator and kept at a constant temperature of 4 °C. Before starting measurement, 20 mL of water sample placed in a 50 mL airtight glass, were taken out the refrigerator and then heated at 25 ± 0.5 °C inside a controlled thermostat-sampling chamber for a headspace generation time of 10 min. For the electronic nose, the sampling system consisted of a dynamic headspace (i.e., after 10 min of heating, the waters were kept inside the controlled thermostat-sampling chamber and then their volatiles were continuously stripped by means of a nitrogen carrier gas at a flow rate of 100 mL/min). In this way, the volatile compounds were directly transferred by the carrier gas into the sensor chamber. The vial presented two small holes in its covers to allow the headspace to be analyzed within the electronic nose equipment. For each set of waters that were analyzed, a new airtight glass vial was used. The signal output was measured at 2 s intervals for 10 min which was sufficient for most of the sensors for reaching a steady state.

For the electronic tongue system, the measurements were generally processed using a personal computer and sensor responses were collected as a data file. Potentiometric measurements were performed using an Ion-Analyser ELIT 9808 (8-Channel Ion-Analyser), required to convert the electrical signal from the ISE into a relevant unit of concentration (ppm or mol/L). The potentiometric measurements carried out by the electronic tongue were directly performed on water samples without any sample preparation or pre-treatment and were thermostatically controlled using a water bath at ~ 25°C. Sensors were rinsed with distilled water between measurements for several minutes to reach steady signal in water. The registered data are ion concentrations of Ca^{2+}, K^{+}, Cd^{2+}, F^{-}, Na^{+}, Cl^{-}, NO_3^{-} and H^{+}.

2.5. Data Pre-processing

Pre-processing of electronic nose data consisted of extracting the most significant features from the sensor response curves. Pattern recognition methods are decisive factors for obtaining a versatile instrument that is able to reliably recognize a wide variety of odours [20]. Previous studies have shown that a combination between the initial conductance, the steady-state conductance, the dynamic slope of the conductance and the area below the conductance curve is often well correlated with the type of odour and in many cases includes the main part of the information [21, 22]. For this reason and in this study, we extracted the steady-state conductance calculated as the average value of conductance during the last minute of measurement and the dynamic slope of the conductance calculated between 2 and 7 minutes of exposure to the water sample. Since there were 5-TGS sensors within the array, each measurement was described by 20 variables. To reduce the variability associated to possible fluctuations in the electronic nose signals, and to minimize other sources of variance also affecting the total signal of the sensors, normalized rather than absolute signals were used to construct pattern recognition models. Therefore, to normalize the variables, the mean-centering pre-processing technique was applied to the dataset of 80 measurements × 20 features.

For the potentiometric measurements, sensor work time was estimated at 100 s which was sufficient for reaching the optimum potential. After this time, the equilibrium was reached and the last potential value was selected as the ultimate sensor feature for electronic tongue data analysis. Every water sample had eight features, coming from the eight ISEs: Ca^{2+}, K^{+}, Cd^{2+}, F^{-}, Na^{+}, Cl^{-}, NO_3^{-} and H^{+}.

Data pre-processing was performed using the MATLAB software 7.0.1 (MathWorks Inc., Natick, Massachusetts, USA).

3. Results and Discussion

3.1. Discrimination between Potable and Non-Potable Water by Electronic Nose

Amongst the goals of this study, discrimination between potable and non-potable was attempted. First, a plot of the dynamic conductance when the sensor TGS 842 is exposed to the different kinds of water samples is shown in Fig. 3. It can be seen that one response sensor (i.e., river water) is evidently different to the other responses (mineral, natural, tap, and sparkling waters). In fact, this was expected since the non-potable water contained several species, such as bacteria and heavy metals, etc., which make the flavour of the wastewater unpleasant in comparison to potable ones. This fact was also demonstrated statically by mathematical methods, such as Principal Components Analysis (PCA).

