



## Potentiometric sensor with lipid-polymer membranes for quality assessment of French red wines

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### ABSTRACT

The quality of wines resulting from vineyard management is important for consumers due to the taste and price of wines. In this work, the quality of red wines of three vintages has been investigated by potentiometric sensors with six all-solid-state electrodes. Two series of experiments were carried out. The first involved a qualitative analysis of wine of different vintages from freshly opened bottles. The second consisted of measurements on wine samples of different vintages that had been in contact with air for 15 min every day during 15 or 21 d. This sensor is characterized by good sensitivity to malic acid solutions of different concentrations. The polyphenol content and organoleptic acidity of red wines from different vintages have been established. The older the wine, the more polyphenol compounds it contains. The same sequence of polyphenol content can be observed in wine samples of different vintages that had been in contact with air for 15 min every day during 21 d. The samples of wine from freshly opened bottles from 2013 were more acidic than the wines between 2012 and 2014. The potentiometric results submitted to principal component analysis and cluster analysis have shown that wine samples from different vintages can be grouped according to their organoleptic acidity. The wine samples from freshly opened bottles and wine samples in contact with air belong to separate groups. It seems that the contact of wine with air influences its quality and plays the main role in wine grouping.

*Keywords:* Lipid-polymer membranes; All-solid-state electrodes; Principal component analysis; Potentiometric sensor; Vineyard management; Wine quality

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### 1. Introduction

Vineyard management can be compared to the management of a typical production company. It includes the following stages: planning, monitoring and analyzing all works constituting the wine production process as well as logistical and financial aspects [1,2]. A very important stage in the wine production process is the monitoring of parameters affecting the quality of the wine [1,3]. These parameters are responsible for the organoleptic features, wine quality, price and, above all, customer preferences. Therefore, grape growing, harvesting, production processes [4] and storage

should be properly carried out [5]. The quality of wines is important since it determines wine price and the volume of wine sales [1].

Different varieties of strains are used for the production of red wines. Among them, the Pinot Noir strain is one of the oldest grape varieties cultivated in France and used for the production of various types of wines [1]. Wines of this variety are considered one of the best in the world and are mainly produced in France.

The quality of the wine is determined based on taste evaluation. Taste is developed by many substances present in wine which affect each other. The main components of grape

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wines are water, ethanol, acids, glycerol, polysaccharides and other volatile compounds [6]. Their specific composition depends on the location of the vineyard, climatic conditions, vintage and production methods. Their chemical and organoleptic characteristics change over time [6].

In general, the taste of wine is estimated by organoleptic methods that mainly identify the sensory properties, for example, odor, taste, flavor [7,8]. Molecules responsible for taste in wine are caught by biological membranes of gustatory cells situated on the human tongue. The information is transformed into electric signals which are transmitted by nerve cell fibers to the brain. Such biological sensors are sensitive to taste substances, but the response may be subjective because it depends on the nature of the human [9–12].

Intensive research has been carried out to elaborate an artificial sensor for quality control of liquid food products. Sensor systems are one of the most promising ways for rapid, low-cost and objective liquid food-stuff quality control [13,14].

An electronic sensor based on a sensor array with 23 potentiometric cross-sensitive chemical sensors has been applied to the analysis of Italian red wines [15]. The sensor array included 14 sensors with chalcogenide glass and crystalline membranes, two metallics, six with plasticized polymer membranes and a pH glass electrode. This sensor array was sensitive to multiple substances that determine the taste and flavor of wine [15]. The other sensor array including 29 different chemical sensors has been used for qualitative analysis of mineral water and red wine [15,16]. An electronic tongue based on potentiometric chemical sensors was used to quantify the bitterness in samples of wines from South Africa [17]. It was found that bitter-tasting wines had higher concentrations of phenolic compounds than non-bitter wines. The other potentiometric approach based on permanganate ion fluxes across ion-selective electrode membranes has been proposed for measuring the total phenolic content in red wines [18].

