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Procedia Engineering 47 (2012) 941 – 944

Procedia Engineering

www.elsevier.com/locate/procedia

Proc. Eurosensors XXVI, September 9-12, 2012, Kraków, Poland

A novel humid electronic nose based on voltammetry

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Abstract

We report herein the design, manufacture and use of a "humid electronic nose" prototype. Its operation is based on the use of voltammetric techniques, and it consists of an array of four working electrodes (i.e. Au, Pt, Ir and Rh) that were housed inside a homemade stainless steel cylinder and a fabric mesh made of nylon damped with a NaCl aqueous solution which was used as the supporting humid membrane. The "humid electronic nose" was tested for the discrimination of different samples displaying a different "aroma". The samples chosen involve aqueous solutions of different simple volatile samples (i.e. ammonia, acetone, acetic acid and 6-amino-1-hexanol) and different food samples (i.e. onion, coffee and Roquefort cheese). PCA studies from the response obtained by the "humid electronic nose" allowed discriminate between the different samples studied.

Keywords: Voltammetry; Wet electronic nose; PCA

1. Introduction.

Dodd and Persaud introduced the idea of an electronic nose as a device to mimic the discrimination of the mammalian olfactory system for smells [1]. For this goal the authors used three different metal oxide

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doi:10.1016/j.proeng.2012.09.301

gas sensors and identified several substances by using the measured steady-state signals of these sensors. One of the initial hopes in this area was to instrumentally assess attributed descriptors such as fruity, grassy, earthy, malty, etc. relying on the results of an electronic nose measurement [2]. The resulting receptor pattern, of biological inspiration, determines an impression of the odour in an attempt to mimic the principles of smelling that gives a view of the whole scene of volatiles. The sensor data are analyzed to extract features which can be evaluated as a whole to eliminate redundancy and to arrive at a description of the overall mix of volatiles [3].

Based in these previous concepts it was in our aim to demonstrate that it is possible design "humid electronic nose" using voltammetric techniques in order to discriminate some substances and complex products producing volatile aromas. In particular, and as a part of our interest in the design of electronic tongues and noses, [4,5,6,7] we report herein the design and application of a voltammetric "humid electronic nose" that uses a simple set of metallic electrodes placed on a wet setting. This proposed system detects vapours that emanate from the sample and which are apprehended by a moist environment when the electrodes are included. The use of such a "humid electronic nose" for the detection of volatile compounds is, as far as we know, new and allows the use of classical electrochemical techniques such as voltammetry, commonly employed in electronic tongues, for the analysis of volatile substances.

2. Materials and Methods

2.1. Samples

The response of the "humid electronic nose" was studied in the presence of eight different samples: ammonia (1 mol dm^{-3}, 20ml), acetic acid (1 mol dm^{-3}, 20ml), acetone (1 mol dm^{-3}, 20ml), onion (chopped, 20 grams), 6-amino-1-hexanol (1 mol dm^{-3}, 20ml. to be referred as Am-Hex), coffee (ground, 20 grams) and Roquefort cheese (20 grams). The baseline current was measured with "humid electronic nose" using an aqueous solution of NaCl (0.01 mol dm^{-3}, 20ml) placed in the measuring chamber. For all the samples the membrane containing the electrodes in the "humid electronic nose" was wet with an aqueous solution containing NaCl (0.01 mol dm^{-3}).

2.2. Equipment

A system for the implementation pulse voltammetry was designed in the Centre of Molecular Recognition and Technological Development (IDM) at the Universitat Politècnica de València. The system consists of a software application that runs on a PC and the electronic equipment [6,8].

The equipment generates a sequence of up to 50 pulses with an amplitude in the range of [-2V to +2V]. The width of the pulses can also be configured of [1 ms to 800 ms]. The equipment includes a potentiostat that applies the voltage to the counter electrode of the electrochemical cell and measures the voltage at the reference electrode and the current at the working electrodes (up to 8).

2.3. Measurement procedure

The measuring chamber consisted of a sealed glass cell with a top cover with five inlets. The cell has been designed so no gas leaks take place. One inlet was for the "humid electronic tongue", one for the reference electrode, two for the argon line (inlet and outlet) and one for sample addition. The cell was thermostatized with a commercial recirculating temperature controller. This glass reactor was used as the measuring chamber where a certain sample was included.