PCA is a powerful linear unsupervised pattern recognition method that reduces the dimensionality of a multivariate problem and helps to visualize the different categories of taste and odour profiles [23, 24] by highlighting similarities and differences between sample clusters. The results showed that the total variance of the first three principal components (PC1, PC2 and PC3) was 86.98 %. Based on PCA (Fig. 4), two clusters can be obviously distinguished. Similarities in different kinds of waters (mineral, natural, tap, and sparkling waters) were easy to find (i.e., all potable waters formed a unique group). However, non-potable water (Oued Bourouh) was located far from potable waters. While, electronic nose technique cannot detect the changes in taste, the potentiometric electronic tongue has been used to characterize the different studied potable waters.

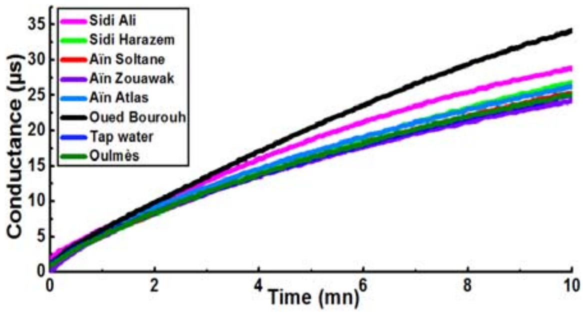

Fig. 3. Electrical conductance of TGS 842 sensor towards exposures to the studied waters.

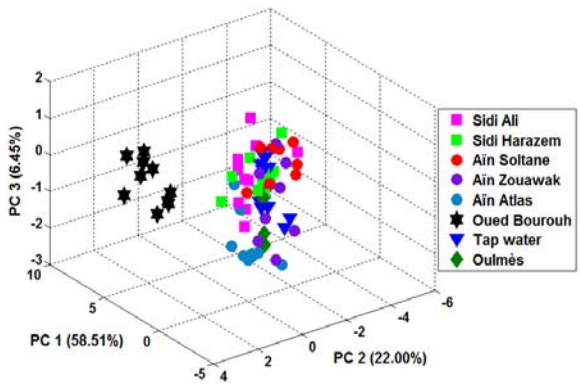

Fig. 4. Scores plot of a PCA performed on eight waters measurements gathered using the five TGS sensors.

3.2. Chemical Parameters of the Studied Waters

In this study, eight chemical parameters (Ca^{2+}, K^{+}, Cd^{2+}, F^{-}, Cl^{-}, NO_3^{-}, Na^{+} and H^{+}) have been deduced from the studied waters by using the eight ISEs. An example of potentiometric electronic tongue data for Aïn Atlas mineral water is shown in Fig. 5.

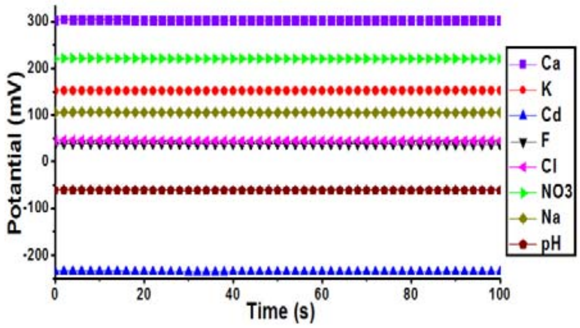

Fig. 5. Potentiometric electronic tongue data acquisition from Aïn Atlas (mineral water).

Horizontal axe is acquisition time and vertical one is potential response when the eight potentiometric sensors were immersed in Aïn Atlas. Each curve represents a different potentiometric sensor response as a function of time (100 s). In order to compare the different studied chemical parameters for each water source, a 3D-representation of the absolute final potential of ISEs is presented in Fig. 6. As it can be observed, the chemical parameters of each water were different from one to another. Moreover, Oued Bourouh river water has a remarkable amount of cadmium. These results form a good starting point for an electronic tongue approach.

3.3. Radar Plot Representations

Graphical displays are particularly suitable for illustrating relationships and trends concisely. The radar plot, used as a method to display data, is much recommended when it is associated with statistical analyses, which can often anticipate the classification of clusters.