A combined system consisting of a potentiometric electronic tongue and electronic nose has been used for the analysis of wine spoilage after contact with the air [19]. Taste sensors made from ultra-thin films have been used for red wine classification [20]. The combined application of an electronic nose and electronic tongue to samples of red wines with the same denomination and were produced in the same year from different vineyards has been discussed. It was shown that the electronic nose and electronic tongue yield different results [21].

One of the artificial taste sensors based on potentiometry is composed of a set of ion-selective electrodes (ISE) with different lipid-polymer membranes [8,22]. Such sensors were successfully used for discrimination of liquid products of various companies, such as coffee, beer, wine, milk, tomato juice and mineral drinking water [13,14]. The disadvantage of the potentiometric taste sensor with a set of ISE described above is the presence of inner solutions in each electrode. This results in lower stability and reproducibility of the electrode. Therefore, a new kind of taste sensor with several electrodes called the all-solid-state electrode (ASSE) was proposed for qualitative and quantitative analysis of tonic waters [23], discrimination of mineral water with

different CO<sub>2</sub> level [24], quality control of different natural waters [25] and for distinguishing of red wines from Polish and French vineyards [26].

In this paper the possibility of using the potentiometric sensor with ASSE elaborated in our laboratory for qualitative analysis of the red wine Barton & Guestier (B&G) of different vintages (2012, 2013, 2014) is presented. The red dry wines Pinot Noir have been produced in a French vineyard situated in Blanquefort within the Bordeaux Metropole. The effect of quality loss after 15 and 21 d of opening an appropriate wine bottle (every day for 15 min) has been also investigated. The stability and sensitivity of all ASSEs of taste sensor in solutions of malic acid have been established. Polyphenol content and organoleptic acidity in samples of these wines have been also investigated. The potentiometric results have been performed using principal component analysis (PCA) and cluster analysis (CA).

## 2. Experimental

### 2.1. Materials and reagents

Dry, red, Pinot Noir wines from the B&G vineyard of three vintages—2012, 2013, and 2014—were tested. Fifteen samples of each red dry wine taken from different bottles of appropriate vintages were analyzed. The taste sensor was tested in malic acid and sucrose solutions of 10<sup>-5</sup> M to 10<sup>-2</sup> M.

The following reagents were used for electrode preparation: (3,4-ethylenedioxythiophene (EDOT), poly(sodium 4-styrenesulfonate) (NaPSS), tetrahydrofuran (THF), poly(vinyl chloride) high molecular weight (PVC), hexadecylamine (Fluka, Sigma-Aldrich Chemie GmbH, Industriestrasse 25, 9470 Buchs, Switzerland), dioctyl phenylphosphonate (DOPP – 99,5%, Sigma-Aldrich Chemie GmbH, Industriestrasse 25, 9470 Buchs, Switzerland), hexadecyltrimethylammonium bromide, palmitic acid (99%, Aldrich), lauric acid (99,5%, Aldrich), decanoic acid (99%, Aldrich) and cholesterol (Aldrich). Electrolyte solutions containing malic acid (Alfa Aesar, Thermo Fisher (Kandel) GmbH, Erlenbachweg 2, 76870 Kandel, Germany), sucrose (Fluka), and potassium chloride (POCH), all of which were analytical purity grade in distilled water, were used.

### 2.2. Potentiometric method

The potentiometric taste sensor (Fig. 1a) was constructed from six working all-solid-state electrodes, a reference electrode (Ag/AgCl/Cl<sup>-</sup>) and a voltmeter Atlas (Sollich Company, ATLAS-SOLLICH Zakład Systemów Elektronicznych, Żłota 9, 80-297 Gdansk, Poland). It was connected to a computer. All electrodes were immersed in an appropriate tested aqueous solution. Each ASSE (Fig. 1b) contains a different lipophilic compound in a polymer membrane (ASSE preparation). These lipophilic compounds react with taste substances dissolved in the tested solution.

