An-Najah National University

Faculty of Graduate Studies

Studying Olive Oils' Quality Using Sensors and Multivariate Data Analysis

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Dedication

I dedicate this thesis to my family and my friends. Thank you all for your help and support.

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I would like to express my gratitude and appreciation to all who participated in putting this thesis together.

Special thanks and appreciation goes for Prof. Hassan Abu Qaoud for his guidance, time and his valued information, who supported me all time, and helped to make this thesis critical insight and rewarding experience and in preparing me for the academic work ahead.

Special thanks and appreciation to the others who provided me with their support, encouragement, thoughts, and these special individuals are not limited.

الإقرار

أنا الموقع أدناه مقدم الرسالة التي تحمل العنوان:

Studying Olive Oils' Quality Using Sensors and Multivariate Data Analysis

اقر بان ما شملت علية هذه الرسالة إنما هو نتاج جهدي الخاص، باستثناء ما تمت الإشارة الية حيثما ورد، وان هذه الرسالة ككل، أو أي جزء منها لم يقدم لنيل أي درجة أو لقب علمي أو بحثي لدى أي مؤسسة تعليمية أو بحثية أخرى.

Declaration

The work provided in this thesis, unless otherwise referenced, is the researcher's own work, and has not been submitted elsewhere for any other degree or qualification.

Student Name: **اسم الطالب:**

Signature: **التوقيع:**

Date: **التاريخ:**

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Abstract

This study aims to investigate the possibility of applying the electronic tongue (ET) on olive oil in order to distinguish between edible olive oil and inedible olive oil, as well as assessing extra virgin olive oil, in terms of positive sensory characteristics. In addition, the ability of the electronic device to support the work of the national committee for sensory evaluation.

One sample of olives oil that's is not suitable for human consumption (lampante), and 23 samples of extra virgin olive oil were analyzed. Samples were taken from the Palestinian Standards Institution. The samples have been tested by the National Sensory Evaluation Committee. These twenty-three samples have successfully passed all sensory and chemical tests and reach the final stage of the Golden Olive Oil Competition.that organized annually by the Palestinian Standards Institution. Data analysis was done by combining the (electrochemical) ET with multivariate data analysis. This analysis is based mainly on a statistical principal component analysis (PCA) and partial least squares (PLS). In which PLS linear regression was applied to study the relation between electronic tongue signals and quality parameters. The study showed, in regards to fruity, with full cross valiation for calibration sets, R^2 = 0.946 and RMSE = 0.182. And in regards to

bitterness $R^2 = 0.81$ and RMSE = 0.23. Also, in regards to pungent $R^2 = 0.34$ and RMSE = 0.47. ET could best model fruity flavor, followed by bitterness and next pungent. The results indicate, that the performance of the ET is generally satisfactory. And this technology can be taken into account for the taste and quality of olive oil.

Chapter One

Introduction

Olive oil sector is considered the main pillar of the Palestinian agricultural sector. It plays a role in providing food security not only to the families who produce it, but also to the families that participate in its production processes. It also provides seasonal employment opportunities (Abdullah and Hussen, 2016).

Effective marketing of olive oil is considered one of the most important challenges facing olive oil producers, and this relevant agricultural commodities due to technical, health restrictions, and the protection measures exercised by the importing countries, which set international standards for accepting the entry of olive oil. Because of the increase in production of Palestinian olive oil, access to international markets requires chemical and sensory tests for olive oil (Abdullah and Hussen, 2016).

According to the physical and chemical assessments, olive oil can be classified into: Extra Virgin Olive Oil, Virgin Olive Oil, Ordinary Virgin Olive Oil and Lampante (Fares et al., 2016).

Extra virgin olive oil is the highest quality olive oil. The positive sensory flavor of extra virgin olive oil are: fruity, bitter and pungent. Negative practices such as harvesting, transporting, storing and producing the olive oil process lead to changing the flavors of olive oil and obtaining negative sensory flavors (Veloso et al., 2018).

Olive oil is described as an extra virgin oil, being the only vegetable oil that does not undergo refining, and is consumed after squeezing it and extracting it directly from the olive fruits. In contrast, rest of the vegetable oils cannot be consumed human before conducting refining operations for them, to get rid of impurities that are not suitable for human consumption olive oil is not being refined, because all the non-fat compounds present in it, are beneficial to humans, protective against diseases, and have many health benefits (Dias et al., 2016).

The olive oil is exposed to many sensory defects, which affect the quality of olive oil there are several techniques to help detect the defects of olive oil and evaluate the negative sensory characteristics, according to the International Council of Olive Oil, and it recommended the need to assess the olive oil for quality assurance (Slim et al., 2017). At present, the Palestinian Institution for Standards and Metrology (Ministry of National Economy) undertakes the sensory evaluation process through the tasting laboratory, which was established to classify olive oil (extra virgin – ordinary virgin –virgin) there is the Palestinian national team to taste olive oil, performs the physical examination in the institution's laboratory, which it is equipped according to the requirements of the International Olive Council (IOC) specification. In order for the connoisseur to correctly identify the defects and positive attributes present in the oil sample, and through his findings, the oil sample is classified (Olive Oil Lab, 2019).

Once again, the sensory evaluation is carried out under the direction of the International Olive Council, a standardized method anywhere in the world. This process of sensory evaluation allows to implement a commercial classification of extra virgin olive oil, which is of great importance for its marketing, and has a direct impact on the selling price and the price quality ratio perceived by consumers (Souayah et al., 2017).

The use of traditional methods of sensory assessment of olive oil requires a lot of time and cost, in addition to the need to train a team of examiners. These simple analytical tools are far from economical for olive oil producers (Slim et al., 2017). Therefore, researchers recently sought to apply devices that work through electrochemical in order to characterize the olive oil sensitively, and quality control during storage and export operations. It was necessary to develop an electronic device for the sensory assessment of olive oil, which is known as the electronic tongue (ET). So that the electronic tongue, as a tool helps to detect the defects of olive oil, and reduce the risk of dependence on self-assessment (Marx et al., 2017a; Souayah et al., 2017).

One of the benefits of the ET is detecting olive oil adulteration. Olive oil operations aim to achieve an additional profit, because olive oil is expensive in production and extraction processes (Dias et al., 2016). While vegetable oils and seed oils are less expensive to produce and extract than olive oil. Harzalli et al. (2018) found that the trained team members were unable to distinguish between virgin olive oil samples from those adulterated. While the ET, was able to determine the extra virgin olive oil, from the olive oil adulterated and determine the proportion of adulteration.

The electronic tongue helps to evaluate the olive table taste quality, in terms of medium density of acid, bitter taste and salty (Marx et al., 2017b). As more over, the main functions of the ET is controling process of storage of extra virgin olive oil. It can also be determined and assess the shelf life of olive oil. ET is able to verify the compliance of olive oil quality with labeling (Rodrigues et al., 2016). Also, sensory analysis is a basic standard for the formal control of extra virgin olive oil (EVOO) quality in parallel with physical and chemical quality standards (Bhnsawy et al., 2017).

In literature and studies, many work applied electronic tongue. The electronic tongue was applied for discriminate edible from non-edible olive oils, and classify extra virgin olive oil, as well as for detecting defects and shelf life. Also, electronic tongue analysis allow to rank olive oils based on their flavor (Slim et al., 2017) to differentiate Tunisian olive oil according to its quality level (such as extra virgin olive oil, virgin or lampante oil) (Burattia et al., 2018). This study was based on the verification of the use and integration of electronic tongue, electronic nose and electronic eye, in order to distinguish between edible and non-edible olive oil,and determine the shelf life of olive oil. In addition, Marx et al. (2017a) got satisfactory expectations, through the application of the electronic tongue to assess the defects of olive oil, as well as in reducing the effort and time of the trained committee to conduct the sensory evaluation. A study was carried out through the application of the electronic tongue, based on a linear discrimination analysis in order to determine the geographical origin of olive oil in Tunisia Souayah et al. (2017).

Despite the importance of previous studies and literature, in applying the electronic tongue, in order to distinguish between extra virgin olive oil and inedible oil, this study is an extension of previous studies in order to obtain a positive sensory description of extra virgin olive oil. For the three sensual flavors (fruity, bitter and pungent), this study is considered the first in Palestine in terms of using the electronic tongue to characterize olive oil sensually.

Therefore, this study aims to verify the possibility of applying the electronic tongue on olive oil, in order to distinguish between edible olive oil and inedible olive oil, as well as assessing extra virgin olive oil, in terms of positive sensory characteristics. more over, the electronic device support the work of the national committee for sensory evaluation.

1-1 Problem statements

Olive oil is very important in the world economy. Therefore it is possible that the olive oil is exposed to fraud. Thus, leading to defects in the sensual flavors of olive oil. In addition, too bad practices in harvesting, production, transport and storage.may lead to sensory defects To reach consumer, satisfaction, and protect them from fraud, the sensory evaluation for olive oil is necessary. The use of traditional analytical techniques is limited by the need for expensive instrumentation and complex sample preparation. Sensory analysis takes long time and requires an optional sensory plate, which can only evaluate a few samples daily. In this work, the potential use of the electronic tongue was assessed as a flavor sensor, to determine sensory advantage of olive oils.

