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Microplastic Detection in Water Using a Sensor Network, An Electronic Tongue and Spectroscopy Impedance

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Abstract: *Plastic is a material widely used today because of its advantages; however, in some cases, its poor disposal can generate particles caused by the degradation of larger plastic products because of its interaction with the environment. This is the case, for instance, of microplastics found in rivers, where the friction of bottles and other plastic waste with its surrounding environment results in microplastic pollution. From this point of view, it is necessary the development of monitoring systems to detect these kinds of materials and understand their dynamics with the environment in which they are found. The use of this kind of system can allow the development of strategies for mitigating their possible impacts. In this sense, this work shows the preliminary development of a methodology for detecting microplastics in water for human consumption using the electrochemical technique of impedance spectroscopy. The methodology was evaluated in water samples, and 160 experiments were carried out by adding different contaminants. Results show the potential of the methodology in the detection of microplastics by using an electronic tongue that makes use of impedance spectroscopy and principal component analysis.*

Keywords: *Impedance Spectroscopy, Water, Microplastics, PCA, and Water Monitoring.*

1. Introduction

Human interaction has generated impacts on the environment. Some include deforestation, pollution, and movement or changes in the ecosystems. However, humans depend on their physical environment and the resources that they can provide. In this sense, regulations and the monitoring of the ecosystems and the impact of human activity are needed to avoid more negative effects and generate warnings to act before producing irreversible damage. One example of this problem is the pollution by plastics and specifically by microplastics which results in small particles that are present in water and all the elements of the ecosystems.

Microplastics in the water represent one of the great environmental challenges facing humanity; this is due to the possible impacts on people's health[1] and ecosystems [2]. These materials have been detected in bodies of water used for human consumption [3], and in some frequently consumed foods such as salt, [4], their origin is due to fragmentation caused by interaction with their surrounding environment, such as solar radiation and friction with water, rocks and organic material[5] as well as its implementation in the cosmetic, [6] and textile[7] industries. Some techniques that have achieved the best results in the detection, such as Raman spectroscopy [8] or fluorescence microscopy[9] have a high financial and technical cost. One alternative to these kinds of techniques is impedance spectroscopy which can be implemented in data acquisition systems to inspect using sensors and can provide information in a short time without the need for more sophisticated techniques or very specialized equipment. As a contribution to the detection of microplastics, this work introduces a microplastic detection methodology that considers a pattern recognition point of view.

This paper is organized as follows. First, this introduction is in section 1, after a brief theoretical background about some concepts considered in the methodology are introduced. Section 3 describes the methodology for the detection of microplastics. Section 4 presents the experimental setup and section 5 the experimental results. Finally, conclusions are included in Section 6

2. Theoretical Background

Although there are different techniques for the detection of microplastic[10] most of them require specialized equipment and personnel in laboratory conditions; likewise, the methods to quantify these particles are very long and require significant technical capital for their collection and analysis.

Microplastics have been detected in water bodies at different studies around the world since the 1970s[11] although more recent investigations date them back to the 1980s, with an important turning point in 1998 when high levels began to be detected of microplastic in the northern South China Sea [12]. To understand some of the concepts aborded in this work, this section introduces some of them. However, it is recommended that the readers review the references for a better explanation of the elements and concepts in the section.

2.1. Electronic Tongues

Electronic tongues are systems that allow the electroanalysis of substances. These are based on a low-selectivity sensor network, a data acquisition system such as a potentiostat, and a data analysis and pattern recognition unit. More details can be found in[13]. Fig. 1 shows a general overview of an electronic tongue system with some of the components used in this work.

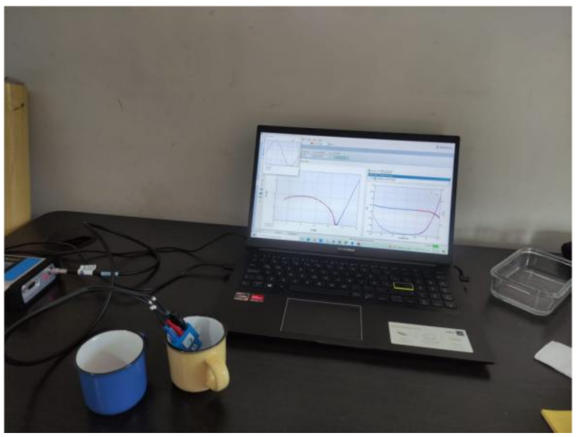

Fig. 1 Electronic tongue system

2.2. Potentiostat

The potentiostat is an electronic curve tracer device designed for electrochemical analysis. In the case of this work[14] potentiostat was used. This device can be used as a Potentiostat, Galvanostat, and Impedance Analyzer. More information can be found directly on the webpage of the company Palmsens.