A high-input-impedance voltmeter was used for measuring the responses of ASSEs.

ASSEs were prepared in the following way. The conductive poly(3,4-ethylenedioxythiophene) polymer (PEDOT) was obtained by galvanostatic electrochemical synthesis [25–27]. Each ASSE consists of two layers (Fig. 1b). The inner

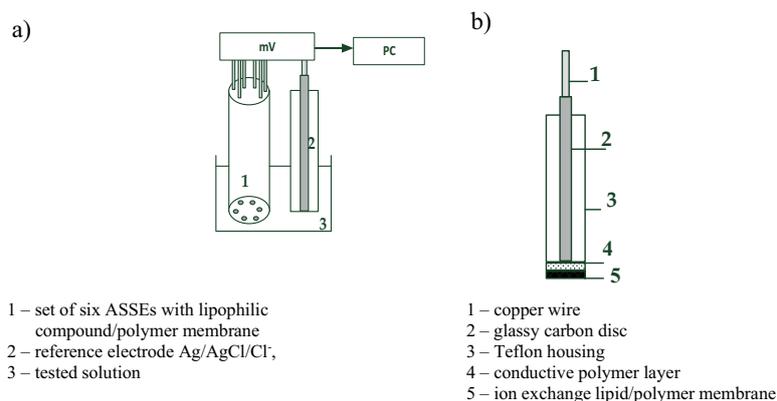


Fig. 1. (a) Experimental set-up and (b) working electrode of ASSE type.

layer is the conductive polymer (PEDOT) covered directly by GC. The outer layer is the lipid-polymer membrane prepared with PVC (~wt. 61%), plasticizer DOPP (~wt. 38.5%) and appropriate lipophilic compounds (~wt. 0.5%). The set of lipophilic compounds used in ASSE electrodes (e\_1 – e\_6) is presented in Table 1.

An appropriate lipophilic compound, PVC, and a plasticizer were dissolved in 5 ml of THF, put directly on GC, and dried for 5 h. Subsequently, all ASSEs were conditioned in the 10<sup>-3</sup> M KCl solution for 24 h before measurement.

### 2.3. Physicochemical method

The total content of phenolic compounds in wine samples was determined by the colorimetric method in the presence of the Folin-Ciocalteu reagent [28]. This reagent oxidizes the phenolic compounds present in red wine, and its reduction yields a blue solution. The total concentration of phenolic compounds was determined spectrophotometrically using the absorption band with a maximum at the  $\lambda = 780$  nm wavelength. The THERMO EVOLUTION 300 UV-Vis spectrophotometer was used for all measurements.

### 2.4. Organoleptic method

Acidity estimation of wines was performed by a group of selected sommeliers (five persons) using the organoleptic method. These individuals have worked in five-star hotels and have previous well established organoleptic experience. Acidity estimation was carried out in standard conditions as described in ISO standard [29]. The acidity of wines was

evaluated using the five-point Likert scale, in which the number five represents the most acidic solution.

### 2.5. Solution preparation and measurements

The sensitivity of the potentiometric sensor was measured in malic acid and sucrose solutions in the range 10<sup>-5</sup> M to 10<sup>-2</sup> M. The measurements were started from the lowest concentration of these compounds in the solution. The stability of taste sensors was investigated by measuring electrode potentials in the same malic acid solution every day over 5 d.

The B&G wine samples from three vintages (2012, 2013, 2014) were taken from appropriate bottles and used without dilution. Two series of experiments were carried out. The first involved a qualitative analysis with the taste sensor. The second consisted of measurements realized on samples taken from each bottle of wine immediately after their opening and also 15 and 21 d after their opening. During this period, each bottle of wine was opened every day to allow for air contact for 15 min. In each case, all ASSEs were conditioned in 10<sup>-3</sup> M KCl before and between measurements.

