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A New Kind of Chemical Nanosensors for Discrimination of Espresso Coffee

Giuseppe Greco [1], Estefanía Núñez Carmona [2,*], Giorgio Sberveglieri [1,3], Dario Genzardi [1] and Veronica Sberveglieri [1,2]

[1] Nano Sensor Systems S.r.l. (NASYS), Spin-Off University of Brescia, Brescia, Via Camillo Brozzoni, 9, 25125 Brescia, Italy; giuseppe.greco@nasys.it (G.G.); giorgio.sberveglieri@nasys.it (G.S.); dario.genzardi@nasys.it (D.G.); veronica.sberveglieri@ibbr.cnr.it (V.S.)
[2] National Research Council, Institute of Bioscience and Bioresources (CNR-IBBR), Via J.F. Kennedy, 17/i, 42124 Reggio Emilia, Italy
[3] Department of Information Engineering, University of Brescia, 25123 Brescia, Italy
* Correspondence: estefania.nunezcarmona@ibbr.cnr.it

Abstract: There are different methods to extract and brew coffee, therefore, coffee processing is an important factor and should be studied in detail. Herein, coffee was brewed by means of a new espresso professional coffee machine, using coffee powder or portioned coffee (capsule). Four different kinds of coffees (Biologico, Dolce, Deciso, Guatemala) were investigated with and without capsules and the goal was to classify the volatiloma of each one by Small Sensor System (S3). The response of the semiconductor metal oxide sensors (MOX) of S3 where recorded, for all 288 replicates and after normalization $\Delta R/R0$ was extracted as a feature. PCA analysis was used to compare and differentiate the same kind of coffee sample with and without a capsule. It could be concluded that the coffee capsules affect the quality, changing on the flavor profile of espresso coffee when extracted different methods confirming the use of s3 device as a rapid and user-friendly tool in the food quality control chain.

Keywords: gas sensors; coffee capsule; Volatilome; Small Sensor Systems

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1. Introduction

Coffee is one of the most consumed beverages in the world. Moreover, it is the second one after water and its consumption constantly increases [1]. Coffee brewing methods can be changed depending on the geographic, cultural and social environment as well as individual preferences [2]. Espresso coffee (EC) is the traditional Italian extraction method that requires more control of many parameters, such as temperature, pressure, grind size and knowledge. Bartender training is fundamental. He must adopt a series of measures in the extraction phase and perform checks and interventions of ordinary management and periodic maintenance of the equipment. The quality of coffee is globally appreciated. However, it is difficult to manage. Big companies have developed portioned coffee segments to overcome the aforementioned issues. The application of this new strategy to extract EC is related to coffee capsules. The coffee capsule is a single-dose coffee product, which is roasted, ground and perfectly portioned in a small aluminum or plastic container (generally in cylindrical form). Furthermore, coffee capsules exhibit an extended shelf life of the product compared to the not portioned ones. This is possible owing to the capsule packaging mainly using polypropylene. The coffee aroma is composed of over 800 volatile substances that characterize the mixture and its intensity can be changed depending on the extraction method [3]. The well-known and the most abundant classes of chemical compounds are aldehydes, phenols, ketones, pyridines and furans. These compounds can be easily extracted by several coffee extraction methods combining the pressure and the temperature of processes. Coffee supply chain have a lot of steps, until reaching the final one of extraction. Coffee brewing is a solid-liquid extraction process and the extraction kinetics is heavily influenced by the aforementioned parameters regarding the different chemical compounds present in roasted coffee that will develop subsequently the volatile fraction. The common consumer is careful to the most important coffee characteristics. One of these is the coffee because it is considered to be one of the fundamental parameters in order to categorize its overall quality [2]. The right processing of the raw material (green coffee beans) is crucial to obtain a high-quality product. There are a lot of factors that could influence coffee final properties. Specifically, the main ones that surely have an influence on coffee extraction and its sensor profile are six [4]. These are: plant varieties, growing region/conditions, processing methods (from coffee cherries to green coffee bean), roasting levels, grinding size and brewing methods [5]. The conditions and the parameters of the roasting process are so important in order to let the precursors developed in the desired way. The green beans contain them, and their influence can change regarding the changeable aforementioned conditions [6]. Although the aroma and flavors are characterized by the origin of green coffee, the roasting process affects the production of volatile compounds resulting in differences in the complexity of coffee aroma. In general, roasting ruptures the cell structure of green coffee beans exposing it to heat that drives out the moisture and releases the aromatic compounds that have been chemically bound in the beans [4].

