Qualitative and quantitative monitoring of drinking water through the use of a smart electronic tongue

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Abstract. The aim of this work was to evaluate a smart electronic tongue device as an alternative for qualitative and quantitative monitoring of drinking water. The smart electronic tongue consisted of a voltametric polypyrrole sensor array, coupled with a multi-channel electronic system (multipotentiostat) based on PSoC technology controlled from a smartphone with data acquisition and a control app. This device was used in the monitoring of drinking water from the Sincelejo city water supply system; also, water samples collected and analyzed by the public health agency were used. The voltammetric measurements carried out with the smart electronic tongue showed cross-sensitivity of the polypyrrole sensor array, which allowed the discrimination of the samples through of principal component analysis by artificial neural networks. In addition, the voltammetric signals registered with the smart electronic tongue allowed, through Partial Least Square (PLS) by artificial neural networks analysis, estimating the concentrations of some important analytes in the evaluation of the physicochemical quality of drinking water with R2 values higher than 0.70. The results allowed to conclude that the smart electronic tongue can be a valuable analytical tool that allows, in a single measure, to perform qualitative and quantitative chemical analysis (alkalinity, calcium, residual chlorine, chlorides, total hardness, phosphates, magnesium, and sulphates), it is also a fast, portable method that can complement traditional analyzes.

1 Introduction

In recent decades, there has been an increase in interest and concern for the quality control of food, drinking water, beverages, and in general, products for human consumption. To accomplish this control, in addition to reliable methods, it has been sought to have fast methods that allow real-time and online surveillance. In the particular case of drinking water, analyses are usually carried out using techniques and methods that mostly require sophisticated and specialized equipment, such as UV-Vis spectrometers, chromatographs, mass spectrometers, infrared spectrometers, atomic absorption spectrometers, among others (Richardson et al., 2017; Rice et al., 2017). In general, this kind of equipment is expensive and requires qualified personnel for their handling, they are also bulky equipment that consume significant amounts of energy, and can only operate in facilities or laboratories suitable for their operation. Furthermore, most of the analyses require sample pre-treatment, long processing times, and generate a considerable amount of chemical waste. These conditions and restrictions in traditional analytical approaches have led to the development of cheaper, faster, easier, and more efficient alternative technologies. The above has led to the generation of new technologies, among which there is the electronic tongues (Arrieta et al., 2019; Atas et al., 2020; Dias et al., 2015; Legin et al.,2019).

Electronic tongues are analytical devices, made up of a non-specific chemical sensor array, with cross-sensitivity, coupled to a multichannel measurement system and an app or software that allows pattern recognition (Vlasov et al., 2005). A certain analogy can be established between the human gustatory taste system and electronic tongues, in the sense that we can find some approximations in its structure and principles of operation. Figure 1, it is presented a comparative scheme that shows the similarities between the functioning of the human taste system and the artificial system.