### 2.6. Mathematical methods

The obtained data from the potentiometric taste sensor array (voltages in mV) were interpreted using PCA and CA. PCA is a multivariate analysis commonly used in the literature [30] to reduce information included in multivariate-dimensional data space to that in two-dimensional space. Calculations were made with the Statistica v12 program for

Table 1  
Lipophilic compounds used in lipid/polymer membranes of six asses

	Number of electrodes					
	e_1	e_2	e_3	e_4	e_5	e_6
Lipid substance	Hexadecyltrimethylammonium bromide	Hexadecylamine	Palmitic acid	Lauric acid	Decanoic acid	Cholesterol
Charge of lipid substance	Positively charged		Negatively charged			Neutral

PCA and CA analysis. For other data (radar plot), MS Excel 2015 was used.

### 3. Results and discussion

Since wine, among other beverages, contains sour and sweet substances, the sensitivity of ASSE of the potentiometric taste sensor to these substances was investigated. Therefore, the sensor responses to malic acid concentration were examined in the range of  $10^{-5}$  M to  $10^{-2}$  M. The results are presented in the form of a radar plot (Fig. 2), in which the e\_1 to e\_6 axes show appropriate electrode responses (in mV).

It can be seen (Fig. 2) that the responses of negatively charged electrodes (e\_3 to e\_5) differ significantly with malic acid concentration. The slopes ( $S$ ) and correlation coefficients ( $r$ ) of  $E = f(c)$  are  $S = 41.2$  mV,  $r = 0.981$  for e\_3,  $S = 40.5$  mV,  $r = 0.983$  for e\_4 and  $S = 42.2$  mV,  $r = 0.9802$  for e\_5. This means that these electrodes are quite sensitive to malic acid concentration. This is not the case of positively charged electrodes (e\_1 and e\_2), for which the slopes of  $E = f(c)$  are 1.8 and 3.6 mV, respectively. The neutral electrode, e\_6, is characterized by small potential values in all solutions of different concentrations. Its potential changes

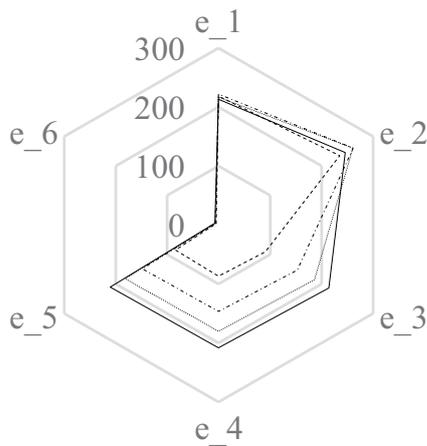
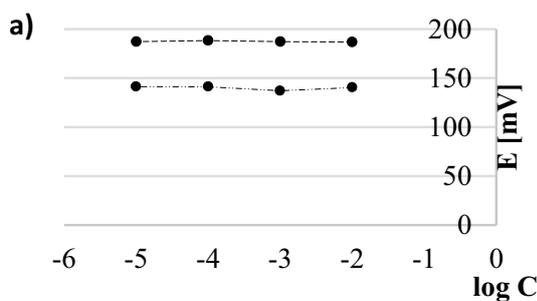


Fig. 2. ASSEs (e\_1 to e\_6) responses (in mV) in malic acid solutions of different concentrations: (a)  $10^{-2}$  M, (b)  $10^{-3}$  M, (c)  $10^{-4}$  M, and (d)  $10^{-5}$  M.



with acid concentration are within 5%. However, since the potentiometric sensor is characterized by global selectivity [31], all electrode responses contribute to its final result.

The responses of positively charged electrodes (e\_1 and e\_2) in sucrose solutions are presented in Fig. 3a, while the responses of the other electrodes are in Fig. 3b. The latter responses are characterized by small potential values. As shown, the differences of potential with sucrose concentration for all electrodes are small and in the range of experimental error (up to 3%). This means that all electrodes are not sensitive to the sucrose concentration in the range of  $10^{-5}$  M to  $10^{-2}$  M. This concentration effect was also observed in sucrose solutions measured by taste sensor with ISE [22].