1-2 Research Questions

1. Is electronic tongue able to distinguish between inedible olive oil and edible olive oil?

2. Is electronic tongue able to distinguish between the positive sensory characteristics, for extra virgin olive oil (fruity, bitter and pungent)?

1-3 Research Objectives

This research attempts to find out the range of possible applications of the electronic tongue, to evaluate olive oil. So the objectives of this research are to examine:

1. Verification of electronic tongue ability to distinguish between inedible olive oil and extra virgin olive oil.

2. Verification of electronic tongue ability to distinguish between the positive sensory characteristics, for extra virgin olive oil (fruity, bitter and pungent).

Chapter Two

Literature Review

2-1 The Economic Importance of Olive Oil in Palestine

The cultivation of olive trees in Palestine dates back thousands of years, and this is justified by the presence of long-standing olive trees. The importance of this sector in Palestine lies in the large number of olive trees; more than 10 million trees; and the size of the area covered by this tree. The cultivated area is estimated at 938,000 dunum, according to the statistics of the Ministry of Agriculture and the Palestinian Central Bureau of Statistics (OSHA, 2018).

The average production rate of olive oil in the good year is estimated at about 30 thousand tons; while in poor years, is about seven thousand tons. These estimates have declined in recent years, reaching 22 thousand tons of oil in 2016 (Oxfam, 2017).

Agriculture in Palestine contributes to the operation of a large number of households in the harvest season. This is due to the fact that the number of households operating in the olive sector in Palestine is close to 100 thousand. Agriculture operates a large number of industrial and non-industrial establishments, directly, throughout the agricultural season, of which more than 250 presses, a good number of packaging facilities, marketing companies and others (Abdullah and Hussen, 2016).

In Palestine, olive oil is the main product of the olive sector. The amount of fruit used for the production of olive oil constitutes 90% of the total production, while the rest is used in the production of pickles in a purely traditional manner. In recent times, the olive sector in Palestine has declined in its economic value for many reasons, including: the climatic causes and the decline in rainfall; olive cultivation in Palestine depends entirely on rainwater. On the other hand, there are social reasons such as the ownership rate has become very low; the impact on the care of olive trees has been reduced (Meneley, 2011).

2-2 Olive Cultivars in Palestine

The varieties of olive cultivated in Palestine are very similar, making it difficult to distinguish between them. It is also difficult for the researcher to identify the small spot in which they were planted. Nabali, for example, has more than 1,000 years of trees in many locations in Palestine. In the villages of Nablus and Jerusalem, Nabali trees are estimated to be one hundred years old. The following is a list of the most important varieties cultivated in Palestine with the most important features (Sameer, 2016):

A. Nabali; the most important and widespread variety, is preferred by olive farmers, to follow the oil ratio. It farmed in highlands with rain more than 400 ml per year. Where 34%. of fruit weight is oil.

B. Al-Nabali Al-Mohsen; which comes second in the cultivated varieties in Palestine. It is cultivated in irrigated areas and supplementary irrigation areas, according to the intensive farming system which is one of the highest productive varieties. The percentage of oil in this type, about 22-30% of the weight of the fruit in full maturity. There are many types of olives, less common and important such as suri, K18 and Mallisi.

2-3 Classification of olives oil in terms of quality

Olive oil can be classified into different grades, which reflect the quality of that oil through simple chemical tests and sensory tests, according to the International Council of Olive Oil, where olive oil is classified accordingly into four categories:

Extra Virgin Olive Oil (EVOO)

It is fit for human consumption and high quality. This type is the most important and the best type of olive oil, and it is the highest price. EVOO is produced from the first era of olive, with acidity within that type only 0. 8%. The lower acidity, the higher quality oil, the higher purity, and the higher price. This type is extracted by natural methods as a method of pressure without heating, so as not to change the specifications of the oil, the oil retains its original taste and smell of the original olive and retains all the vitamins, minerals and antioxidants. EVOO also contains a high proportion of nutrients and essential fatty acids, and vitamin E, and has a distinctive taste warns of its use at high temperature because it loses a large part of its health benefits (Serrano et al., 2018).

Virgin Olive Oil

It is fit for human consumption but for a lower quality than the first. Different from the type EVOO in the level of acidity, making it less quality than the previous type due to high acidity to 2%, which affects the taste and smell and health benefits.

Ordinary Virgin Olive Oil

It is an oil suitable for human consumption but very low quality. Ordinary olive oil or pure virgin blend of plain and virgin type is ranked last, in terms of quality and processing method. This type is used for cooking and is darker than other types and affects the taste. Acidity of this kind is less than 3.3% (Table 2-1).

Lampante

This is considered unfit for human consumption and can be used in non-food industries. This type of olive oil is considered one of the quietest kind, and therefore it is low in price to half the price of the previous kind. It does not contain any health benefits, nutrients, taste or smell, and is manufactured through olive groves. Remaining from the manufacture of previous kind from olive oil. As some harmful chemicals are added to it, which loses him any health benefit.

2-4 Sensory characteristics of olive oil

Olive oil is different from other vegetable oils, as it contains a small percentage of non-fatty compounds, and therefore it is noted that olive oil has a color, taste and flavor, while the rest of the vegetable oils refined, no taste and no flavor and color, because all materials have been removed, during the refining of those oils.

Due to the fact that olive oil, virgin and non-refined oil, any bad treatment during harvesting, transporting, storing, extracting oil or storing and storing oil, will lead to negative sensory in olive oil.

There are positive features of the positive olive oil, which is supposed to be in the oil if fruits harvested correctly. There are also, negative sensory that may exist in olive oil, due to misbehaviour, when dealing with the fruits of olives, and oil extracted from them.

The following is a description of each positive sensory character, presumed to be present in olive oil and any negative sensory character, likely to be present in olive oil, the reasons for the appearance of each characteristic, and how it is perceived and identified (Muzzalupo et al., 2012).

2-5 Positive sensory characteristics of olive oil:

1. **Fruity**

It is a set of sensations that are distinguished mostly by a sense of smell through the back of the nose, after taking a deep breath to distinguish the smell (sniff) sample of olive oil, after putting it in a cup and warm it to a temperature of 28-30°C. With the palm of the hands with the closure of the opening of the cup from the top by hand, before smelling the sample to help exit the smell of the sample. It is known that distinctive characteristic of the oil extracted from the fruits of olive fresh, healthy and mature or immature. Thereafter, the tested sample is spread widely over the mouth.

2. **Bitter**

It is a light bitter taste, which is felt in the back of the tongue, when the sample of the oil is tasted according to the conditions referred to above, especially after swallowing a small amount of it and this characteristic of olive oil extracted from the fruits of green or green converted to violet or black.

3. **Pungent**

This is characteristic of olive oil extracted from olive fruits at the beginning of the season or that the fruits of the olive have not yet fully matured or are in the early stages of maturity.

2-6 Negative sensory characteristics of olive oil:

1. **Fusty, muddy sediments**

They are the result of anaerobic fermentation due to the preservation of olives in bags stacked on top of each other before extracting oil from it, or as a result of contact the oil, while storing it with the sediments remaining in the bottom of the packages due to non-filtration of oil.

2. Musty – Humid

It is the negative sensory characteristic resulting from the storage of olives in a humid atmosphere for several days, which multiplies the number of yeast and molds in olive and feel close to the sensation associated with chewing samples of old nuts, which were saved in bad condition.

3. Winey – Vinegary

Which is the negative sensory properties caused by acetic acid, ethanol and ethyl acetate in large quantities during olive fermentation before extracting oil from it.

4. Earthy

A negative sensory characteristic, which may be found in olive oil extracted from olive fruits collected directly from the ground, and was in direct contact with the soil and mud and was not washed properly.

5. Grubby

The negative flavor found in olive oil extracted from olive fruits was attacked and damaged by larvae from the olive fly.

6. **Hay- wood**

A negative flavor that resides in the extract from the olive mill pomace olive oil or olive oil was dried before extracting it.

7. **Cucumber**

It is a flavor that appears in olive oil when it is stored tightly and for very long periods.

Each flavor is given with certain points or degrees, from 9 to 1.

There is a relationship between sensory and chemical tests to judge the quality and validity of olive oil where (Table 2-2):

In the case of median good flavor "olive flavor or fruit" gets between 9 and 6.5 points,

- A weak flavor that has a number of 6.5points

- Flavor with weak negatives that get a number of 5 points

- Flavor with medium negatives with a number of 4 points

- Flavor with significant negatives that takes a number of 3 to 1 points

2-7 Sensory evaluation of Palestinian olive oil, based on the Standards Institution of Palestine

In order to reach the olive sector to the level of international quality, so that it can compete with its counterparts in the countries producing olive oil and takes a wide spread in the global markets, the efforts have been collected and supported the Palestine Standards Institution through the enhancement of economic and social productivity through the establishment of laboratories for olive oil sensory testing. Where the members of the team to taste the olive oil, conducted a taste test in the laboratory of the institution, which has been processed according to the requirements of the IOC. The taster can determine the defects and positive qualities in the oil sample correctly. The results are obtained by the tasters, who must be at least thirteen and no more than seventeen, where the oil is classified and the results are entered into a special program that classifies the sample of the oil through the computer and then the laboratory issues the examination report for the sample examined.