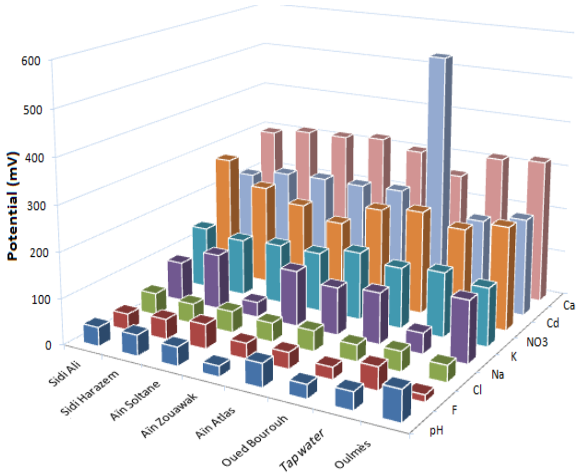

Fig. 6. Absolute final potential for every ISEs for the eight studied waters.

Radar-like plots with unitary radius were used in order to see if there are pattern differences (i.e. fingerprints) between water samples from different sources. Fig. 7 shows a representative case.

To construct these plots, the values of final potential were divided by the value corresponding to ISE cadmium, which showed the maximum potential. This helped visualizing the differences among typical response patterns. Indeed, a clear pattern variation exists between mineral, natural, sparkling, river and tap waters. It must be highlighted that even if Sidi Ali, Sidi Harazem, Aïn Soltane and Aïn Atlas samples belonged to mineral waters, clear differences were found in their signatures. Therefore, any water source can be easily discriminated via the radar plot representation. A fortiori, this was due to the concentration changes of the chemical parameters in the studied water sources.

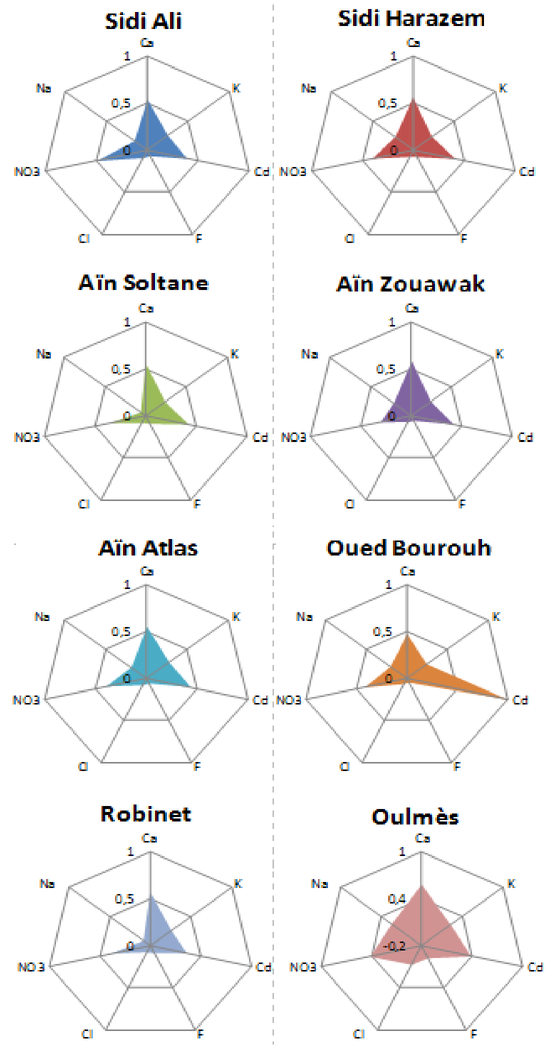

Fig. 7. Radar plot for the different types of waters. The eight plots have radius equal to 1.

3.4. PCA Loading Analysis

Extracted data features were assessed by PCA loading analysis prior to performing PCA on water samples in order to evaluate every sensor contribution in the classification task. It means that one sensor may be switched off for analysis if it has a rather small influence on the identification process. ISEs with loading parameters close to zero for a particular PC have a low contribution to the total response of the array whereas high values indicate discriminating ISEs. Fig. 8 shows the projections of the experimental results on a two-dimensional plane (PC1-PC2). The loading plot shows a net distinction between all sensors, which means that every ISE will contribute effectively in the classification process with different loads. For example, calcium, cadmium, fluoride, chloride, sodium ISEs and pH present a higher influence, while nitrate and potassium ISEs present minor influence. No sensor groups presenting almost identical loading parameters can be found. Therefore, all ISEs will be used in the forthcoming data analysis.