It should be noted that the lipid-polymer membrane of electrode responses is not only linked to charge-transfer but also to change of double-layer capacity and its potential. Adsorption processes at the membrane interface should also influence the electrode response. The same response of ASSE in different sucrose concentrations may be caused by the non-ionic nature of sucrose molecules.

The stability of ASSEs was investigated in  $10^{-2}$  M malic acid solutions over 5 d. The results for three electrodes—positively (e\_1) and negatively (e\_3) charged and neutral (e\_6)—are presented in Fig. 4. The responses of e\_1 and e\_3 are the same over 1 d, within experimental error. After that period, an increase of electrode potentials was observed. In contrast, the neutral electrode (e\_6) is stable for a longer time. This means that after 1 d of measurements,

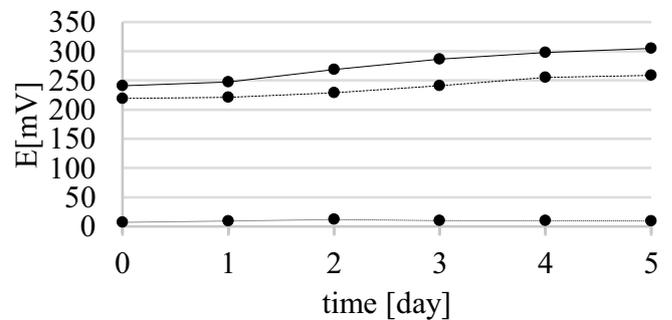


Fig. 4. Stability of ASSEs with e\_1 – hexadecyltrimethylammonium bromide, e\_3 – palmitic acid, e\_6 – cholesterol in malic acid of  $10^{-2}$  M concentration during the time.

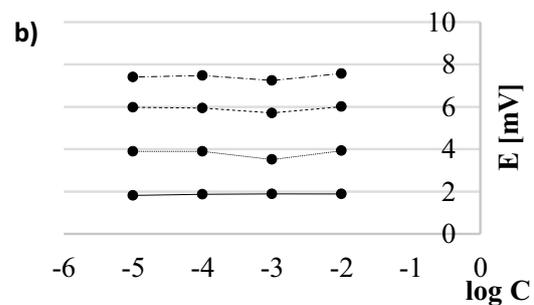


Fig. 3. Potentiometric sensor responses (in mV) in sucrose solutions of different concentrations: (a) e\_1; e\_2 and (b) e\_3; e\_4; e\_5; e\_6.

the electrodes should be renewed. It should be added that in hydrochloric acid solutions of different concentration, the ASSEs of our potentiometric sensor are stable for a longer time (3 d) [27].

The polyphenol content in samples of wines from freshly opened bottles and samples after 21 d, during which bottles were exposed to air for 15 min each day, are presented in Table 2. The mean values of sommeliers' estimations of organoleptic acidity in the various samples of B&G wine vintages (2012, 2013 and 2014) from freshly opened bottles are also included. It should be added that the wine samples in repeated contact with air were not appropriate for organoleptic analysis.

The electrode responses of e\_1 to e\_5 differ significantly with the vintage of wine. This is not the case for the e\_6 electrode, for which the potential values are very small compared to those of the other electrodes. The raw data from Fig. 5 were analyzed by PCA. The results are shown in Fig. 6. The first two principal components (PC 1 and PC 2) explain 99.98% of the variability of the data. This indicates the existence of a strong correlation in the data.

The points corresponding to wine samples from the same vintage were distributed in different quarters on the PCA plot (Fig. 6a). The samples of B&G 2012 are characterized by negative PC 1 and PC 2 values, while the opposite is true of B&G 2013. The solid line represents the increase of acidity in wine samples, while the dotted line shows the increase of polyphenol content.