The institution conduct sensory evaluation process, through the laboratory of taste, which established to classification of olive oil (extra virgin, virgin, ordinary virgin), and the national team (seventeen members) to taste olive oil, make a sensory examination in the laboratory of the institution, which was equipped according to the requirements of Specification International Olive Council. In order to determine the defects and positive qualities found in the oil sample correctly, and through the results obtained, the oil sample is classified.

Sensory evaluation to judge the quality of olive oil:

Sensory evaluation is used as one of the methods of assessing the quality of olive oil. Sensory tests are performed in specific rooms with special specifications and under certain conditions; each flavor should be given certain points or grades (i.e. 1-9). There is a relationship between sensory and chemical tests to judge the quality and validity of olive oil, where in the case of good-flavored (i.e. good fruity flavor) take 6.5-9 points; weak flavor take 6.5 points, the weak flavor take 5 points, the flavor of the medium-cons take 4 points, the flavor of the big cons take 3 points to one point.

The Institution of Standards organizes the Golden Olive Oil competition annually to raise the quality and excellence of extra virgin olive oil and strengthen the image of this product in the local and foreign markets to give the production chain a positive opportunity to compete through the application of good agricultural and production practices for development of oil quality.

After the end of the olive harvest season, the Foundation invites farmers and producers to progress and compete by participating in the competition under certain conditions.

Sampling tests shall be conducted through the laboratory of the Standards Institution by conducting a sensory evaluation and a chemical analysis for the selection of samples corresponding to the requirements of the Palestinian Standard No. 188 for olive oil for the classification of extra virgin oil. Based on the evaluation, the organizers select the best samples to move to the second and final stage.

Samples that are eligible to participate in the second stage, which meet the following requirements: acidity less than 0.7%, the peroxide index is less than 15 Milliequivalent of Oxygen/ kg of oil, and the absence of sensory evaluation (taste test) of defects.

Table (2-1) Acidity Standard of olives oil

Acidity Standard				
	Extra Virgin	Virgin	Ordinary	Lampante
International Codex	< 0.08%	< 2%	< 3.3 %	> 3.3%
European EU	< 0.08%	< 2%		
Peroxide level < 20.0 mEq O_2 / kg Oil				

Source: Palestine Standard Institution (Ministry of National Economy)

Table (2-2) Sensory Characters of olives oil

Sensory Standard				
	Extra Virgin	Virgin	Ordinary	Lampante
International Codex	MD = 0 MF > 0	0 < MD ≤ 3.5 MF > 0	3.5 < MD ≤ 6.5 MF > 0	MD > 6.5 MF = 0
European EU	MD = 0 MF > 0	0 < MD ≤ 3.5 MF > 0	3.5 < MD ≤ 6.5 MF > 0	MD > 6.5 MF = 0
MD. Median Defect:	MF. Median Good Feature (fruity taste)			

Source: Palestine Standard Institution (Ministry of National Economy)

2-8 Procedures for traditional sensory assessment

The following procedures is according to International Olive Council (IOC) (2015)

Requirements

The following supplies must be provided to the tasters to perform their task correctly so that they are in hand:

- Bottles (uniform) containing samples, numbered with a symbol, covered with a glass cover transparent and kept at 28°C ± 2°C.

- A hard copy of the datasheet or an electronic copy provided that the requirements of the datasheet are met, together with instructions for use if necessary.

- Ink pen or stylus pen that cannot be erased or removed.

- Cup of water at room temperature.

Sensitive assessment procedures and classification of extra virgin olive oil

Sensory taste method

The tasters carry the glass sample and keep it sealed with the lid and then gently stir it and rotate it until the inside is almost homogenous. After these Steps, the tasters remove the glass cover and start smelling the oil sample while taking a breath deep slow during oil assessment. It should not take more than 30 seconds to clean the oil. If the connoisseur does not reach a conclusion after following these steps, he can take a break short before trying again.

After a test the sense of smell, tasters assess the senses (smell, taste and touch) to do so, they must take a small sip (about 3 ml) of oil. It is very important that the oil is distributed throughout the mouth cavity, from the front part of the mouth and tongue, along the sides to the back and throat, where it is known depending on the area where the taste occurs, as the taste and taste vary in intensity of the tongue or throat. The taster focus on taste stimuli, taste of bitterness or pungent.

When performing the sensory assessment of virgin olive oil, it is recommended that the assess is four samples at the maximum of one session, and three session at most per day. to avoid the effect of variation that can be produced by successive tasting samples immediately.

The process of tasting the oil, lead to feeling tired or loss of sensation caused by the taste of previous samples, so it is necessary to take a product that can help eliminate the taste of oil remaining in the mouth.

Use the datasheet by the tasters

The details of the data sheet for use by the tasters are listed in Table 2-3. Each tasting must smell and taste the oil to be evaluated. Then it must enter the degree of both negative and positive properties of oil within the scale of the 10 cm table shown in the datasheet (IOC).

Table 2-3: Data sheet of the tasters used by IOC and Palestinian team.

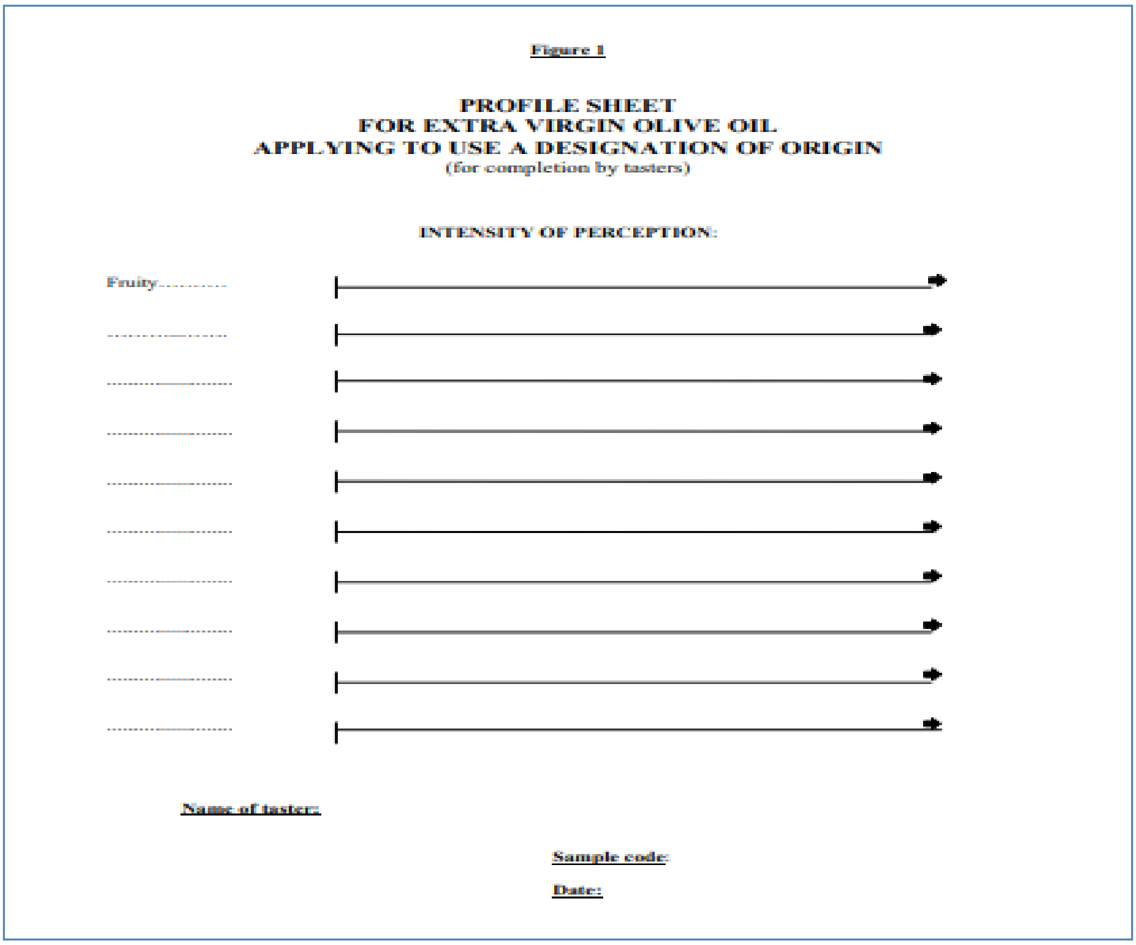

Figure 1

PROFILE SHEET
FOR EXTRA VIRGIN OLIVE OIL
APPLYING TO USE A DESIGNATION OF ORIGIN
(for completion by tasters)

INTENSITY OF PERCEPTION:

Fruity...........

Name of taster:

Sample code:

Date:

If tasters notice any negative oil properties not included in the definitions, they may register them under "other attributes", using the term that describes this attribute more accurately.

The committee chair collects the data sheets that the tasters completed and then reviews the degree of severity of each oil characteristic. If the auditor references radical differences in the results of the sensory assessment, he or she may summon the connoisseur to review the data he or she has filled, or to repeat the sensory evaluation process.

The Chairman of the Committee shall enter the evaluation data into a computer program in order to calculate the evaluation results. Statistically based on calculation of average data. The data are entered into a particular sample with the help of a nine-column table representing nine sensory characteristics and a number of lines representing the number of members of the committee who made the tasting process.