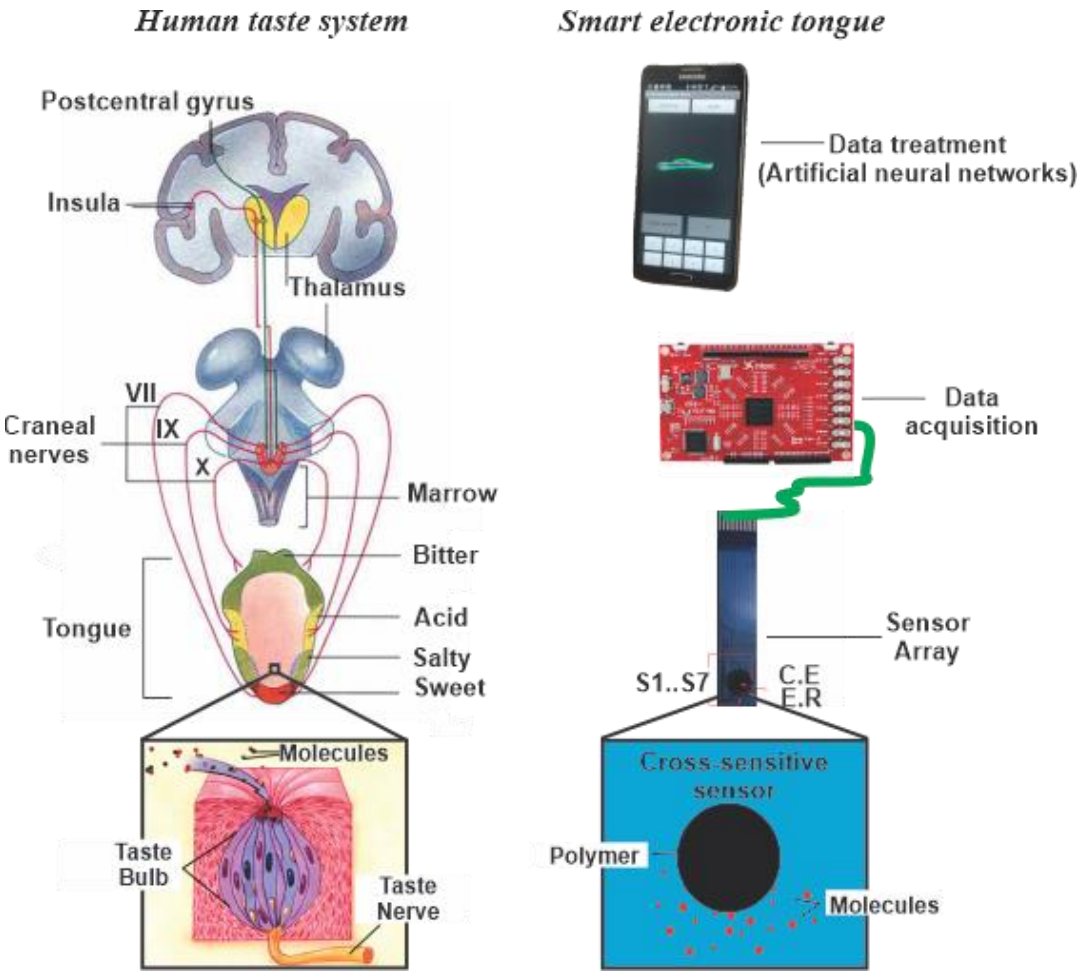

Figure 1. Functional similarities between the human taste system and an artificial system (smart electronic tongue).

Although in the electronic tongue devices have been used various analytical principles such as optical, mass, frequency measurements, among others (Khan et al., 2016; li et al., 2019; Kovacs et al., 2020; Sehra et al., 2004; Aydemir and Ebeoglu, 2018), the ones based on electrochemical measurements have been the most widely accepted. Devices based on potentiometric and voltammetric electrochemical measurements have been more widely accepted and have shown their effectiveness in the analysis of different types of beverages (Arrieta et al., 2019; Belugina et al., 2020; Totova and Nachev, 2020; Marx et al.,

2017). Electronic tongues based on voltammetric measurements have advantages such as greater ease of sensors elaboration, low sensitivity to electronic noise, high analytical sensitivity, and versatility in terms of the voltammetric technique used (square wave, cyclic, pulse, etc.).

The electronic tongues have been used in the analysis of mineral waters (Sipos et al., 2012), waste waters (Legin et al., 2019), bottled waters (Dias et al., 2015). and qualitative (sample classification) and quantitative analyses on analytes such as Na^+, K^+, Ca^{2+}, Cl^-, NaCl, NaN_3, $NaHSO_3$, ascorbic acid, and NaOC (Winquist et al., 2011; Atas et al., 2020), among others. However, no reports have been found on the application of this technology in the analysis of drinking water from distribution networks and on the analytes of greatest interest in the evaluation of its physicochemical quality such as hardness, alkalinity, chlorides, sulphates, chlorine, etc.

The reported electronic tongue devices are mostly laboratory equipment, which limits their portability for on-site analysis. In this work, the application of a portable smart electronic tongue is reported, made up of a miniaturized polypyrrole (PPy) sensor array, a multichannel device made under PSoC (Programable System on Chip) technology and a smartphone equipped with an Android app. The recorded data were analyzed with methods of pattern recognition and regression by Partial Least Squares based on artificial neural networks. This smart electronic tongue was used to qualitatively and quantitatively analyze samples taken from the 22 points (hydrants) of the distribution system.

2 Materials and Methods

2.1 Collection of samples and sampling area

The samples were taken from the drinking water supply network at the hydrants defined by the drinking water service provider company (ADESA SAESP), located in communities 1, 2, 3, 4, 5, 6, 7, and 9 of the city of Sincelejo – Colombia (Sincelejo mayorship, 2017), located in the northeast of the country at 9° 18" north longitude, -75° 23" latitude, west of the Greenwich meridian, altitude of 213 MSL. For the sampling, the national guidelines on the minimum number of samples and the distribution of sampling points established for the populations according to their number of inhabitants were taken into account. The sampling hydrants were defined taking into account the programming of the operating company of the water supply system and the entity of surveillance and control of the quality of drinking water. Table 1 presents the summary of the programming of the sampling carried out, in which the location or geographical area was noted; commune (C), sector (S). and the place of sampling point or hydrant (H). For the sampling procedure, the protocols established by the national health authority were followed (National Institute of Health, 2019).

The samples were divided into aliquots to carry out the different analyses. The characterization of the physicochemical analyzed parameters was carried out in the facilities of the departmental reference laboratory of Public Health of the Sucre Department, an entity in charge of exercising control and monitoring of water for human consumption and its characteristics. The methods and techniques used for each of the parameters analyzed were those established in the standard analysis methods required by national regulations (Richardson et al., 2017; Rice et al., 2017).

Table 1. Drinking water sampling location data.