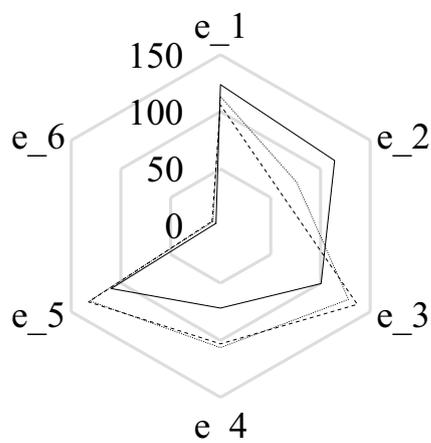


Fig. 5. ASSEs responses in samples of B&G wines of different vintages: ---, 2014, —, 2013, ···, 2012.

Table 2  
Physicochemical and organoleptic results

No.	Wine	Polyphenol content (mol/dm <sup>3</sup> )	Polyphenol content (mol/dm <sup>3</sup> ) after <sup>b</sup>	Organoleptic acidity <sup>a</sup>
1	B&G 2012	0.391 ± 0.02	0.367 ± 0.04	2.67 ± 0.47
2	B&G 2013	0.380 ± 0.04	0.355 ± 0.03	3.17 ± 0.69
3	B&G 2014	0.367 ± 0.03	0.342 ± 0.03	2.33 ± 0.47

<sup>a</sup>Mean value of results obtained by a group of sommeliers; <sup>b</sup>from bottles 21 d after opening, during which time bottles were exposed to air for 15 min each day.

The direction of lines agrees with the organoleptic acidity of wines from different vintages and with the polyphenol content estimated spectrophotometrically (Table 2).

The CA analysis (Fig. 6b) shows that these results can be divided into two groups. One group consists of wine samples from the year 2013. This wine is more acidic compared to other vintages of B&G wines. The second group is made up of wine samples from other vintages (2012 and 2014). These wines have the same acidity, but their acidity is lower in comparison to wine from the 2013 vintage. This means that each group is formed by wine samples of similar acidity. This is not the case for polyphenol content. It seems that the main feature affecting the grouping of wine samples is their acidity.

Therefore, PCA and CA results suggest that the sensor with six ASSEs can be used for grouping of wines of similar acidity.

The next series of experiments involved the potentiometric measurements of wine samples of different vintages 15 and 21 d after opening, during which time the bottles of wine were opened for 15 min every day.

The ASSE responses in samples of wines from three vintages are presented in Fig. 7.

As shown, the e\_1 to e\_5 responses in samples of wine after contact with air differ significantly compared to those of wine samples from freshly opened bottles. This is not the case for the e\_6 electrode, for which responses are very small and differences are within experimental error.

The raw data presented in Fig. 7 were also subjected to PCA and CA analysis (Figs. 8a and b). As shown in Table 3, the first two principal components explain 96.58% of the variability of the data. This suggests that both components play significant roles in the model.

The points for wine samples from freshly opened bottles (no. 1) are located on the negative side of PC1 (Fig. 8a), independently of wine vintage. In contrast, points representing samples of wine after contact with air (no. 2 and 3) are situated on the positive side of the PC1 axis.

To group the wine samples, CA analysis of raw data was performed (Fig. 8b). The examined samples of wine belong to two main groups. The first one is composed of wine samples that were in contact with air (no. 2 and 3) for several days. The other one is made up of wine samples from freshly opened bottles (no. 1). This means that the grouping of each wine sample depends on the contact time with air, which results in acidity increasing and diminishing of polyphenol content in those samples (Table 2). This effect is the major contributor to wine grouping. It should be added