2-9 Electronic tongue device

The device consists of a sensor arrays; these sensors are made of ion-selective field-effect transistors (ISFETs) with different organic layers, these layers are equipped with appropriate sensitivity and selective sensors. This device measures organic compounds dissolved in liquids, where five types of basic tastes, which contain compounds of flavor, are identified and measured. These tastes include (sweet, sour, salt, acid and the taste of monosodium-glutamate) as it shown in Figure 2-1.

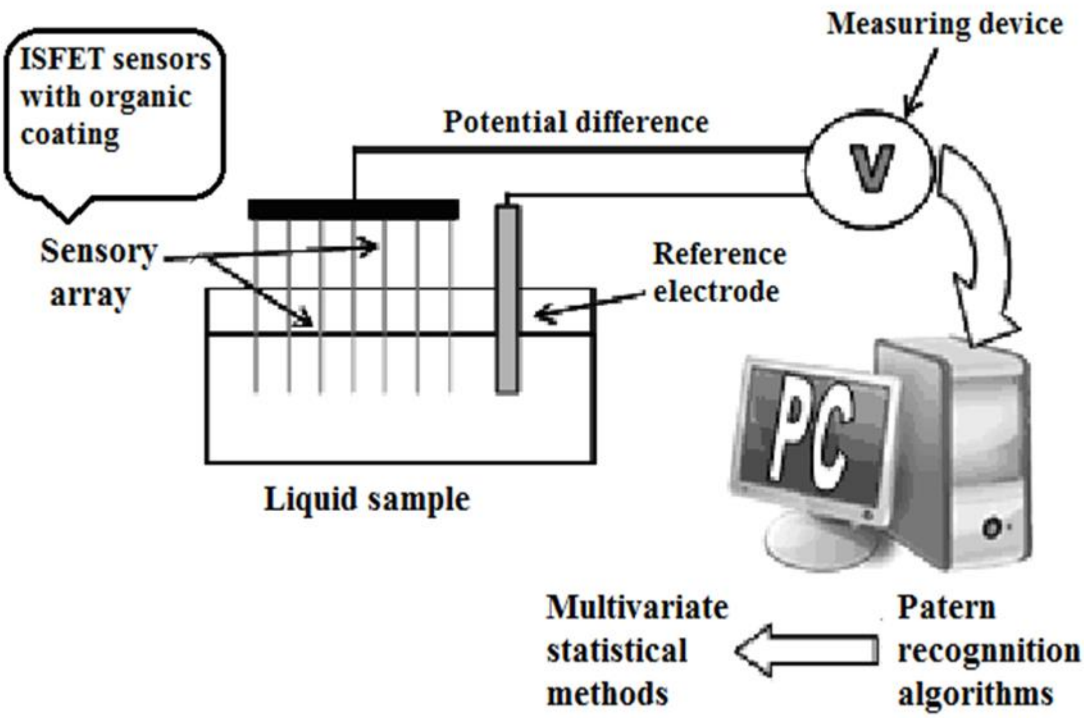

Figure 2-1. Schematic diagram of a potentiometric type electronic tongue, showing seven ISFETs sensors immersed in complex liquid sample, the corresponding potential difference signals are sent to a signal processor system to be analyzed using pattern recognition algorithms (Kovacs et al., 2009)

The electronic tongue converts the variables and translates them into primary data and results, through the analysis of multivaraite data analysis, and one of them is principal component analysis (PCA).

PCA is a mathematical process belonging to data analysis, which is to convert a number of interrelated variables into fewer non-interrelated variables. Variables resulting from the conversion process are called main compounds (or components or axes). The value added of the process is to facilitate the interpretation of complex data, by enabling the researcher and statistician to achieve the optimum compatibility between reducing the number of variables descriptive data, and the loss of the original information (variance) resulting from the reduction of the original dimensions.

This analysis aims to identify key components that illustrate the correlation pattern between groups of variables observed. It is also commonly used to identify fewer factors that largely explain the observed change in a larger number of observed variables. The main components are estimated in the form of linear combinations, independent of each other and composed of observed variables, and for the number of variables observed.

Electronic tongue device was used to distinguish between basic tastes and to develop a typical solution, where the aim was to control the quality of the liquid, and the accuracy levels were less than the human sensor. In addition, the aim of the electronic tongue was to detect the interactions between tastes (Deisingh et al., 2004).

The electronic tongue was also used in environmental applications, in order to detect pollutants in water, it was able to measure heavy metal, alkali, alkali-earth cations and inorganic anions from groundwater models with good results (Rudnitskaya et al., 2006).

The electronic tongue was also used in the process of controlling fermentation industries. Where organic compounds and biomass could be measured, detecting small concentrations, achieving good results, predicting, flavor fermented materials (Esbensen et al., 2004).

The electronic tongue was used in the pharmaceutical industry, it was possible to distinguish different types of sweeteners and to detect defects related to the taste of some compounds (Legin et al., 2004).

The application of the electronic tongue was applied in many food sectors, as follow (Kovacs, 2009):

- Product conformity to quality.
- Detecting relevant taste assumptions.
- Study of masking the taste.
- Identification of sample origin.
- Identification of the sensory intensity of the product.
- Identification of the validity of product consumption, (shelf life).
- Detection of defects and fraud related to olive oils and liquids.

2-10 Previous Studies

The study of Peter and Rwothomio (2011) described olive oil in terms of its properties. Oil was collected from three different geographical regions in New Zealand, in order to study the composition of fatty acids and tocopherol as well as to know the sensory properties of olive oil. The olives was extracted by accelerated solvent extraction (ASE) throughout the season and by cold pressed (CP). Through cold pressing, the sensory properties of olive oil were assessed and a chemical test was measured to determine bitterness. The results showed a strong positive correlations between sensory and sensitive bitterness. Olive oil from different geographical areas, emphasizing the different sensory characteristics distinctive and smell and flavor and described as "bitter salad", fresh "green beans", "vanilla died", "walnuts" and "black pepper". The researcher concluded that in order to maintain acceptable levels of sensory of bitterness and fruity, olives should be harvested at maturity.

Slim et al. (2017) developed modern techniques in order to ensure the health and quality of olive oil. The traditional techniques for measuring the sensory quality of olive oil are expensive, time-consuming, and not strictly applied.

The electronic tongue works on the basis of the distinction between Tunisian olive oil and its quality level (extra virgin, virgin or lampante olive oils), through transparent lipid membrane sensitivity. The study showed that the reference forms for extracts of hydroxytyrosol olive oil, allowed olive oils to be distinguished, according to the physical quality level (a 25-point classification model that enables correct ratings of 84 ± 9% for repeated K-fold verification). Olive class (a classification model based on 20 signals with an average sensitivity of 94 ± 6% for repeating K fold over validation), regardless of geographical origin, olive variety or olive quality, respectively. The results confirmed, for the first time, the potential discrimination of electronic tongue, attributable to the observed quantitative response of electronic multi-lingual sensors towards standard solutions for polar compounds (aldehydes, esters and alcohol) in olive oil which are related to the positive qualities of sensory own such as green and fruity.

A research by Burattia et al. (2018) aimed to identify and verify the ability of the electronic tongue to classify the olive oil (extra virgin, olive and pomace) during storage at different temperatures. 24 samples of olive oil were obtained. The samples were distributed as 10 samples of extra virgin olive oil, and 10 samples of virgin olive oil. And 4 pomace oils. In order to analyze the data, physical and chemical analyzes were carried out and processed by Principal Component Analysis (PCA). The data sheet was used through physical and chemical sensors. The results showed that the sensory electronic tongue was able to characterize the olive oil into different categories. It also determined the appropriate conditions for olive oil storage and identification shelf life.

In a study conducted by Apetrei et al. (2017), they added that the use of the electronic tongue individually, can obtain an accurate sensory characterization, it was necessary to use the nose electronic side by side of the electronic tongue through the use of technology integration between the two devices for the sensory evaluation of extra virgin olive oil.

Other application for electronic tongue measuring the olive oil sensory during cooking. The use of olive oil in cooking leads to a decrease in the concentration of phenol, as well as a change in sensory levels, as a result of heat. It was found that indicates that the use of heat and microwave, reduced the content of phenol, with a decrease fruit intensity, sweet, bitter, especially for fruits and sensations. In addtio , the density of the bitter fruit was directly proportional to the high heat. This indicates that, in fact, maybe a satisfactory performance rating associated with electronic tongue, known as the ability to interact with taste materials (Prata et al., 2018).

Using the electronic tongue as a taste sensor, helps to determine the perceived defect in olive oils. The use of a tasting sensor, as an integrative approach, assists in the classification of olive oil and identification of defects, which helps to determine the high quality and ease of marketing, as well as the electronic tongue machine is useful in relation to practices of olive oil production, harvesting, storage and transport (Veloso et al., 2018).

Total phenol content was assessed by chromatography. Absorption after extraction and interaction with the Folin-Ciocalteu detector, three EVOOs were evaluated for their phenol content group through sensory analysis, which relates to the relationship between storage methods, container type, phenolic content and hydrogenation acceptance followed by sensory study. The analysis showed that olive oils differed significantly in the concentration of phenolic compounds; sensory analysis showed a preference for low to moderate olive oils of phenolic compounds. Comparing the market analysis of locally available olive oils, the price was not linked to consumer preference or phenol compound content (Madeleine, 2018).