Sample code	Location (Commune and sector)	Sampling location (Hydrant)	Geographical coordinates
M1	C1S3	H2014	Latitude N 9° 18´25.30´´/Longitude O -75° 24´40.70´´
M2	C2S18	H2016	Latitude N 9° 18´38.50´´/Longitude O -75° 24´03.34´´
M3	C3S7	H2015	Latitude N 9° 17´26.24´´/Longitude O -75° 24´43.10´´
M4	C3S8	H2013	Latitude N 9° 16´58.67´´/Longitude O -75° 24´24.26´´
M5	C3S8	H2012	Latitude N 9° 17´07.08´´/Longitude O -75° 24´22.16´´
M6	C4S12	H2011	Latitude N 9° 17´21.76´´/Longitude O -75° 23´53.74´´
M7	C4S12	H2008	Latitude N 9° 17´49.72´´/Longitude O -75° 23´34.10´´
M8	C4S15	H2029	Latitude N 9° 18´01.62´´/Longitude O -75° 23´26.57´´
M9	C4S15	H2007	Latitude N 9° 18´15.46´´/Longitude O -75° 23´57.88´´
M10	C5S25	H2030	Latitude N 9° 18´25.66´´/Longitude O -75° 23´40.97´´
M11	C5S26	H2027	Latitude N 9° 18´13.86´´/Longitude O -75° 23´15.38´´
M12	C5S33	H2004	Latitude N 9° 17´56.10´´/Longitude O -75° 23´19.42´´
M13	C5S33	H2005	Latitude N 9° 18´01.62´´/Longitude O -75° 23´26.57´´
M14	C5S34	H2028	Latitude N 9° 18´27.19´´/Longitude O -75° 22´52.60´´
M15	C6S23	H2019	Latitude N 9° 18´46.03´´/Longitude O -75° 23´57.16´´
M16	C6S23	H2017	Latitude N 9° 19´09.85´´/Longitude O -75° 23´47.25´´
M17	C7S27	H2003	Latitude N 9° 18´52.52´´/Longitude O -75° 23´02.86´´
M18	C7S34	H2026	Latitude N 9° 18´09.36´´/Longitude O -75° 23´43.21´´
M19	C7S49	H2001	Latitude N 9° 18´12.13´´/Longitude O -75° 22´45.44´´
M20	C7S51	H2006	Latitude N 9° 18´16.93´´/Longitude O -75° 23´22.80´´
M21	C9S40	H2022	Latitude N 9° 17´49.89´´/Longitude O -75° 23´03.97´´
M22	C9S40	H2024	Latitude N 9° 17´50.47´´/Longitude O -75° 22´41.30´´

2.2 Smart electronic tongue device and measurements

The smart electronic tongue developed in our laboratory consisted of a voltammetric PPy sensor array and a portable multipotentiostat controlled with a smartphone. For the elaboration of the sensor array, a card with screen-printed electrodes

from BVT Technologies (AC9C) was used, which consists of an auxiliary or counter electrode (CE), an Ag/AgCl reference electrode (ER), and seven working electrodes of graphite, which were used as substrates for the generation of the sensors. Thus, the sensor array consisted of seven PPy voltammetric sensors doped with seven different doping agents: PPy / DBS (PPy doped with sodium dodecyl benzene sulfonate), PPy / SO4 (PPy doped with sodium sulphate), PPy / SF (PPy doped with sodium persulfate), PPy / FCN (PPy doped with sodium ferrocyanide), PPy / TSA (PPy doped with p-toluene sulfonic acid), PPy / AQDS (PPy doped with disodium salt of the acid anthraquinone-2,6-disulfonic), and PPy / PC (PPy doped with lithium perchlorate).

The sensor array was prepared by chronoamperometric electropolymerization of pyrrole at 0.8 V, using an EG&G 2273 PAR potentiostat/galvanostat, controlled with PowerSuite software. The PPy with each of the dopants was electrodeposited on the graphite substrates arranged in a circular way on the commercial AC9C card. Table 2 shows the experimental conditions used in the synthesis of the sensor array.

Table 2. Experimental conditions for the electropolymerization of the sensor array

Sensor	**Acronym**	**Concentration Pyrrole/Doping Agent [M]**	**Polymerization time (s)**
S1	PPy/SO4	0.1/0.05	55
S2	PPy/DBS	0.1/0.1	50
S3	PPy/SF	0.1/0.05	65
S4	PPy/FCN	0.1/0.1	60
S5	PPy/PC	0.1/0.1	60
S6	PPy/TSA	0.1/0.1	70
S7	PPy/ AQDS	0.1/0.05	60

The portable multipotentiostat was made on a FREESOC card with a PSoC 5LP microchip (Programmable System on Chip), which was programmed with the PSoC creator software. This electronic device was designed to simultaneously register the voltammetric signals of the seven sensors of the array through seven measurement channels. In addition, a Bluetooth card was incorporated for data transmission to a smartphone equipped with an Android app designed to control the device and record data. Details on the electrochemical polymerization techniques, the development of the electronic device and the control Android app have been previously reported (Arrieta and Fuentes, 2016; Arrieta et al., 2015, Arrieta et al., 2016). Figure 2 presents an image of the smart electronic tongue and its three fundamental components are highlighted.

The measurements carried out with the smart electronic tongue were carried out on 10 mL of sample at room temperature and without previous treatment. 7 replicates of each measure were made. The voltammetric signals were recorded at a sweep rate of 100 mV s^{-1}, in a potential range of -1.0 V to 0.5 V with an initial potential of 0.0 V.

2.3 Data processing and evaluation of the qualitative and quantitative analysis carried out with the smart electronic tongue

From the obtained signals during the measurements carried out with the smart electronic tongue, the current data generated by the sensor array was recorded. Each sensor generated a voltammogram of each sample, composed of 100 data, which allowed having 700 data with the entire sensor array, each one of them was a variable in the data matrix for each sample, which constituted a species "fingerprint" of the sample. Thus, when analyzing all the samples, a matrix of 107,800 data was constructed (700 variables x 22 samples x 7 replicates).