2.3. Multiplexer MUX8R2

Manuscript Due to the potentiostat used in this work only having one channel, a multiplexer is required for using multiple sensors. In the case of this work, a multiplexer MUX8R2[1] is used to allow the use of up to 8 sensors. This device is also from Palmsens and for this reason, the inspection of the sensors can be performed in an automatic way. More details can be found directly in the web page of Palmsens.

2.4. Spectroscopy Impedance

Impedance spectroscopy is an electrochemical analysis technique that characterizes the properties of electrochemical systems and materials. This technique allows for the analysis of the electrical properties of a material by using electrical signals at different frequencies.

2.5. Principal Component Analysis

Principal Component Analysis also known as PCA is a technique used for multivariate analysis, data reduction, and feature extraction. PCA allows obtaining of a reduced version of a large dataset by evaluating the variability of the variables and by defining a new representation given by the principal components. This is widely used in multiple applications including the analysis of electronic tongues [13], and structural health monitoring [15], among others. Exists also a non-linear version of this technique that has also been explored in some works, for example in classification tasks on electronic noses [16].

2.6. Damage Indices

There are multiple indices that have been developed to evaluate the variables in the PCA modeling. Two of them are the Q- statistic also known as Square prediction error (SPE) and Hotelling´s T 2 -statistic. The first considers the residual data matrix obtained by the PCA modeling to represent the variability of the projected data within the residual subspace and the second, considers the score matrix obtained in PCA to determine the variability of the projected data in relation to each component. More information about each index can be found in [15].

3. Microplastic Detection Methodology

The detection of microplastics in this work considers a pattern recognition methodology with six stages, as in Fig. 2

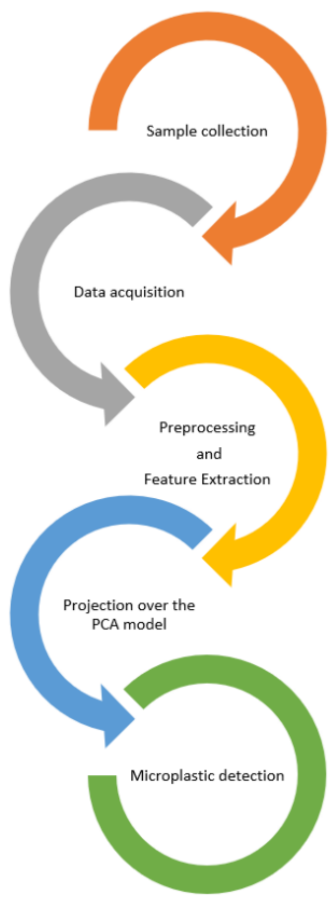

Fig. 2 Microplastic detection methodology

The methodology starts with the sample collection. To do that, different scenarios are considered to determine the differences in the signals from the water under different conditions. This is a sample of water tap for the baseline and water with different sizes of microplastics. After that, data acquisition is performed to capture the signals from all the sensors which are organized for the analysis and the PCA modeling.

Signals from water with and without microplastics were considered; more details of these experiments will be described in the next section. Multivariate data analysis and modeling is the next step of the methodology; it consists of the use of pre-processing techniques and modeling using principal component analysis. This model is the pattern for the detection process, and components are considered for the feature extraction process.

After that, the methodology considers data projection of water data with different conditions; this means water with and without microplastics. Finally, the detection can be obtained by analysing the score plot and detection indices.

4. Experimental Setup

Figure 3 shows the experimental setup for acquiring the data validating the methodology. As is shown, an arrangement of eight SPE sensors were used, these are made of 5 different working electrode materials. All the sensors are directly in contact with the water as in Fig. 3. These are described according to TABLE .

TABLE I: Information about the sensors used by the electronic tongue.

ID	Reference	Working Electrode Material
Sensor1	AC1.W1.R1 DW=2	Gold
Sensor2	AC1.W2.R2 DW=2	Platinum
Sensor3	AC1.W3.R2 DW=2	Silver
Sensor4	AC1.W4.R2 DW=2	Graphite
Sensor5	AC1.W2.R2 DW=2	Platinum
Sensor6	AC1.WS.R2 DW=2	Gold-Platinum Alloy
Sensor7	AC1.W3.R2 DW=2	Silver
Sensor8	AC1.W2.R2 DW=2	Platinum

These are connected to the potentiostat through a MUX8R2 multiplexor. As a potentiostat, the PalmSens4 was used in combination with the multiplexer to handle the sensors.