Chapter Three

Research methodology

3-1 Material and Methods

Methodology of the liquid-liquid extraction and measurement of olive oil samples were conducted as stated by the manufacture of the device (Alpha MOS, 2009). We added 120 mL of solution consist of (80 ml of distilled water and 20 ml of ethanol) to 120 mL of provided olive oil, and then the sample was stirred for one minute followed by putting the samples for 30 min of ultrasonic shaking, and then samples were left for settling. Each sample was filtered with Polytetrafluoroethylene membranes filters (PTFE). The solution finally analyzed with the ET will be constituted of 80 mL of the lower extracted solution and 12 mL of ethanol solution.

Between two analyses, the sensors will be rinsed with a H_2O/EtOH (70/30) solution.

Samples were analyzed 3 times each, to take into account an average measurement, at ambient temperature, with an acquisition time of 120 seconds.

Samples were taken from the Palestinian Standards Institution(Ministry of National Economy) , and have been tested by the National Sensory Evaluation Committee. The twenty-three samples have successfully passed all sensory and chemical tests and reach the final stage of the Golden Olive Oil Competition. Organized annually by the Palestinian Standards Institution (Appendix).

These samples were collected through olive oil companies, associations and farmers. Samples were taken, meeting the criteria of extra virgin olive oil. Olive oil samples back to the harvest season 2017-2018.

3-2 Study procedures

Samples were stored at 17°C. The volume of each sample is 250 ml. The capacity of the electronic tongue device is 16 units. Therefore, the analysis was carried out in two stages. In the first phase, 13 olive oil samples (i.e. 12 from PSI and one lampante from the market) were introduced, in addition to 3 samples of H_2O/EtOH (70/30) solution. In the second phase, 12 olive oil samples (i.e. 11 from PSI and one lampante from the market) were introduced, in addition to 4 samples of H_2O/EtOH (70/30) solution.

Work mechanism: It is done by reading three olive oil samples, and then washing the sensor once through H_2O/EtOH (70/30) solution, and then re-work and read three other samples. Each sample needs 120 seconds to read, while washing the sensor takes 20 seconds. The experiment was repeated three times in order to obtain more accurate data.

As mentioned earlier, the electronic tongue has 7 sensors. Works in 6 rounds. The first and second rounds are used to initialization sensors to work perfectly. The third, fourth and fifth rounds are used for statistically analysis the data the device. The sixth rounds is not taken into consideration for data analysis (Fig 3-1)

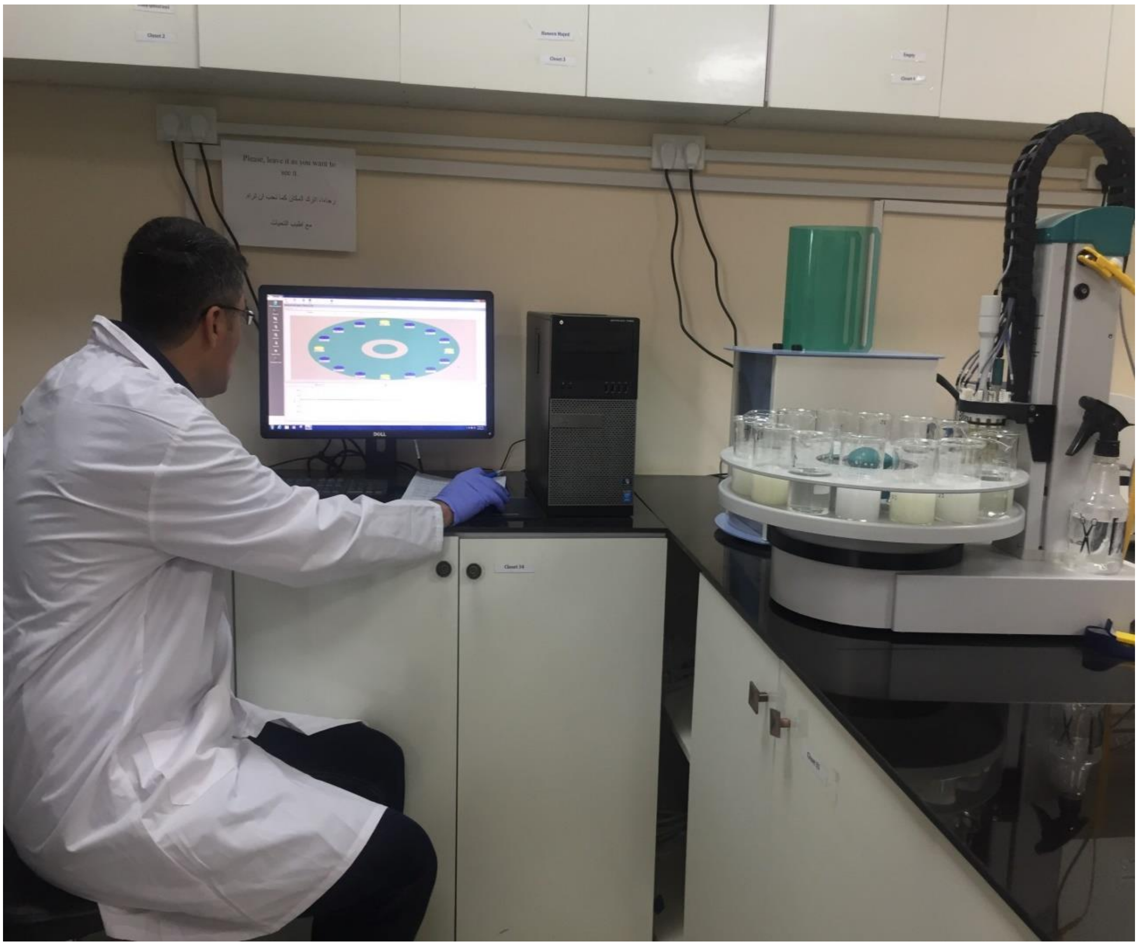

Figure (3-1) The electronic tongue during measure olive oil samples.

3-3 Statistical Analysis

Different type of multivariate data was applied. This analysis is based on a statistical principal component analysis (PCA), which was used to classify samples. Also, partial least squares (PLS) for building model of quality parameters.

Chapter Four

Results and Discussion

4-1 Introduction:

In this section, we analyzed the samples and discuss the obtained results. The electronic tongue converts the variables and translates them into primary data and results, through PCA and PLS. To ensure accurate results, calibration and validation have been applied.

4-2 PCA for detecting outliers sample

Sample no. 24 unfit for human consumption (lampante)were analyzed in order to know the ability of the electronic tongue to classify the oil, which known as lampante.

Figure 4-1 shows the result of the outlier sample when measure sample 1- 12.

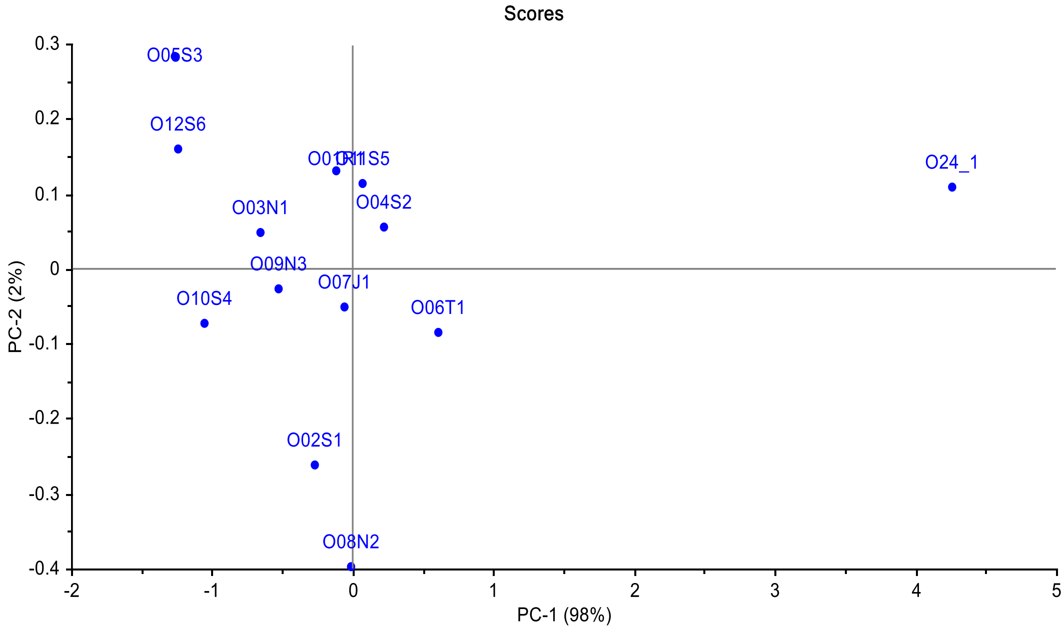

Figure (4-1) PCA scores plot of 12 olive oil samples from PSI and one lampante sample (as one outlier).

PCA scored plot shows that the sample number 24 is far away from the other samples, since it is an outlier.

Figure (4-2) show the result of outlier's sample of (13- 23).