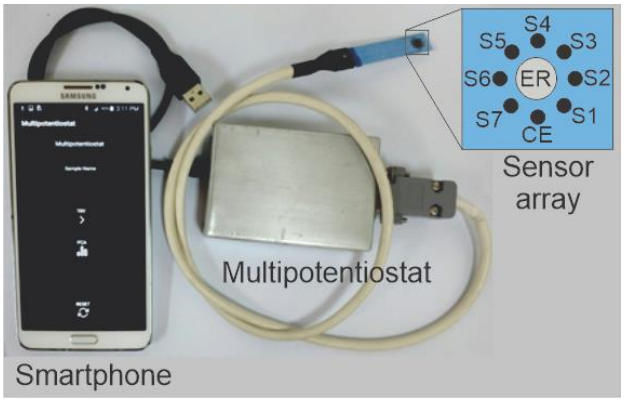

Figure 2. Image of the smart electronic tongue formed by the miniaturized sensor array, portable electronic device and smartphone with an Android app.

To validate the classification capacity (qualitative analysis) in drinking water samples, the matrix was subjected to a pattern recognition analysis by applying artificial neural networks for principal component analysis. By evaluating the results and the reproducibility of the method, the measurement procedure was repeated on a different group of samples, sampled 15 days after the first discrimination test and with the same sampling protocol, measurement with the smart electronic tongue and treatment of data were applied. The purpose of these experiments, was to verify the repeatability of the results obtained with the smart electronic tongue.

On the other hand, a quantitative analysis was carried out from regression models using artificial neural networks for Partial Least Squares, to establish a correlation between the voltammetric measurements registered with the smart electronic tongue and the concentrations of eight physicochemical parameters related to drinking water quality (alkalinity, calcium, residual chlorine, chlorides, total hardness, phosphates, magnesium, and sulphates) were evaluated. The physicochemical parameters were determined using the traditional methods validated by the norms and standardized methods (Richardson et al., 2017; Rice et al., 2017). That is, created prediction models were generated from the data obtained in the characterization process with the smart electronic tongue (matrix X, independent variables) and the physicochemical parameters determined using the traditional methods in each water sample (Y matrix, dependent variables). In this way, the concentrations of the physicochemical

parameters of drinking water determined by traditional methods were evaluated against those predicted by smart electronic tongue through regression models.

The chemometric treatment of the data was carried out using specific artificial neural networks designed under the MATLAB V 7.12 program using Neural Network Toolbox v.3.0 (Kong et al., 2017). The data were not pretreated and to select the number of latent variables, a "cross-validation" was performed before building the prediction model. Calibration and validation were performed from the concentrations determined by the methods and techniques established in the standard analysis methods required by national regulations (Richardson et al., 2017; Rice et al., 2017).

3 Results and discussions

3.1 Voltammetric Response of Smart Electronic Tongue

Once the samples were collected at the sampling points, the respective measurements were done by using the smart electronic tongue in an aliquot of 10 mL and the measurement time was 4 minutes per sample. The voltammetric signals showed cross sensitivity in the sensors; each sensor presented a particular response in the same sample, which means that each one provided information about the analyzed sample, which constitutes the “fingerprint”, with anodic and cathodic processes of the PPy against the samples (Arrieta et al., 2004). In Figure 3, the response of the sensor array against sample M1 (C1S3-H2014) is presented as an example. It can be showed in the graphs, that the voltammetric signal of the sensor S1 (PPy/SO4), shown an anodic process at - 0.249 V and in the cathodic sweep a reduction process could be observed at - 0.875 V. The response of the sensor S2 (PPy/DBS) shown a redox process, with an oxidation peak at - 0.109 V and a wide reduction peak in the cathodic scan at - 0.799 V.

The signal recorded with S3 sensor (PPy/SF) consisted of two anodic processes at 0.249 V and - 0.351 V. On the other hand, the voltammetric response of S4 sensor (PPy/FCN) presented a signal with poorly defined anodic and cathodic process at 0.287 V and - 0.124 V, respectively. The voltammetric responses of the S5 sensor (PPy/PC) and S6 (PPy/TSA) shown in both cases a redox process, composed of an anodic peak at 0.03 V for PPy/PC and - 0.252 V for PPy/TSA. Whereas cathodic scanning it could be seen that PPy/PC presented the reduction peak at - 0.747 V, while the PPy/TSA cathodic scanning shown the reduction peak at - 0.821 V. The voltammetric signal of the S7 sensor (PPy / AQDS), presented an oxidation process in the cathodic wave at 0.202 V.

Besides, the cross sensitivity was evaluated, which is the capacity of the sensor array to generate particular signals in front of each one of the samples. In Figure 4 the behavior of the S1 sensor (PPy/SO4) against some water samples taken at different sampling points (M1, M2, M3, M4, and M5) is shown as an example. Accordingly Thus, the main differences are observed in the position of the peaks (redox potentials) of each of the sensors and the shapes of the curve. This allows obtaining information from the analyzed water samples. Starting from this fact, and to extract the information contained in the signals, a pattern recognition analysis was performed using artificial neural networks for principal component analysis.