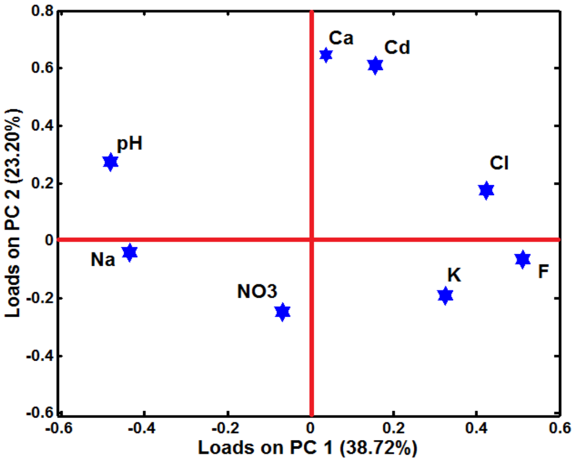

Fig. 8. Loads of each ISE in PCA analysis. A net separation between the different ISEs of the electronic tongue can be seen.

3.5. PCA-electronic Tongue on Moroccan Waters

The discrimination amongst different Moroccan water profiles through potentiometric electronic tongue was elaborated by PCA analysis.

Fig. 9 displays a visual appraisal of the discrimination of each water source. Scores (i.e. the projections of measurements in an orthogonal base of PCs) suggest that waters could be discriminated by using an array of potentiometric sensors. As one can see, the variances explained by the first and the second PCs were 38.72 % and 23.20 % respectively. Thus, the analysis was extended until the third component (14.80 % of the total variance) and it was reported on a three-dimensional space.

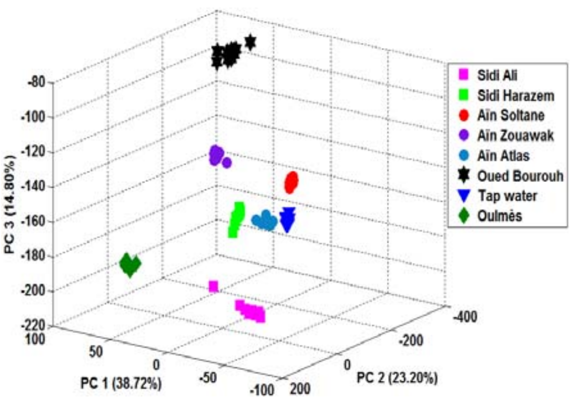

Fig. 9. 3D-PCA plot performed on eight water sources with processed data from the potentiometric electronic tongue.

Hence, the first three PCs captured 76.72% of data variance. The clusters corresponding to the Moroccan water sources were clearly well separated from each other. This indicated that samples could be noticeably discriminated. The mineral waters as well as the natural, tap and sparkling waters were also distinguished from each other. These results highlight the potential application of the potentiometric electronic tongue in recognizing waters from different sources.

3.6. LDA-electronic Tongue on Moroccan Waters

Linear Discriminant Analysis (LDA) is one of the most used classification algorithms. It has been widely used and proven successfully in many applications of electronic nose [25, 26] and electronic tongue [23, 27, 28]. In fact, using this method, data are separated in k a priori defined classes by using linear combinations of the variables in each group to create k−1 new discriminant axis. LDA is widely recognized as an excellent tool to obtain vectors showing the maximal resolution between a set of previously defined categories. LDA tries to find a linear discriminant function along which the classes are better separated. For theoretical background and details of the algorithm, reader should refer to [29].

LDA was conducted on the data set as classification tool, at a 5 % significance level. Initially and in order to check the classification capability of LDA, the model was built using all the available samples as training set. It was found that all the water samples were correctly classified in their origin source. LDA results are shown in Fig. 10.