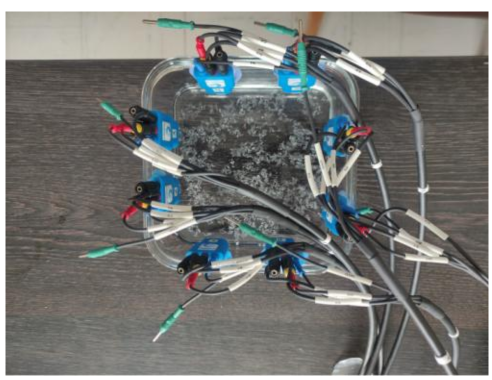

Fig. 3 Sensor configuration

In the collection of samples, different types of materials were chosen, between organic and inorganic, with the aim of artificially adding noise to the water and measuring the change in charge transfer that the new material caused in the water. A previously analyzed water sample was made by adding a specific portion of PET plastic, then inert organic, inorganic, and living organic test materials were added. For each type of material added, each of the 8 sensors takes twenty experiments, this means that 162 measurements were obtained in each experiment. *TABLE 2* shows the performed experiments.

TABLE II: Experiments

Number of experiment	Material	Characteristics
1	Water	tap water
2	water with microplastic	0.05 mm-2 mm
3	water with microplastic	0.5 mm-1.5 mm
4	water with microplastic	1 mm-2 mm
5	water with microplastic	
6	Mixture 1	
7	Mixture 2	
8	Mixture 3	

PET (polyethylene terephthalate) was used for the experiments. It was fragmented into pieces that varied between 200µm, and 2000µm. Inert organic samples and nonorganic samples are used for the mixtures. These are composed of leaves, wood, rocks, and sediments. Their sizes are close to the microplastics considered; this is 200µm and 2000µm. Live organic samples, which are composed of ikura fish eggs. The following figure shows the portions of the materials used. The characteristics of each of the mixtures used are summarized below.

i. The first mixture corresponds to water plus microplastic. In this experiment, the measurement was made with four sizes of plastic.
ii. In the second mix, inert organic matter was added and it was achieved for each of the plastic sizes.
iii. In mixture three, living organic matter was added, for which ikura fish eggs were used. This is for each of the plastic sizes.

The experiments carried out measured the impedance of microplastic in tap water at frequencies between 1 and 5 Hertz at voltages between 50 and 200 millivolts, to characterize the microparticles. Fig. 4 shows the parametrization used in the potentiostat for the data acquisition.

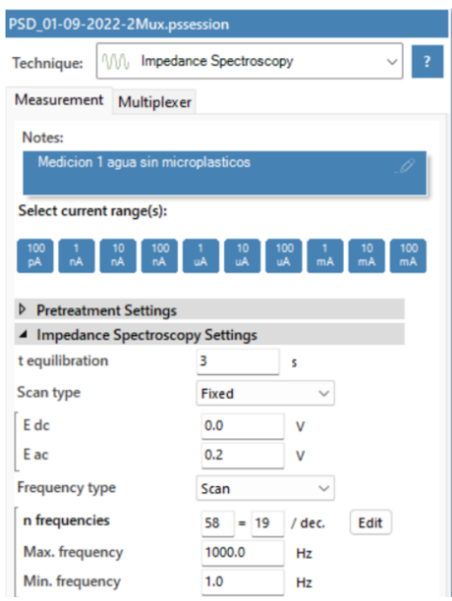

Fig. 4 Parametrization used in the Potentiostat

All acquired signals were normalized and group scaling was applied to provide a baseline for the comparison. This is necessary to provide the same scale to all the signals and give the same opportunity to contribute to the PCA modeling.

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5. Experimental Results

The results of the acquired data are shown in Figures 5 and 6. These Figures show the Nyquist and Bode diagrams of the measurements when only water without polluting materials is considered. The colors in the Figure represent each one of the sensors used for the inspection. As it is possible to observe, each sensor provides a different way to interact with the water. This is because different sensors were used and each of them provides different signals from the interaction with the substance.