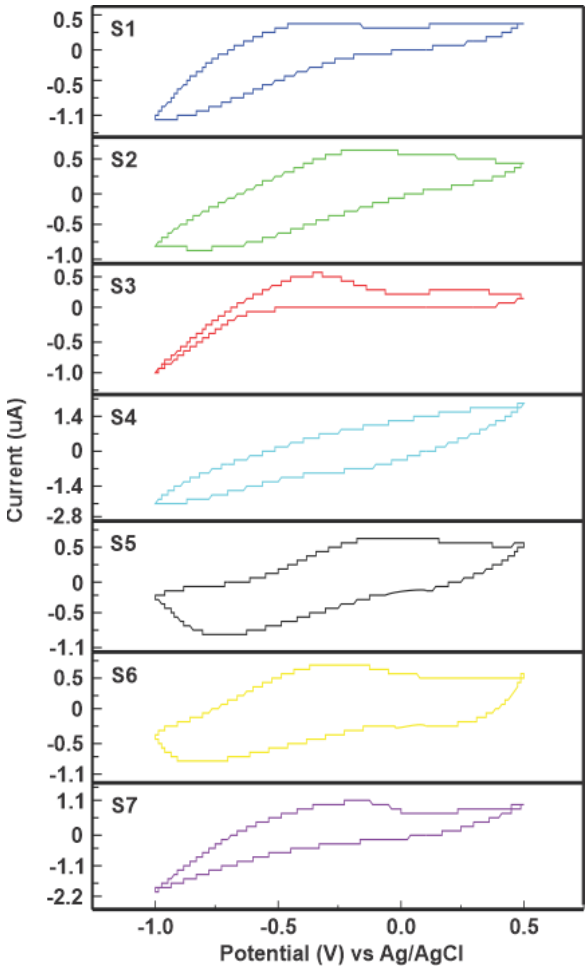

Figure 3. Voltammetric signals from the smart electronic tongue sensor array recorded in the drinking water sample M1 (C1S3 - H2014)

In summary, it could be shown that the shape and position (redox potentials) of the peaks in the voltammetric signals were markedly different in each of the sensors and a different signal pattern was recorded in each sample, allowing them to have together a "fingerprint" of each one. In general terms, the signals were related to the entry and exit of ionic species from the water samples in the polymeric film of the PPy sensor to maintain its electroneutrality, which is why the obtained signals contain information of each of the samples analyzed (Arrieta et al., 2004).

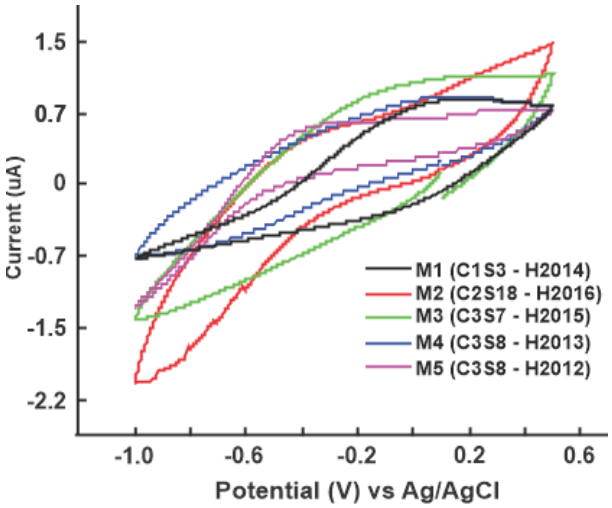

Figure 4. Voltammetric signals of the S1 sensor (PPy/SO4) recorded in different samples of drinking water.

3.2 Qualitative analysis

From the recorded signals, a matrix was constructed with the data obtained in each of the measurements. The matrix was used to perform a pattern recognition analysis to classify the samples, Figure 5 shows the result obtained from the pattern recognition analysis by artificial neural networks for principal components, applied to the values supplied by the voltammetric signals recorded for the different water samples. The two principal components represented show a variance of 72.09%.

In Figure 5, each point corresponds to a sample taken from a hydrant or sampling point taken in the respective geographical area (commune C, sector S, and hydrant H). The first principal component (PC 1) summarizes the most information with 56.49% and the second principal component (PC 2) also collects a significant amount of 15.85%. As can be seen, the different analyzed samples are remarkably distributed in the plane of the principal components with a higher concentration close to zero in both axes. In the area located in the lower right part of the graph (Figure 5 a), groups of samples may appear to be overlapping due to the high concentration of points (samples). However, when enlarging the area, it can be seen that none of the samples overlap (Figure 5 b).

The samples with the greatest separation in the plane of the principal components: M1 (C1S3-H2014), M2 (C2S18-H2016), M3 (C3S7-H2015), M4 (C3S8-H2013), and M5 (C3S8-H2012), belong to communes 1, 2, and 3, which are found in the western part of the city, with sample M1 being the one with the highest degree of separation and the only sample from commune 1. Whereas samples M4 and M5 belong to the same commune and the same sector present a certain proximity. This trend in the spatial distribution of the samples without forming defined groups in the principal components plane, may be due to the fact that the water supply is carried out from the main treatment site and reaches different points where temporary storage is carried out and re-pumping towards the geographical location areas. This distribution process with different storage sites can generate slight differences in the composition of some components due to the lack of homogeneity in the re-pumping points where there may be differences in storage temperature, possible mixtures, different cleaning protocols, among others. In

addition to other factors such as differences in sampling hours, maintenance of distribution lines, etc. This result showed the discrimination capacity of the smart electronic tongue against drinking water samples.