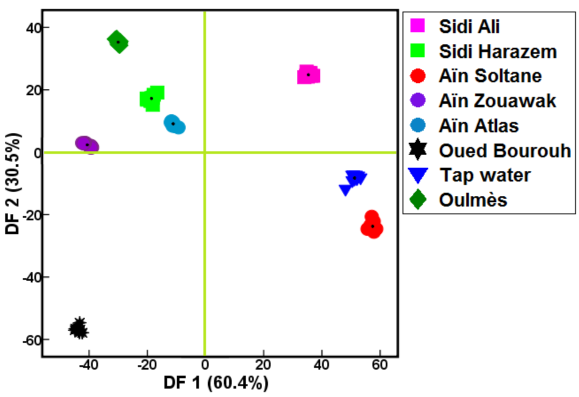

Fig. 10. Projections of water samples in the space defined by the LDA Discriminant Function (DF) 1 and 2. The centroid on each class is indicated as the black point.

Plots of the first two discriminant functions show high separation of the eight groups; all waters are perfectly classified. 90.90 % of the total variance of the data is displayed by the two discriminant functions. Indeed, Function 1 seems to discriminatemostly between Oued Bourouh and Sidi Ali, Aïn Soltane and tap water, while in the vertical direction (Function 2), there was an evident discrimination between Oued Bourouh and Aïn Zouawak, Aïn Atlas, Sidi Harazem and sparkling water (Oulmès). It must be noted that the river wastewater (non-potable) is located in the lower left-side of the DF1-DF2 plane and consequently it is far from the other potable waters.

Using PCA and LDA analysis, it is possible to classify the waters into eight clusters according to their origin. When the potentiometric electronic tongue is performed with LDA, a better classification rate is observed.

3.7. Validation Analysis of Sensor Data Using LDA

Validation analysis was performed using *leave-one out-cross-validation* procedure. This assumes that, with the given *n* measurements (e.g., in this study, n = 80), the model was trained *n* times using *n-1* training vectors. The vector left out during the training phase was then used for the test. The performance of the given model was estimated as the average performance over *n* tests.

LDA results obtained by using the *leave-one-out cross-validation* method are shown in Table 3 as the confusion matrix of the LDA model. Rows indicate true categories and columns predicted ones. It is clear that the ideal situation occurs when all the water samples end up on the diagonal cells of the matrix. That is, every water source was correctly classified according to its origin class leading to a 100 % success rate in the classification of the eight Moroccan water sources. This result is successfully obtained, as presented in Table 3.

Table 3. Confusion matrix for the *cross-validated LDA by leave-one-out* in the identification of the Moroccan waters. (1): Sidi Ali, (2): Sidi Harazem, (3): Aïn Soltane, (4): Aïn Zouawak, (5): Aïn Atlas, (6): Oued Bourouh, (7): Tap water and (8): Oulmès.

Actual	Predicted							
	1	2	3	4	5	6	7	8
1	**10**							
2		**10**						
3			**10**					
4				**10**				
5					**10**			
6						**10**		
7							**10**	
8								**10**

Conclusions

The results obtained in this study show the potential of the electronic nose coupled with an electronic tongue to characterize different water sources in Morocco, namely, Sidi Ali, Sidi Harazem, Aïn Soltane, Aïn Atlas (mineral waters), Aïn Zouawak (natural), Oulmès (sparkling water), tap water and Oued Bourouh (wastewater). The metal oxide TGS sensors together with PCA, allows us to discriminate amongst the potable and non-potable water samples with a total variance of 86.98 %.

The characterization results show that the system of potentiometric electronic tongue, comprising eight ISEs, is expected as a good analytical tool in distinguishing the waters according to their origin sources. Thanks to loading analysis results, all potentiometric sensors data has been used in the water classification. Besides, PCA and LDA results stated that clear representation of the differences between the Moroccan water samples was accomplished, due to the ionic content changes in the different waters. An accuracy of 100 success rate in the recognition of the eight Moroccan water sources was achieved by LDA with *leave-one-out-cross-validation* method.

This study has confirmed again the usefulness of a hybrid system based on an electronics nose coupled with an electronic tongue as rapid analytical tools for the characterization of food and beverage products.

Acknowledgements

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