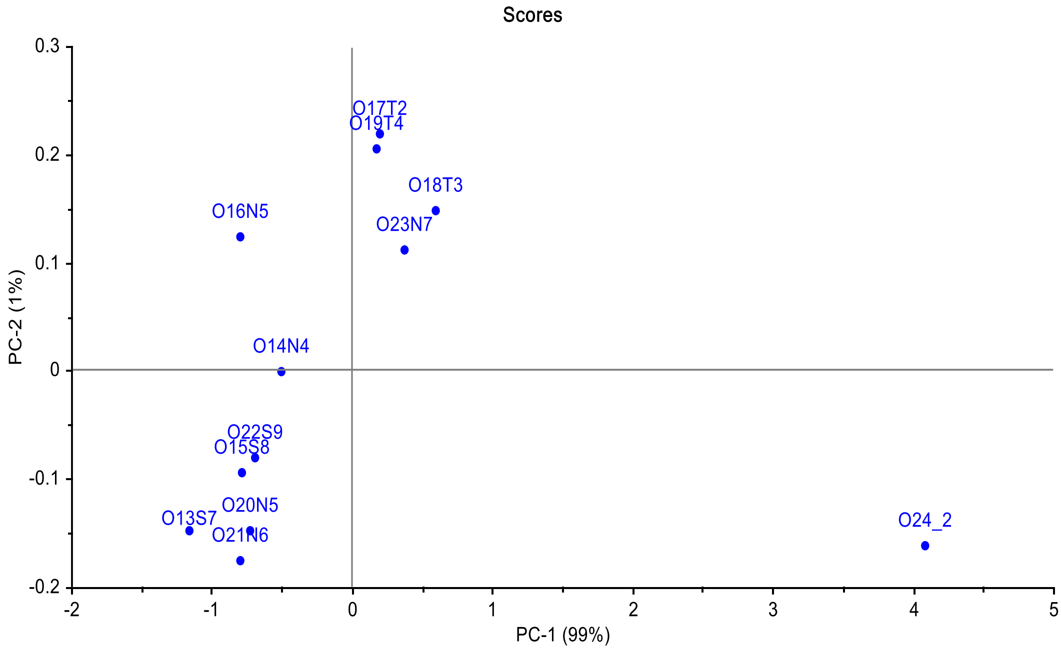

Figure (4-2) PCA scores plot of olive oil samples (13-23) showing outlier sample

Figure 4-2 also shows the sample number 24 as an outlier.

4-3 PLS for modeling quality parameters

In this analysis, it will show the results of extra virgin olive oil, for its positive sensory characteristics.

1- Fruity

Calibration set

In this analysis, will be shown the results of the predicted fruity taste scores (by PLS) versus reference taste panel scores for calibration set for samples 1-23.

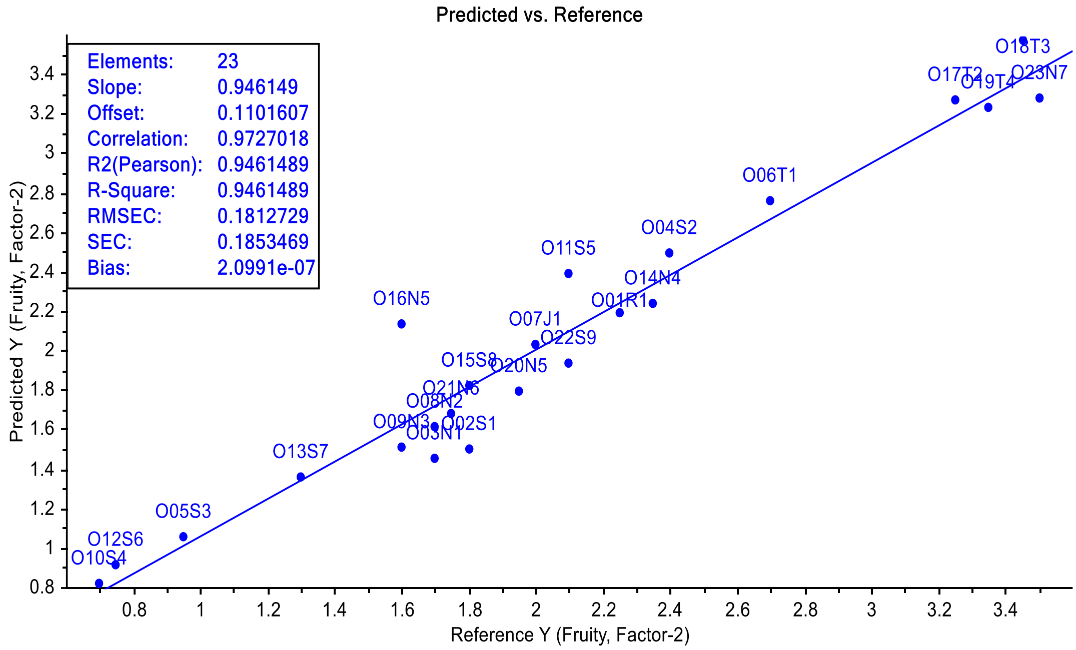

Figure (4-3). The predicted fruity taste scores (by PLS) versus reference taste panel scores for calibration set for samples 1-23.

The models enabled the prediction of the fruity median intensities. With Pearson correlation of 0.94 (for full cross- calibration) (R^2 = 0.946 and RMSE = 0.182, for full cross- calibration procedures). Correlation: 0.972.

Validation set

In this analysis, it will show the results of the predicted fruity taste scores (by PLS) versus reference taste panel scores for validation set for samples 1-23.

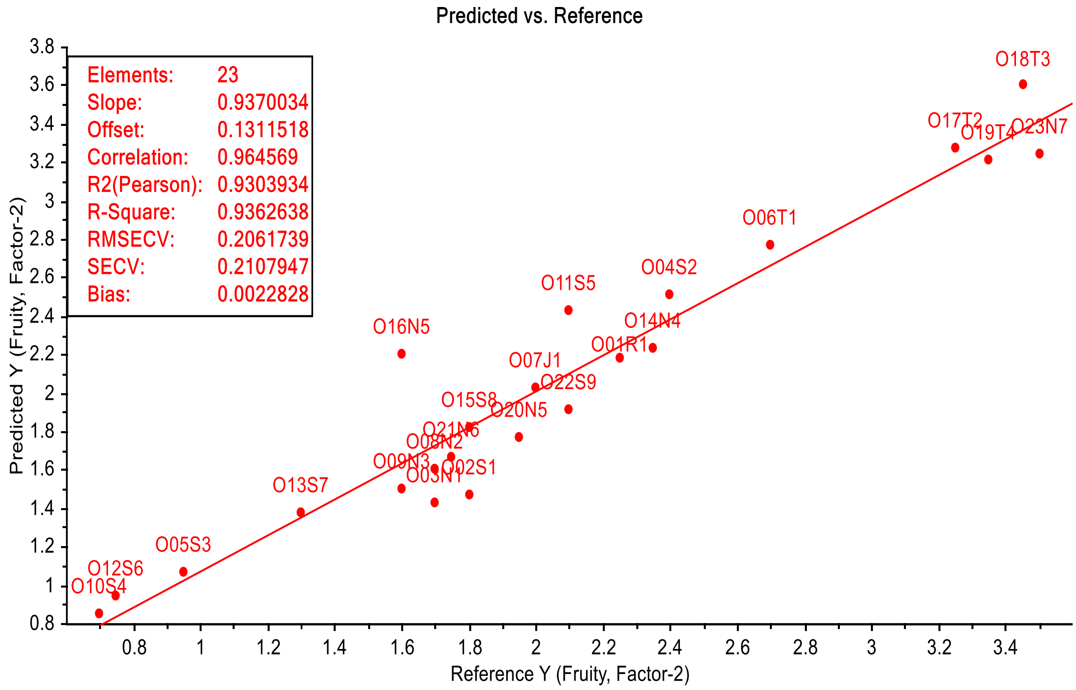

Figure (4-4). The predicted fruity taste scores (by PLS) versus reference taste panel scores for validation set for samples 1-23.

The models enabled the prediction of the fruity median intensities. With Pearson correlation of 0.93 (for full cross- validation) (R^2 =0.936 and RMSE=0.206, for full cross- validation procedures). Correlation: 0.964.

2- Bitter

Calibration set

In this analysis, it will show the results of the predicted bitter taste scores (by PLS) versus reference taste panel scores for calibration set for samples 1-23. See figure (4-5).

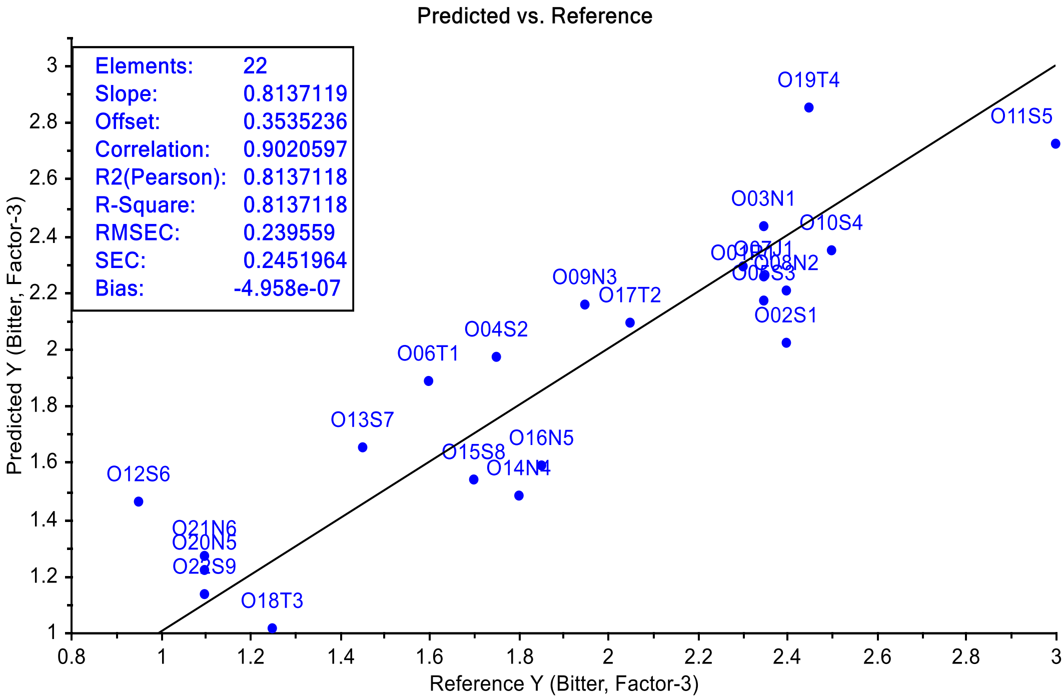

Figure (4-5). The predicted bitter taste scores (by PLS) versus reference taste panel scores for calibration set for samples 1-22 (one sample, i.e. 23, was removed as an outlier).