Several classes of molecules are volatilized from the bean such as: carbon dioxide, aldehydes, ketones, ethers, acetic acid, methanol, oils and glycerol. The volatilization of different volatile compounds can change with regard to different temperatures, and as pyrolysis continues, the volatile fraction of the product continues to develop until the process ends [7]. Hence, the extraction process radically changes the aroma fingerprint. Regarding to this, new types of chemical gas sensors have recently been studied in order to classify food samples through their volatiloma, and, consequently, to have a better knowledge of the volatile fraction.

The necessity of developing accurate analytical methods has prompted the present study. The other techniques, such as Gas-Chromatography and Mass Spectrometry (GC-MS), are useful but they need intense training, they are time consuming and generally have higher costs. On the contrary, the use of an array of metal oxide (MOX) gas sensors allows classification of different kinds of samples with high sensitivity, quick responses and low costs. MOX sensors are conductometer sensors, so called thanks to their ability to transduce a chemical signal in an electrical resistance signal [8]. Interactions happen between volatile compounds and sensing material. MOX gas sensors are non-specific sensors; hence, they can be suited for different classes of compounds with different sensibility.

Furthermore, this is the main reason that sensor arrays are used in most applications. Overall, the aims of this work are:

- Discrimination of four different typologies of "Molinari" coffee capsules (Biologico, Deciso, Dolce, Guatemala) using a new innovative technology based on semiconductor metal oxide sensors (MOX).
- To underline differences between coffee extracted with and without capsule packaging.

2. Materials and Methods

2.1. Sample Preparation

This study is based on the analysis of four types of coffee capsules: "Biologico", "Deciso", "Dolce" and "Guatemala". A professional machine "Spaziale S2" (LA SPAZIALE S.p.A., Casalecchio di Reno, BO, Italia) was used for the coffee extraction. The coffee extraction was performed in 18 L containers of "Rocca Galgana" (Citerna di Fornovo di Taro, Parma, Italy) mineral water to overcome the inhomogeneities due to the water [1,7].

This study was carried out considering two approaches: (i) coffee was prepared by entering the coffee capsule in the correspondent extraction tool; (ii) coffee was pulled out from each capsule and extracted without capsule packaging. The quantity of coffee extracted was 62.5 mL (espresso shot coffee [9]) and it was collected using a beaker. Then, 20 mL chromatographic vials were filled with 1.2 mL of coffee. 36 vials for each sample were made obtaining 144 vials (36 Biologico, 36 Deciso, 36 Dolce, 36 Guatemala) of coffee extracted with capsule and 144 vials (36 biologico, 36 deciso, 36 dolce, 36 guatemala) of coffee extracted without capsule (Table 1).

Table 1. Samples Analyzed.

Sample	With Capsule	Without Capsule
Biologico	36	36
Deciso	36	36
Dolce	36	36
Guatemala	36	36
Total	**144**	**144**

The vials containing the sample were sealed with an aluminum cap and a of poly-tetrafluoroethylene (PTFE) and silicone septa. In order to heat samples and achieve the equilibrium of the volatile compounds between the headspace [10] and the liquid phase and to reduce variables, the vials were initially incubated at 30 °C for 10 min. Afterwards, the extraction phase was performed, where the autosampler syringe is exposed in the head-space of the vial for 1 min to allow the absorption of volatile compounds.

2.2. Small Sensor System (S3)

2.2.1. Analysis Conditions

The S3 device, acronym for Small Sensor Systems, is constructed by Nano Sensor Systems S.r.l. (Reggio Emilia, Italy, www.nasys.it (accessed on 18 February 2022)) This device has been already used with remarkable success in other previous studies in the field of quality control and food technology [7]. S3 is connected to the autosampler HT2010H (HTA s.r.l., Brescia, Italy) equipped with a carousel with 42 positions to accommodate the vials for sampling. The system allows to collect and analyze the data acquired in the cloud making S3 an IoT device for the management and control of signals [8]. S3 is composed of three essential parts [7]:

1. The sensor steel chamber which contains the six MOX sensors. This allows the sensor to be separated from the environment, except for an inlet and an outlet path for the passage of volatile compounds. Other types of sensors are placed in order to control several parameters during the analysis. These are the temperature, humidity, and flow in the chamber.
2. The fluid dynamic circuit is composed of a pump (Knf, model: NMP05B), polyurethane pipes, a solenoid valve and a metal cylinder, which contains activated carbon. The activated carbon is used for filtering any type of odors present in the environment that can alter the final response. The solenoid valve, positioned at the chamber inlet to control the pump flow with a maximum of 250 sccm.
3. The electronic board elaborates the sensor responses through the detection of electrical resistance. In addition, it controls the operating temperature of sensors, which is an important parameter for the detection of volatile compounds. Finally, the system is able to send the data to the Web App dedicated to the S3 device through an internet connection.

The sensor response is based on the change of its resistance over time caused by interaction with different kinds of volatile compounds or surrounding environment. The reactions between the oxygen species adsorbed on the surface of the sensor and the target molecules lead to the variation in the concentration of charge carriers in the sensing material affecting its electrical conductance [9].

2.2.2. S3 Data Processing

Processing of the S3 sensor signals was performed using MATLAB® R2019b software (MathWorks, Natick, MA, USA) in order to extract the features of the sensor response. Sensors' responses in terms of resistance (Ω) were normalized to the first value of the acquisition (R0). For all of the sensors, the difference between the first value and the minimum value during the analysis time was calculated. Hence, the $\Delta R/R0$ parameter was calculated and has been use as a feature for all the sensors response to the 36 replicates of each sample. The standard deviation of the $\Delta R/R0$ parameter was calculated for each group of sample measurement prior to proceed with PCA analysis revealing a maximum uncertainty of the 10%. Once the data matrix was calculated, principal component analysis (PCA) was applied to the data in order to verify the variation of the VOCs in the different samples [10].

This technique consists of clustering the sample variables through linear combinations that describe the link between one sample to the others. This results in the main components (PC), which are far fewer than the original variables. These new variables are structured in such a way as to be orthogonal to each other (not correlated). Moreover, most of the variability of the samples is present in the first main components. As a consequence PC1 shows the largest variation. Next, PC2, which represents the second largest variation. This can continue until all the variables are explained. These conditions allow the detection of any groupings [11]. They are also known as clusters that represent samples united by characteristics. PCA is not a classification technique, but a technique that may provide the distribution of the samples within the main components considered in the hyperplane.

3. Results and Discussion

3.1. Sensor Response Regarding the Discrimination of Molinari Coffee Capsules

The response curve of a sensor is represented in Figure 1, where on the x-axis is indicated the time (s), while in the y-axis the normalized resistance is reported. The use of normalized parameters allows us to obtain data without dimensions, hence they are a-dimensional variables, where the variability is equal to 1 and the average is 0. The choice to use normalized variables is advantageous in the analysis of samples that have different units of measurement or size, which would prevent accurate analysis of samples.

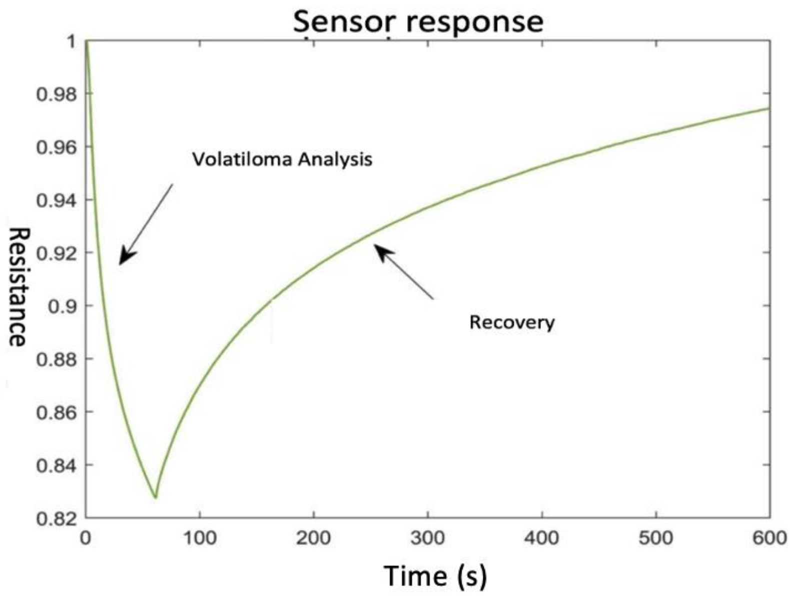

Figure 1. Schemes of the typical sensor response.