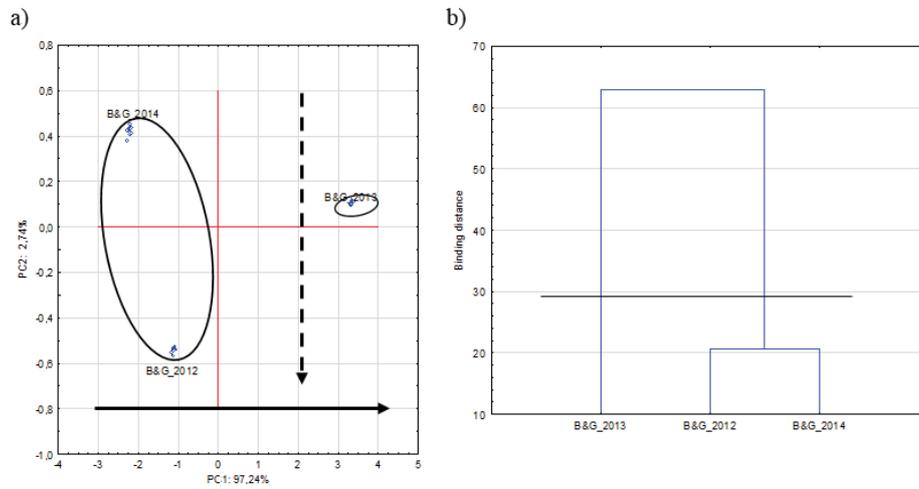


Fig. 6. (a) Biplot of PCA model of the wine data, dotted line – the direction of polyphenol content, solid line – the direction of organoleptic acidity and (b) CA analysis.

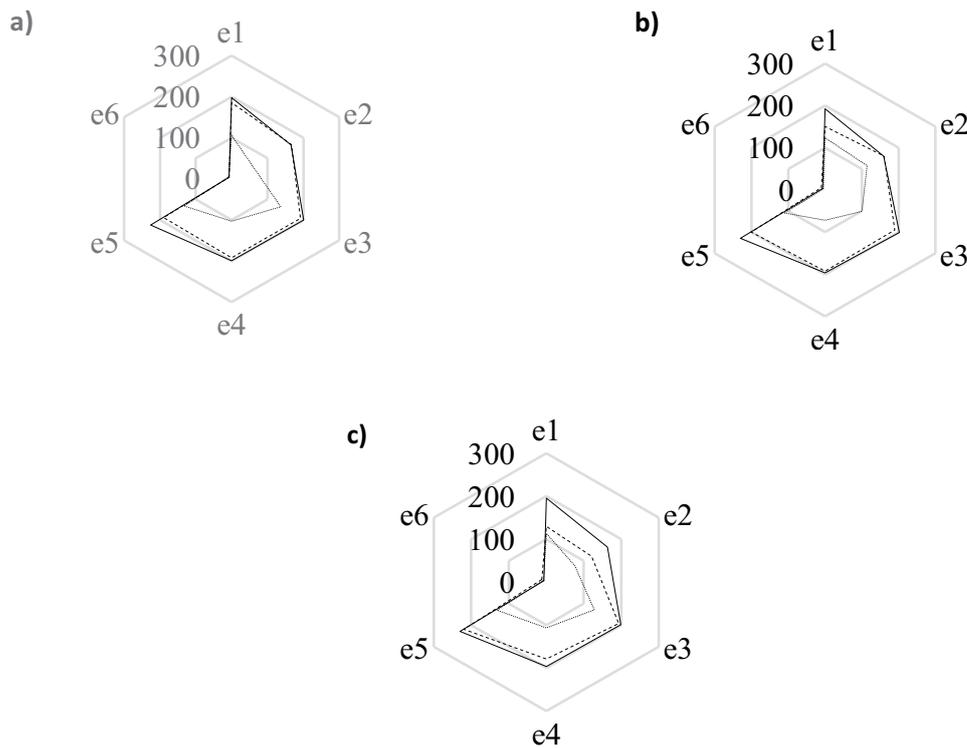


Fig. 7. ASSE responses in samples of B&G wines of different vintages: (a) 2014, (b) 2013, and (c) 2012, ..... from freshly opened bottles, - - - after 15 d of contact with air, — after 21 d of contact with air.