The “humid electronic tongue” device (item 1 in Fig 1) consists of an array of four working electrodes (i.e. Au, Pt, Ir and Rh) with purity of 99.9% and 1mm diameter from ALDRICH that were housed inside a homemade stainless steel cylinder used as the body of the electronic tongue and as counter electrode. The fabric mesh (item 2 in Fig 1) was used as the supporting humid membrane where the vapour got trapped. The reference electrode was a Saturated Calomel Electrode (SCE), which was connected to the damped fabric with a curved LDPE tube with cotton in the end (item 3 in Fig 1). The set of electrodes, the nylon fabric and the LDPE tube were held together with a PVC connector which has two pieces (items 5a and 5b in Fig 1). The nylon membrane was trapped between these two pieces and was hold still with a nylon screw (item 4 in Fig 1).

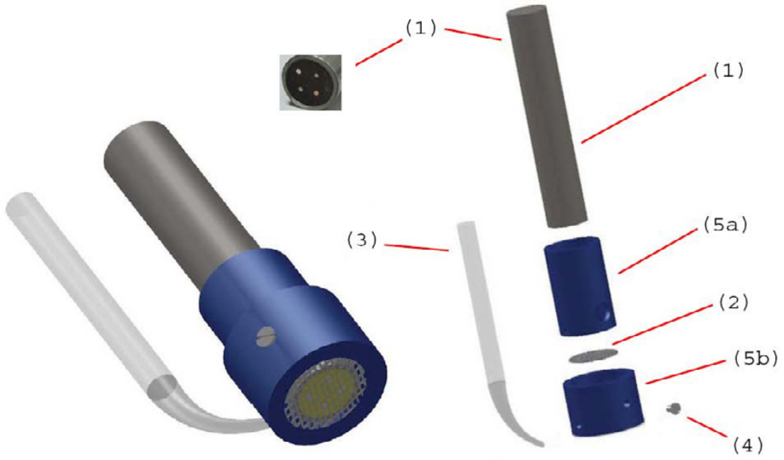

Fig. 1. Wet E-Nose sketch: (1) Electronic Nose; (2) membrane; (3) reference electrode and LPDE adapter; (4) nylon screw; (5) PVC adapters

3. Results

The test settings in the software application were configured to measure several consecutive iterations, this is, the pulse pattern was applied to the 4 working electrodes (in this order: Ir, Rh, Pt and Au) and run the test before the sealed measuring environment had to be opened to discard the sample and prepare the new one. Each sample was measured three times. The last step for the data preprocessing was to make the subtraction of the baseline. Once all the preprocessing was done, the PCA was calculated. Fig 2 displays the resulting PCA for the six samples (three replicates) when using the response all four electrodes in the “humid electronic nose”. As observed, it was possible to discriminate among the samples studied. Moreover when the control data were introduced into the PCA analysis, all the days were situated around day 0, which is in agreement with the systems’ capacity to differentiate the control from the rest of the samples. Moreover, as the control solutions were measured at different days, the results suggest a high degree of reproducibility in the response of the electrodes. The clearer differentiation in the PCA plot was observed for the aqueous samples containing ammonia, acetic acid and for the Roquefort cheese.

4. Conclusions

A “humid electronic nose” prototype has been developed. The “humid electronic tongue” was tested for the discrimination of different samples displaying a different “aroma”. Results suggest that our "humid electronic nose" can be used effectively in a wide variety of applications, such as food industry, toxic compounds identification and many other fields in which substances with different "aromas" are involved.

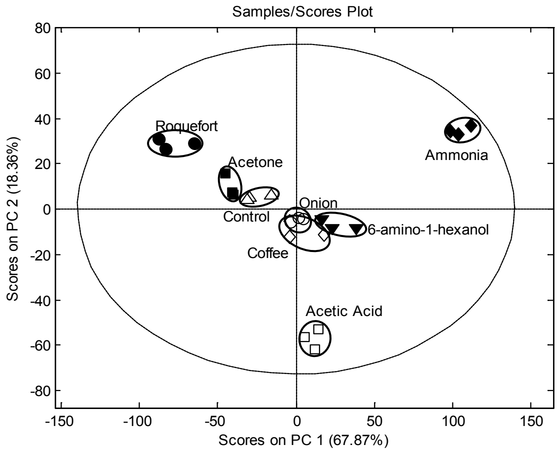

Fig. 2. PCA plot for the complete sample array

Acknowledgements

The financial support from the Spanish Government (project MAT2009-14564-C04) and the Generalitat Valenciana (Valencian Regional Government; project PROMETEO/2009/016).

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