As can be seen in Figure 1, the signal starts from a resistance value of 1, which is reduced. The studying of samples using the sensing system is based on two phases: the first one is the analysis of the coffee volatiloma, where the volatile compounds come into contact with the sensors. This interaction leads to a decrease in the electrical resistance of sensing material. The second stage is called recovery where the base line is restored.

In this case, filtered ambient air is introduced to the chamber, the adsorbed gaseous compounds are desorbed from the surface of sensors and their resistances are recovered to their steady stage values. The first phase lasted 1 min followed by the recovery to the base-line resistance, which required 9 min. Hence the total analysis time is 10 min. Small Sensor System (S3) analysis was able to discriminate the samples, and describe the volatile fingerprint. Figure 2 illustrates a complete separation of the Biologico coffee capsule from the other ones on the hyperplane. This means that it is the only one that doesn't have any match, while Dolce Deciso and Guatemala coffee capsules are on the same side. In particular, Dolce and Deciso are mainly overlapped. Guatemala's are well separated among PC1 and PC2. Therefore, the sensors must have perceived some differences, although not as much as in Biologico's case.

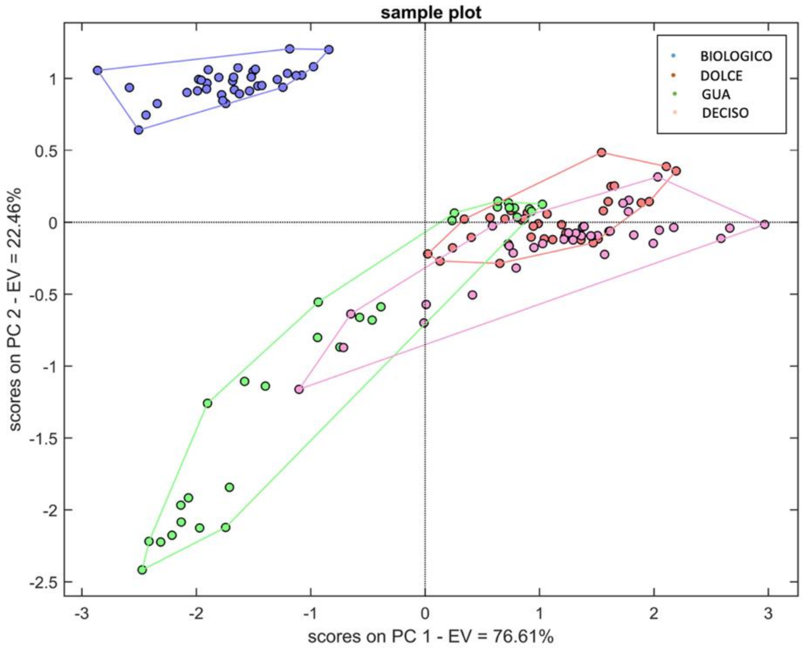

Figure 2. PCA Molinari coffee capsules.

3.2. Coffee with and without Capsule Packaging Comparison

The most important parameter about coffee is its aroma. It is the first factor which take consumer attention and influence his appreciation and selection; hence, it is considered an important quality trait. Espresso quality depends on several parameters which must be gauged in order to obtain the proper ratio of desirable components for the most desirable product [12].

In particular the ratio of water to coffee, time and grind size impact the brewing process. In addition, Espresso Coffee is a product characterized by an intense viscosity caused by high concentration of Total Dissolved Solids (TDS) [13].

The latter is usually a highly desired quality of the espresso brew because it gives an appreciated fullness to the final product. Beyond that, TDS is a non-specific analysis technique generally used to do a quantitative analysis regarding the extraction performance, also called extraction yield. Moreover, the most common methods to assess aroma are based on expensive equipment or human senses through sensory evaluation, which is time-consuming and requires highly trained assessors to avoid subjectivity [14].