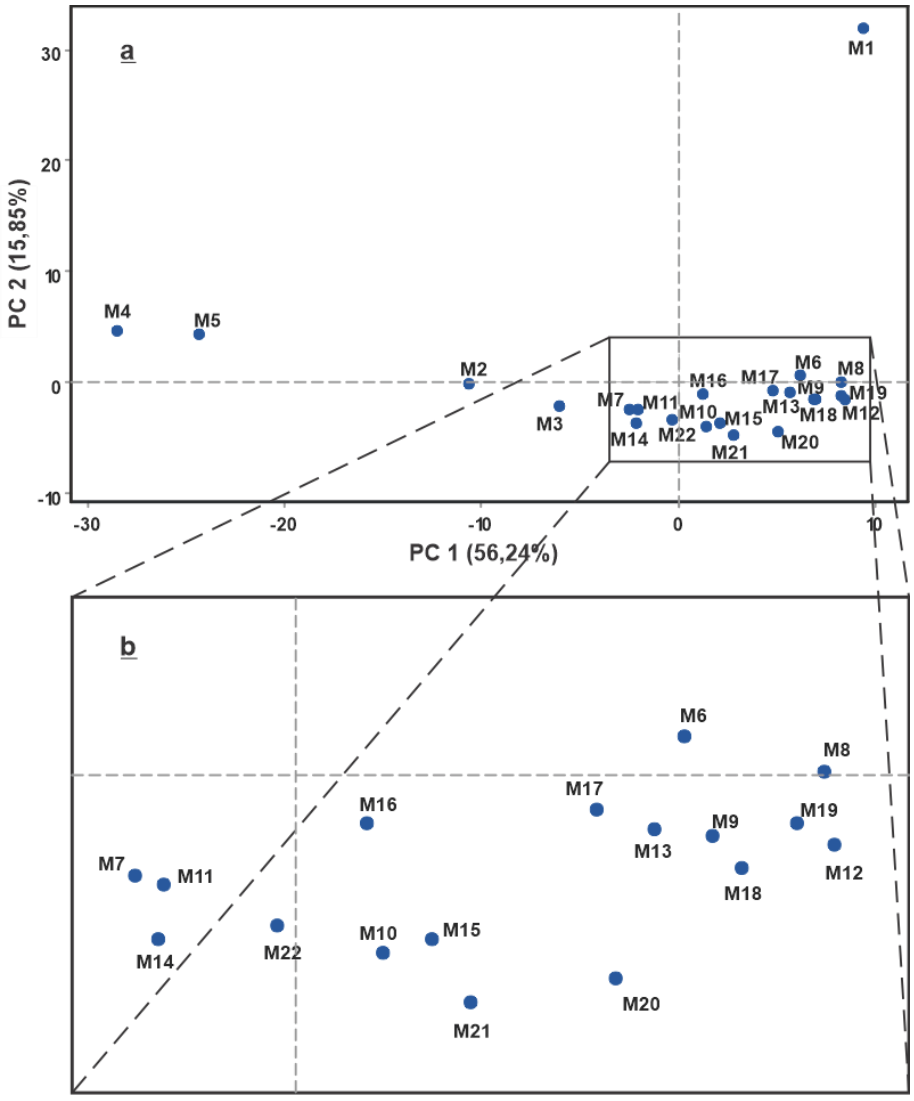

Figure 5. Plot of principal component score of signals collected in drinking water samples by smart electronic tongue.

Furthermore, a second test was carried out to corroborate the quality and reproducibility of these results. This trial consisted of repeating the experiences after 15 days. For this, a new group of samples collected at the same points was used and then followed with the same protocols for sampling and recording signals with the smart electronic tongue. In this way, after treating the data with the artificial neural network method for principal component analysis, a new principal component scores graph was generated from the new experiments.

When comparing the distribution and the positions of the samples with those obtained from the experiments carried out in the first test (Figure 5), it could be observed a great similarity in the results. The information collected for PC 1 and PC 2 was 62.15% and 9.89% respectively, for a total of 72.04% of the information collected for the total variance, a value similar to that obtained in the first trial (72.09%). Although there are small variations, which may be the product of differences between the physicochemical characteristics of the samples, there is a high degree of reproducibility.

3.3 Quantitative Analysis

The ability of the smart electronic tongue to provide quantitative information of the water samples under study was explored, by obtaining correlations between the voltammetric measurements recorded by the smart electronic tongue and the concentration of some compounds or substances present in drinking water samples. For this, the data of the two sets of 22 samples were taken to guarantee the robustness of the resulting models.

To carry out the extraction of quantitative information, regression models of artificial neural networks for Partial Least Squares were used and eight relevant physicochemical parameters were chosen in the evaluation of the quality of drinking water (hardness, alkalinity, chlorides, residual chlorine, sulphates, magnesium, calcium, and phosphates).

The results of the application of the regression analysis are shown in Figures 7 and 8 (the results were divided into 2 figures to improve the visualization). Calibration and validation were performed from the concentrations determined by traditional methods of analysis as explained in the materials and methods section. In figure 7, the regression graphs obtained from the application of the models on the parameters alkalinity, calcium, hardness, and phosphates are presented.