The models enabled the prediction of the bitter median intensities. With Pearson correlation of 0.81 (for full cross- calibration) ($R^2 = 0.813$ and RMSE =0.239, for full cross- calibration procedures). Correlation: 0.902.

Validation set

In this analysis, it will show the results of the predicted bitter taste scores (by PLS) versus reference taste panel scores for validation set for samples 1-23. See figure (4-6).

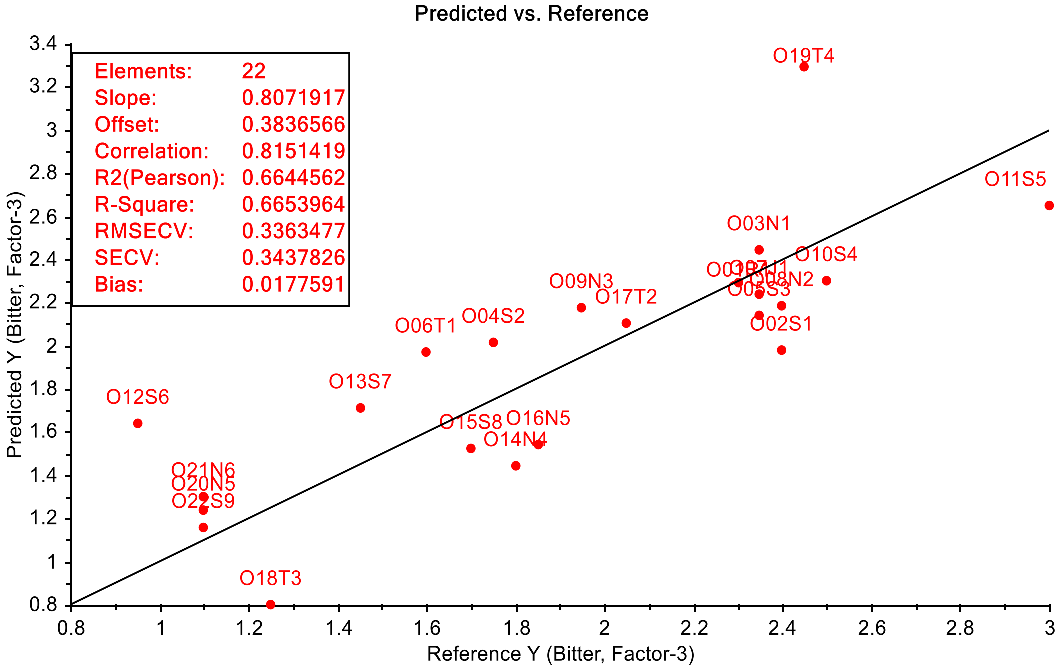

Figure (4-6). The predicted bitter taste scores (by PLS) versus reference taste panel scores for validation set for samples 1-22 (one sample, i.e. 23, was removed as an outlier).

The models enabled the prediction of the bitter median intensities. With Pearson correlation of 0.66 (for full cross- validation) (R^2= 0.665 and RMSE = 0.336, for full cross- validation procedures). Correlation: 0.815.

3- Pungent

Calibration set

In this analysis, it will show the results of the predicted Pungent taste scores (by PLS) versus reference taste panel scores for calibration set for samples 1-23. See figure (4-7).

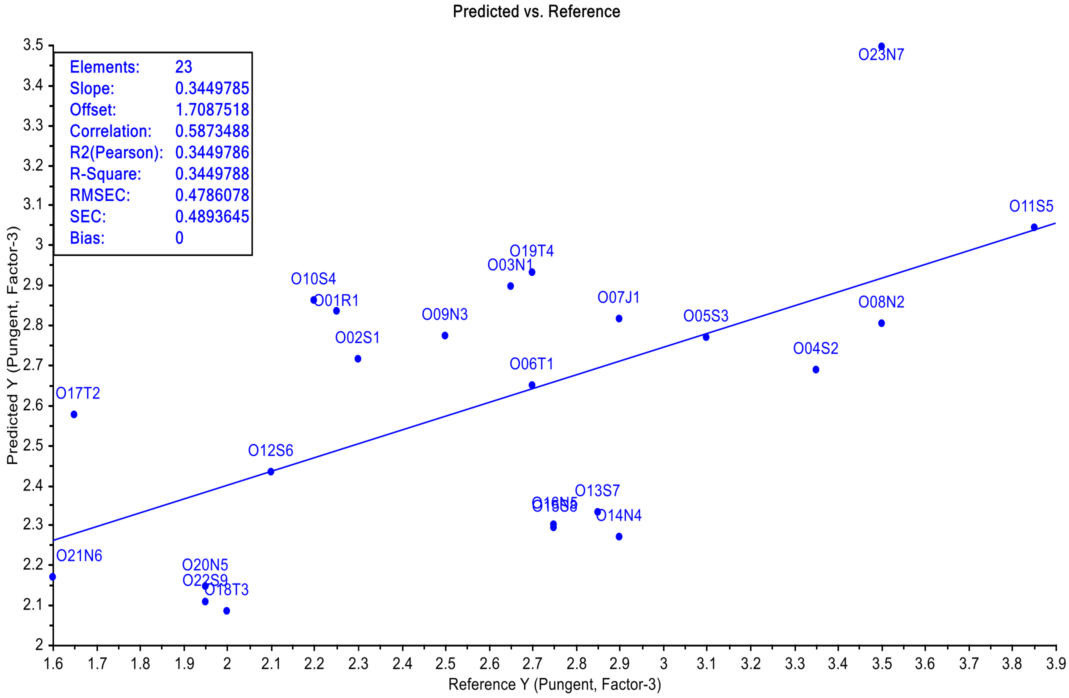

Figure (4-7). The predicted pungent taste scores (by PLS) versus reference taste panel scores for calibration set for samples 1-23.

The models enabled the prediction of the pungent median intensities. With Pearson correlation of 0.34 (for full cross- calibration) (R^2 = 0.344 and RMSE = 0.478, for full cross- calibration procedures). Correlation: 0.587.

Validation set

In this analysis, it will show the results of the predicted of the pungent taste scores (by PLS) versus reference taste panel scores for validation set for samples 1-23. See figure (4-8).

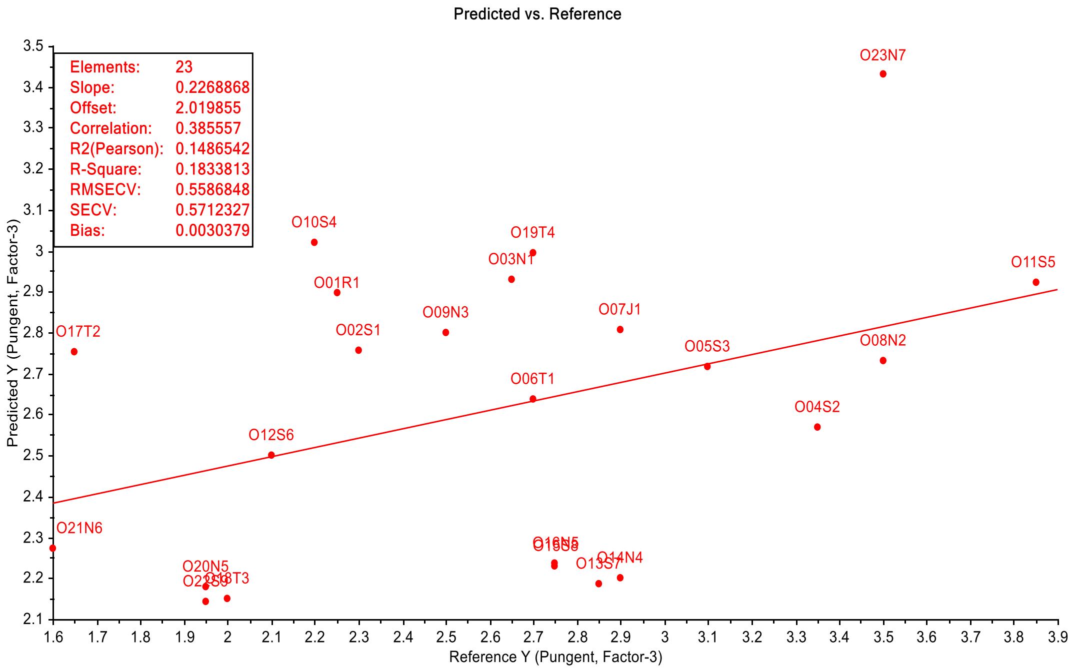

Figure (4-8). the predicted pungent taste scores (by PLS) versus reference taste panel scores for validation set for samples 1-23.