On the contrary, the S3 analysis could easily find volatiloma differences between coffee extracted with and without capsule packaging. Figures 3–6 reports Principal Component Analysis (PCA) performed to process the obtained data. It is possible to say overall that PCA showed the EV never under 78%. This represents an optimum result since at least 78% of the total variability of the samples was enclosed between the hyperplane (enclosed between the first two principal components).

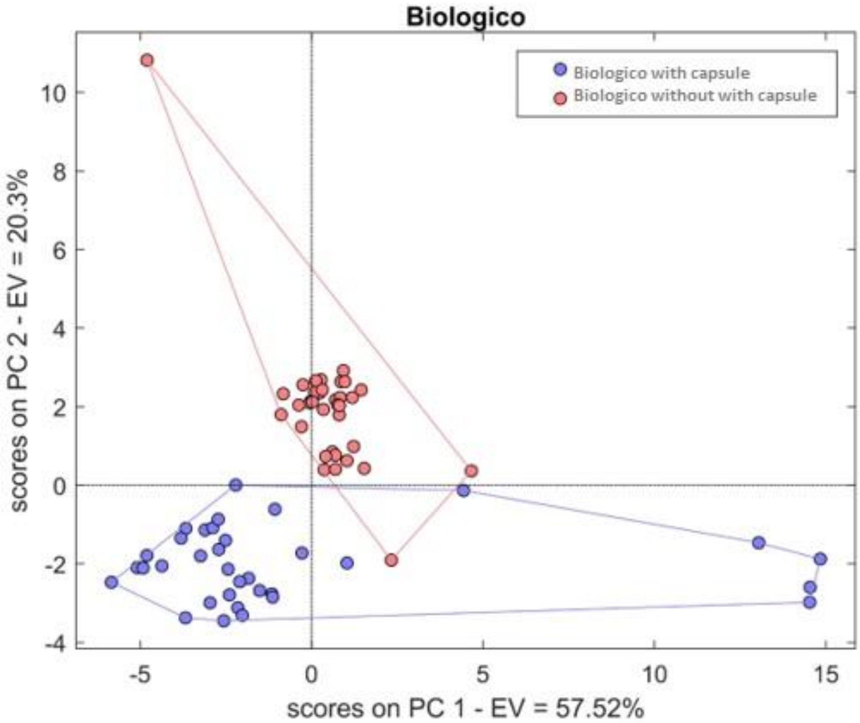

Figure 3. S3 comparison Biologico with and without capsule.

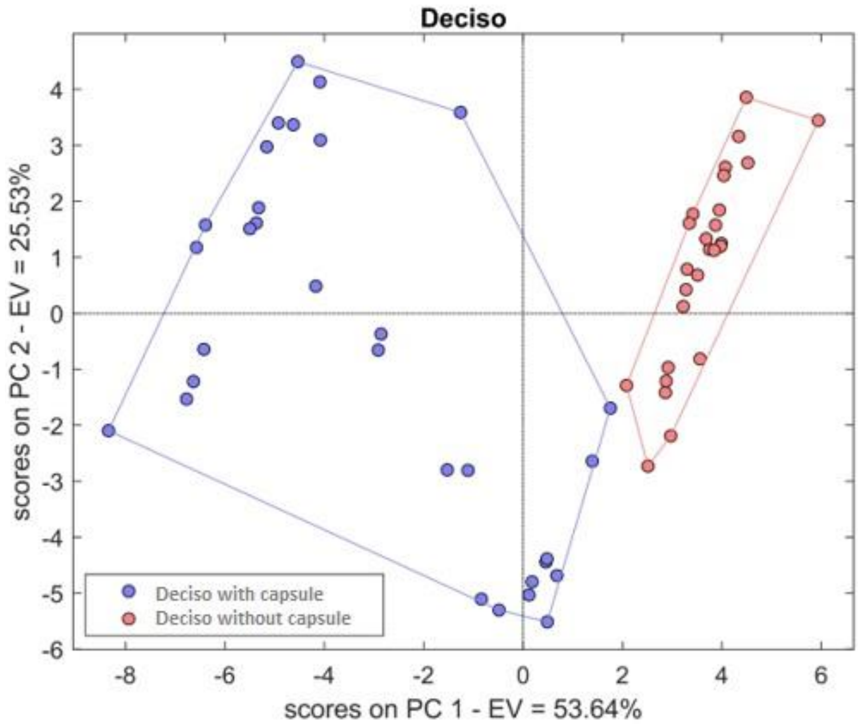

Figure 4. S3 comparison Deciso with and without capsule.