Table 3  
Eigenvalues and variability explained by principal components in the PCA

Principal components	PC 1	PC 2	PC 3	PC 4	PC 5	PC 6
Eigenvalues	4.449	1.346	0.158	0.043	0.003	0.002
Variability (%)	74.147	22.430	2.626	0.724	0.043	0.030
Cumulative (%)	74.147	96.577	99.203	99.927	99.970	100.00

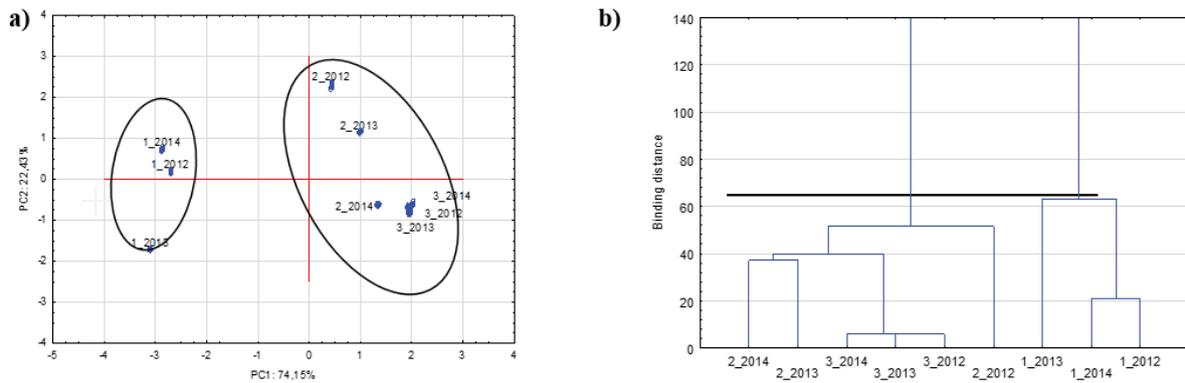


Fig. 8. (a) Biplot for different wine samples from 2014, 2013 and 2012 years, No. 1: samples from freshly opened bottles, No. 2: after 15 d of contact with air, No. 3: samples after 21 d of contact with air and (b) CA analysis.

that the contact of wine with air causes oxidation processes. As a result, the quality of wine decreases. The spoilage of wine when in contact with air was also confirmed by a combined system consisting of a potentiometric electronic tongue and a humid electronic nose [19].

#### 4. Conclusion

The potentiometric sensor with six ASSEs elaborated in our laboratory is characterized by good sensitivity to malic acid solutions of different concentrations. However, the slopes of  $E = f(c)$  of negatively charged electrodes are higher than the slopes of positively charged ones. The stability of ASSEs in malic acid solutions is good over 1 d of usage. After that time, the electrodes should be renewed for long-standing measurements.

The polyphenol content and organoleptic acidity of B&G red wines from different vintages were established. The older the wine, the more polyphenol compounds it contains. The same sequence of polyphenol content was observed in wine samples of different vintages that had been exposed to air for 15 min every day over 21 d. The group of sommeliers estimated that the wine from 2013 was more acidic than the wines from 2012 and 2014.

The potentiometric results obtained by the taste sensor were submitted to PCA and CA analysis. It was shown that wine samples from freshly opened bottles of different vintages can be grouped according to their acidity. The series of experiments that involved wine samples of different vintages that had been exposed to air (for 15 min every day for 15 and 21 d) showed that samples of wine from freshly opened bottles and samples of wine after contact with air are grouped separately. It seems that exposure of wine samples to air resulting in increasing of acidity and diminishing of polyphenol content influences its quality and is the major contributor to wine grouping.

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