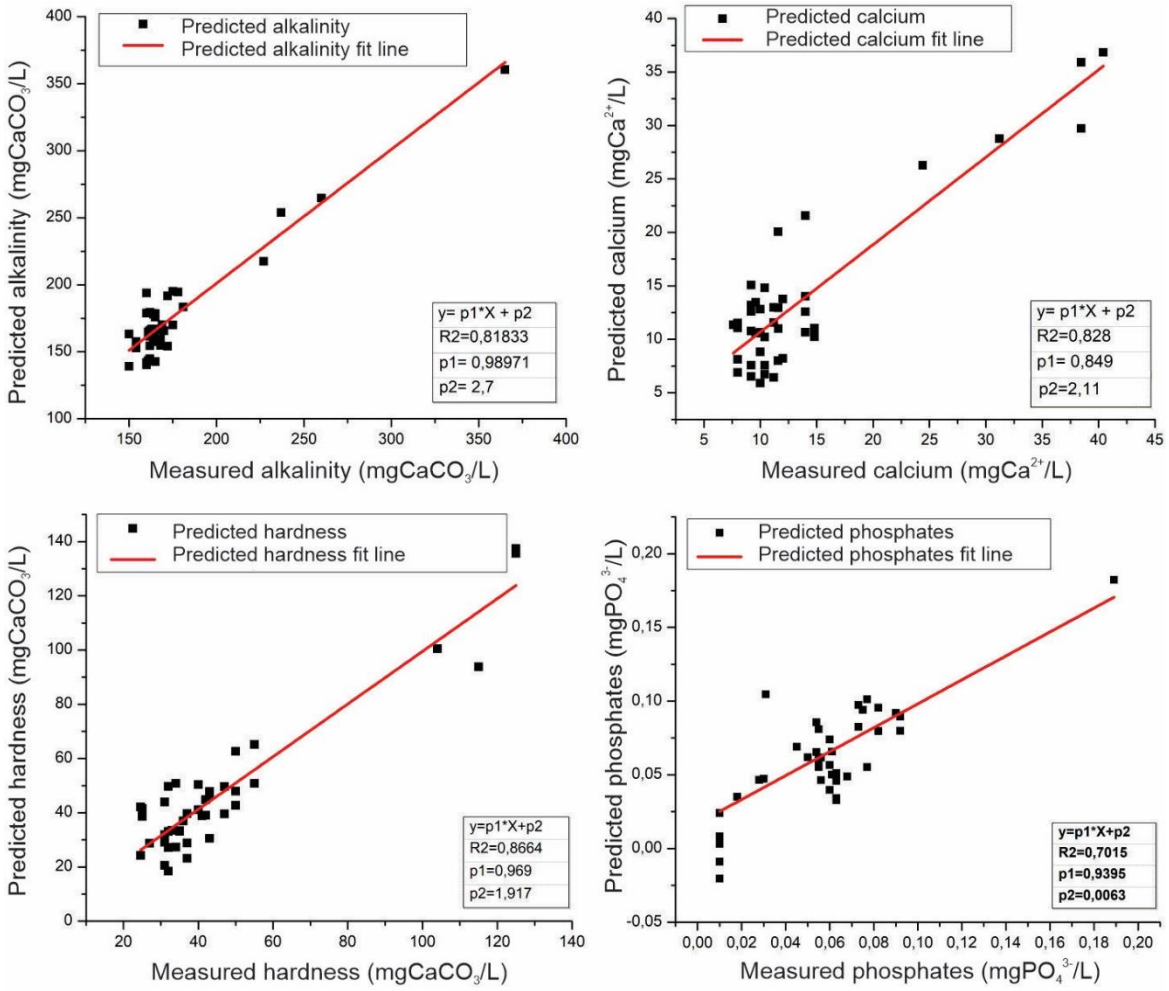

Figure 7. Regression models of physicochemical parameters (alkalinity, calcium, hardness, and phosphates) generated by smart electronic tongue and traditional methods of chemical analysis.

It could be seen that the R2 (coefficient of determination) reached values of 0.701 in the case of phosphate, 0.818 for alkalinity, 0.828 and 0.866 for calcium and hardness respectively. Therefore, it can be considered that the smart electronic tongue presented ability to predict the concentration of these substances.

In Figure 8 the graphs obtained for the physicochemical parameters of residual chlorine, chlorides, magnesium, and sulphates are presented. In this case, a linear correlation can be observed with R2 values of 0.315 for residual chlorine, 0.70 for chlorides, 0.788 for sulphate content, and 0.825 for magnesium content. The R2 values obtained in the case of residual chlorine show low correlation, which may be due to the fact that residual chlorine is a not stable parameter.