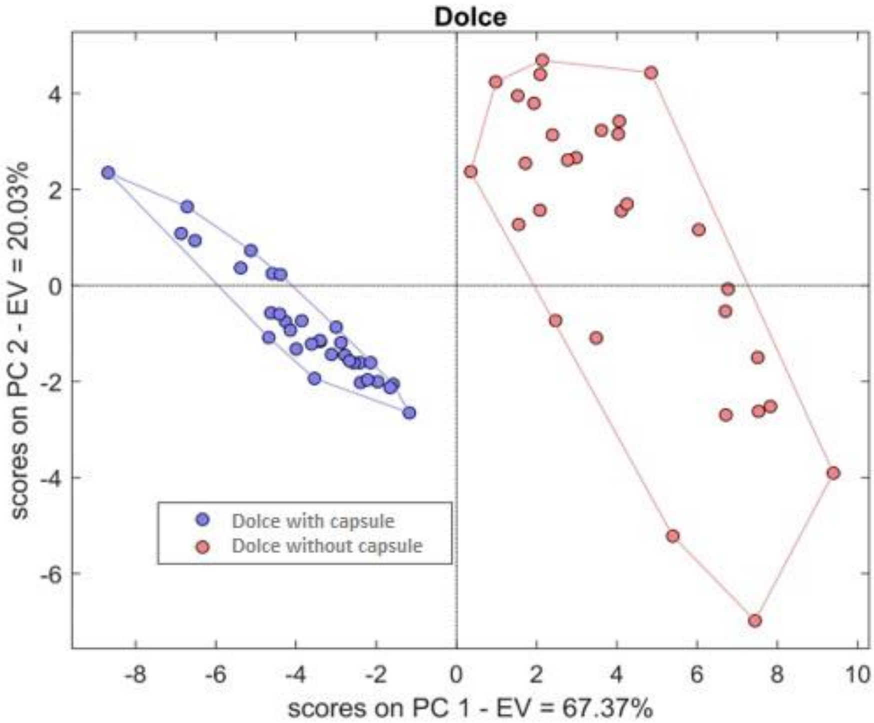

Figure 5. S3 comparison Dolce with and without capsule.

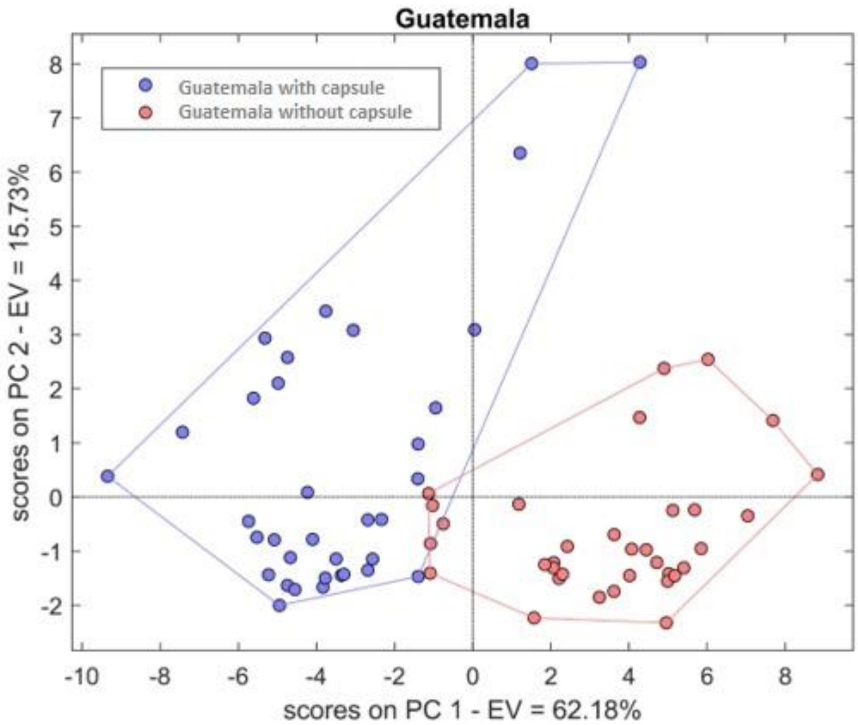

Figure 6. S3 comparison Guatemala with and without capsule.