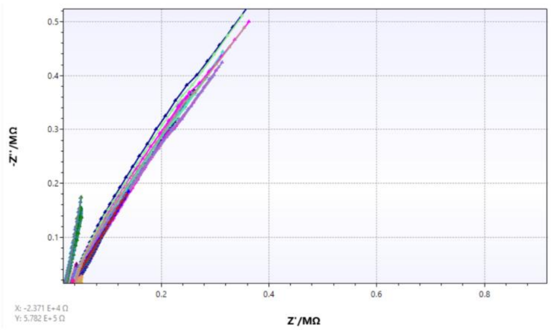

Fig. 5 Representation of charge transfer in the Nyquist

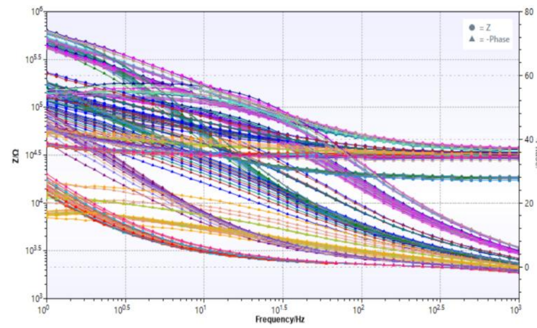

Fig. 6 Representation of charge transfer in the Bode plot

Fig. 7 and 8 show the results of the use of PCA by showing the first two components and zoom of this plot, respectively. As it is possible to observe, different groups can be identified in the Figure. The green color represents the results for water, as it is possible to observe this is mixed with some of the experiments for the other experimental conditions. However, some of these conditions are clearly separated. This result is because two components are not enough to represent the variability of the data.

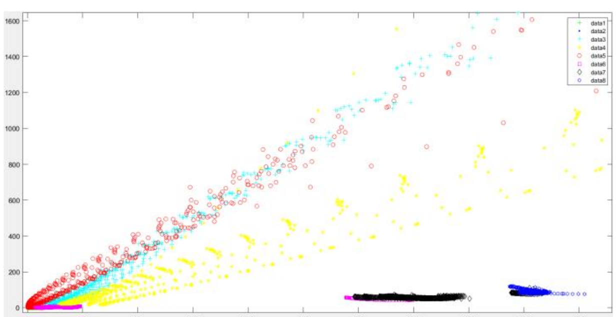

Fig. 7 PCA plot with the first two components

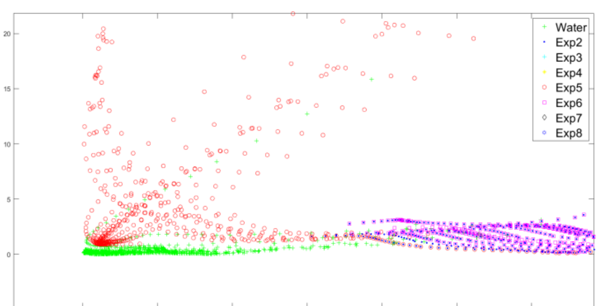

Fig. 8 Fig. 8. Zoom of the PCA plot with the first two components

Figure 9 shows the result when the indices T^2 and Q are considered. As it is shown a better separation is found whit the use of these indices. In this plot, water is depicted with a green color, and the rest of the experiments are represented by a red color.

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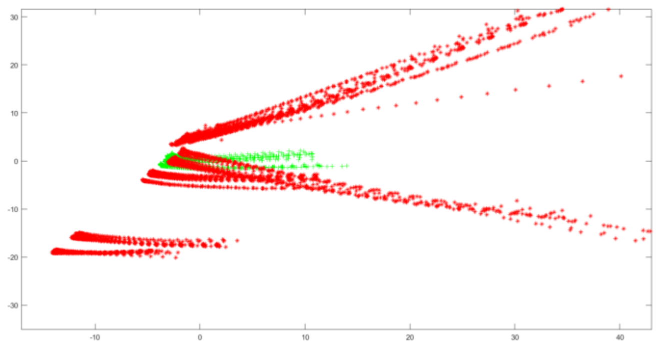

Fig. 9 T^2 vs Q plot

6. Conclusions

Results show that it is possible to detect the presence of microplastics and other elements in the water by using an electronic tongue with the methodology introduced in this work. In this case, the use of multiple sensors allows for covering the sample and combining the data analyzed by the methodology from different points of view. The use of PCA allowed model building and data reduction. Results show that, in this case, only two components can be used to show the differences between the data from the different experiments, however, not all the experiments show differences in the plot because of the use of only two components for the representation. To improve the results, the use of detection indices allows to separate in a different way the data from all the experiments. In future work, more pre-processing methods need to be explored to improve the results and show the difference between the experiments in the used plots. Classification approaches can be also explored for this aim.

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