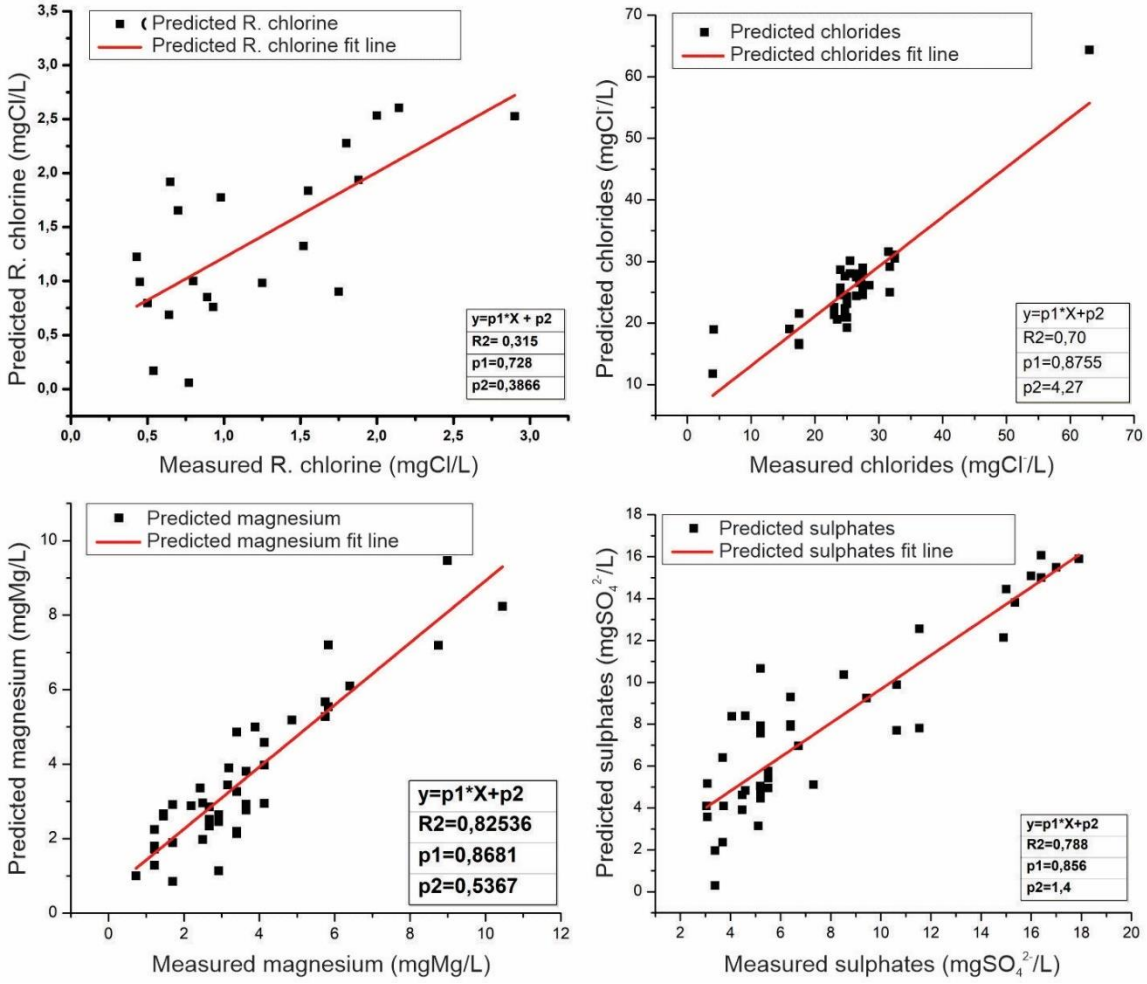

Figure 8. Regression models of physicochemical parameters (residual chlorine, chlorides, magnesium, and sulphates) generated by smart electronic tongue and traditional methods of chemical analysis.

The instability of the residual chlorine in the water can be caused by the volatility of the chlorine, which is highly affected by light and high temperatures and although the samples were refrigerated in the sampling process, the city of Sincelejo is a city withing tropical area that registers an annual average temperature of 27 °C, which can affect both the traditional chemical analyzes carried out in the reference laboratory, as well as the measurements carried out with the smart electronic tongue.

As mentioned above, studies have been reported on the use of electronic tongues for water analysis in which the correlation coefficients are lower than those obtained in this work. However, the strict comparison of the results obtained becomes inaccurate because the analytes (analytical parameters), sample types and nature of the in situ analytical procedure on which this work focuses are different from those reported by other authors (Gutiérrez-Capitán et al., 2019; Carbó et al., 2018).

4 Conclusions

The monitoring of the quality of drinking water through devices capable of providing information quickly, at low cost, and that allow measurements to be carried out in situ, can help improve the quality of life and health in remote populations. This work evaluated the application of a portable smart electronic tongue, made with a PPy sensor array, a multipotentiostat controlled by a smartphone as a drinking water monitoring device. The results of the study allowed to conclude that the voltammetric signals registered by the sensor array of the smart electronic tongue in samples of drinking water showed cross sensitivity, that is to say, each sensor in the array registered a different signal against one drinking water sample, also the signals of the recorded drinking water samples were different from each other, constituting this in a pattern or "fingerprint" of each analyzed sample. Each measurement took about 4 minutes to carried out, which represents a reduced time when compared with the traditional methods of chemical analysis used in the physicochemical characterizations of water samples.

This behavior allowed, through the application of artificial neural networks for principal components analysis, to discriminate between drinking water samples, a fact that reflects a good discrimination capacity of the smart electronic tongue. The results obtained with the analysis of the 22 samples and their replicas, showed discrimination capacity of the smart electronic tongue, with reproducible discrimination results.

Also, it could be shown that the smart electronic tongue provided quantitative information of some of the physicochemical parameters in the evaluation of the quality of drinking water. For this, the data were treated using regression models, with the aim of extracting quantitative information from the signals. Coefficient of determination values higher than 0.70 were established, which evidenced the capacity of smart electronic tongue to provide information on substances of analytical interest that determine the quality of drinking water.

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