PC1 is always the component with a larger load, reaching the maximum value of 67% in the case of the Deciso samples. The six single sensors perfectly discriminated the samples (one for each type) where with and without capsule packaging coffees were compared. The six single sensors perfectly discriminated the samples (one for each type) where with and without capsule packaging coffees were compared. As a matter of fact, Ferini et al. [15] made some investigations about the differences of coffee product obtained with different techniques. One of them was to check possible differences between espresso and coffee capsules through a human sensorial test. At this point the consumers, even if coffee capsules are intellectually judged as similar to espresso coffee, noticed differences when tasting the beverages.

This is probably due to the compression of the coffee capsule, which improves the extraction of the volatile fraction. Thus, the coffee volatile fraction is far from simple, considering the huge number of different chemical compounds that must be considered. As a matter of fact, over a thousand different chemical entities have been detected in coffee beans such as aldehydes, phenols, ketones, pyridines and furans. A significant number of these compounds will be extracted during brewing. It's a comparatively small subset of chemicals that affect the aroma. Among all the variables, there are two main factors which must be considered to investigate the effect of compounds' aroma: the concentration of the compound and the compound's odor threshold, or the minimum concentration at which it is possible to detect its smell [16]. The result of these two factors gives the compounds 'odor activity value' (OAV) [17], responsible parameter of the overall aroma.

Several chemicals are contributors and responsible about the evolution of the coffee volatile fraction. The most significant chemicals which enrich the volatiloma coffee product are the following:

- Aldehyde compounds, which generally add a fruity green aroma
- Furans, which contribute with caramel-like odors
- Pyrazines, which have an earthy scent.
- Guaiacol and related phenolic compounds offer smoky, spicy tones, and pyrroles.
- Alkaloids, as caffeine or trigonelline. Caffeine content is highly variable in a range between 0.64 mg/mL and 4.89 mg/mL [18] considering different parameters (pressure and temperature) values.
- Chlorogenic acids (CGAs), the most important are 3-, 4- and 5-CQA [19].

Over the last 40 years, the term "electronic nose" (EN) has defined a device equipped with an array of gas sensors capable of providing a response as a function of a stimulus provided by volatile chemical compounds (VOCs). Numerous studies have started from this idea, which have led to significant improvements and advantages, especially useful for providing a device capable of monitoring situations and applications in real-time., especially in the food and beverages industry [20], having regard to the gas sensors capabilities of providing a response as a function of a stimulus provided by volatile chemical compounds. Known as also the aroma sensor, mechanical nose, multi-sensor array, artificial nose, odor sensing system, or electronic olfactometry, the initial goal of EN technology was to simulate the mammalian nose to obtain a fast response regarding the characteristics of the analyte, high sensitivity for odours and high discrimination between them [21]. As a matter of fact, EN sensing is mainly used for the classification and characterization of the overall aroma pattern of EC samples, so it is generally coupled with other analytical techniques such as GC [22], which can qualitatively and quantitatively characterize EC odor-active compounds [23].

4. Conclusions

This study is based on the analysis of 4 different types of coffee samples (Biologico, Deciso, Dolce, Guatemala). The analyses were carried out by the Small Sensor System (S3) device, where the sensors were able to identify the differences of the biologico capsule from the other ones and to certify the differences between the samples with and without capsule packaging.

The obtained results (PCA Figures 3–6) indicate that the coffee aroma profile is incredibly influenced by the compression of the coffee pod, which improves the extraction of the volatile fraction. Indeed, the results show how the coffee obtained using the capsules has a different volatile fraction. Moreover, these ones would also provide standardized organoleptic characteristics of each coffee without the need of trained operators. This can be a huge advantage for household use but also for professional use allowing anyone easily to prepare a high quality final product. Overall, two main advantages can be highlighted:

the first one is that the coffee aroma intensity can be maintained over the time and avoid the deterioration that would occur for the coffee without capsule. The second one is that the volatile fraction of the final product of the capsule is higher than the one without the capsule packaging.

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