The models enabled the prediction of the pungent median intensities. With Pearson correlation of 0.14 (for full cross- validation) (R^2 = 0.183 and RMSE = 0.558, for full cross- validation procedures). Correlation: 0.385.

4-4 Summary of Results

Taste parameters		Fruity			Bitterness			Pungent		
Samples of olive oil		1-12	13-23	1-23	1-12	13-22	1-22	1-12	13-23	1-23
PCs used in PLS model		2	2	2	2	2	3	3	3	3
% variance explained	X	100	100	100	81	90	100	94	99	99
	Y	95	97	94	87	90	81	40	52	34
Slope	Calibration	0.94	0.97	0.94	0.87	0.89	0.81	0.39	0.51	0.34
	Validation	0.92	0.94	0.93	0.68	0.65	0.80	0.04	0.03	0.22
Correlation	Calibration	0.97	0.98	0.97	0.93	0.94	0.90	0.63	0.71	0.58
	Validation	0.95	0.95	0.96	0.85	0.84	0.81	0.05	0.03	0.38
R^2 (Pearson)	Calibration	0.94	0.97	0.94	0.87	0.89	0.81	0.39	0.51	0.34
	Validation	0.91	0.90	0.93	0.73	0.70	0.66	0.00	0.00	0.14
RMSE	Calibration	0.13	0.14	0.18	0.17	0.18	0.23	0.42	0.40	0.47
	Validation	0.18	0.25	0.20	0.26	0.30	0.33	0.68	0.87	0.55
RPD	Calibration	4.60	5.90	4.50	2.96	3.23	2.37	1.34	1.50	1.26
	Validation	3.54	3.30	3.96	2.01	1.92	1.68	0.83	0.69	1.08
Relative error (%)	Calibration	8.19	5.80	9.26	8.99	9.13	12.12	15.00	15.90	17.40
	Validation	10.64	10.50	10.30	13.25	15.39	17.02	24.00	34.30	20.30

Table: (4-1) Summary of results

RPD = SD / RMSE (RPD: is a good model when its value is greater than 1.4 (Williams and Sobering (1993)).

RE = RMSE / range

As shown in table 4-1, this study aims to verify the sensitive degree of positive sensory characteristics of olive oil (fruity, bitter and pungent). In regards to fruity, for full cross- calibration, $R^2 = 0.94$ and RMSE = 0.18, with a correlation of 0.97.

$R^2 = 0.94$ shows that strong positive correlation between ET signals and fruity scores.

Correlation; 0.97 this means that the relation between fruity and electronic tongue signals are , very strong relation.

Validation analysis for fruity also shown, that (for full cross-validation) ($R^2 = 0.93$ and RMSE = 0.20.. Correlation: 0.96.

$R^2 = 0.93$ shows that strong positive effect between variables.

Correlation; 0.96 this means that the relation between fruity and electronic tongue signals very strong relation.

In regards to bitterness, (forfull cross- calibration) ($R^2 \geq 0.81$ and RMSE = 0.23, for full cross- calibration procedures). Correlation: 0.90.

$R^2 = 0.81$ shows that strong positive effect between variables. But less than Fruity.

Correlation; 0.90 this means that clear positive relation was observed between bitterness and electronic tongue signals. Also, this relation is less than fruity.

Validation analysis for bitterness also shown, that (for full cross-validation) (R^2 = 0.66 and RMSE = 0.33, for full cross- validation procedures). Correlation: 0.81,

R^2 = 0.66. shows that positive effect between variables.

Correlation; 0.81 this means that the relation between bitterness and electronic tongue signals, very strong relation. But less than fruity.

In regards to pungent, for full cross- calibration R^2 = 0.34 and RMSE = 0.47, for full cross- calibration procedures). Correlation: 0.58.

R^2 = 0.34 shows that a weak positive effect between variables.

Correlation; 0.58 this means that the relation between pungent and electronic tongue signals, a weak relation.

Validation analysis for pungent also shown, that (for full cross-validation) (R^2 = 0.14 and RMSE = 0.55, for full cross- validation procedures). Correlation: 0.38.

R^2 = 0.14 shows that a weak positive effect between variables.

Correlation; 0.38 this means that the relation between pungent and electronic tongue signals, a weak positive relation.

To our best knowledge, there were no previous studies for studying the three positive taste character together of olive oil (i.e. fruity, bitterness and pungent). However, Nery et al. (2016) managed to classify olive oil according to their cultivars and geographic origins. Furthermore, Apetrie (2013) stated that they could quantify the total polyphenol content and discriminate of individual phenolic compounds in 18 extra virgin olive oil samples. However, Alpha MOS (2016) stated that ranked olive oils based on their bitterness, in the same way as sensory panel evaluation. Finally, the result indicated that ET was able to detect the outlier sample, which was lampante olive oil. Also, it was able to model fruity and bitterness in a high accuracy. The ET was able to model the taste panel scores. This may suggest that ET can be used in the future as a support device for a taste human panel.

Chapter Five

Conclusion and Recommendation

5.1 Conclusion

As we indicated at the beginning of the study, this study aim is to know the ability of the electronic tongue to measure and verify the sensitive degree of positive sensory characteristics of olive oil (fruity, bitter and pungent), based on the samples taken from the Palestinian Standards Institution.

The electronic tongue was able to distinguish between the specification of olive oil, which is bad for human consumption, and the olive oil samples, which have positive sensitivity.

In addition to that, the study showed an electronic tongue, which was able to distinguish between the three positive sensory characteristics of extra virgin olive oil. Where the study showed that a sample of extra virgin olive oil has a high fruity flavor and a lesser degree for bitter and pungent flavor.

This study came to support the work of the committees that assess the sensory of olive oil. From here, the importance of the electronic tongue comes, to confirm the accuracy of the work of the electronic tongue, in showing results and data, at the lowest cost and time.

The results obtained from the application of the electronic tongue, by adopting jointly the method of processing statistical data, help in making it easier to distinguish according to the sensory characteristic of olive oil, which contribute to reaching the sensory features of olive oil, and the continuous follow-up in storing olive oil to show the changes that are possible it occurs during the storage process. Also, determine appropriate temperatures for storage. as well as storage conditions(including humidity) which affects the sensory characteristics of olive oil.

The electronic tongue contributes to the marketing of olive oil internationally, as it helps in the rapidly assessment of olive oil sensually, and reduces effort and costs. And avoid fraud in the marketing of olive oil. Where modern analytical and technological techniques help in marking, related to the classification of the quality of olive oil. Traditional techniques are very expensive and require a long time to obtain results, so it is possible to rely on the electronic tongue and accreditation by the International Olive Council. To help farmers and merchants check and put labels on the oil boxes, and then facilitate access to global markets, which require physical and chemical testing as a condition for exporting olive oil. Also, this technology is very important in the pricing of the olive oil product, and therefore enhance the marketing and profitability opportunities for the product. It contributes to enhancing consumer confidence, and ensuring that the price is proportional to the sensory quality of the product. So, this technology must be taken into account for their quality commercial grade classification.

The applications of the electronic sensory tongue are very important in Palestine, especially in the olive oil production sector, as this technology can be used to contribute to providing sufficient information to consumers about the sensory olive oil classifications. With reference to the positive sensory flavor classification, are they flavored with fruity, bitterness and pungent.

This study confirms that to achieve a balance at satisfactory sensory level, between the flavors of olive oil (fruity, bitter and pungent), harvesting must take place at the point of maturity of the fruits.

Finally, the performance of the electronic tongue is generally satisfactory, and the electronic device is considered to have a sophisticated methodology using taste sensors in order to classify the grades of olive oil quality and detect defects. The electronic tongue can also be used to develop a beneficial perception for farmers, to get rid of wrong practices during the process of harvesting, production, transport, storage and shelf life.

5-2 Future Research and Recommendation

Some studies, such as Haddi et al., (2014) study, used the technology of combination between the electronic tongue and the electronic nose, where the electronic nose technique is based on the use of gas sensors. This combination to describe the quality of olive oil with high success rates. So, this approach should be addressed in future studies.

The quality of the extra virgin olive oil is chemically and sensually affected, at different temperatures during storage, as well as packaging materials have a role in influencing the olive oil sensitively. Future studies should take into consideration the setting of olive oil samples in different types of packaging such as glass and plastic gallons, in addition to storing those samples at different temperatures, in order to verify the ability of the electronic tongue, to measure the different sensory specifications of olive oil.

Electronic tongue applications should be used in order to help farmers in decermination the quality of the olive oil tree and the geographical location that matches the specifications of the extra virgin olive oil.

The farmers should be advised, through conducting many scientific researches, using the electronic tongue, in order to set the date for the harvest of olive oil, in addition to conducting sensory checks during the period of production, transportation and storage.

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Appendix

Name	Sample No.	Acidity	Peroxide		Fruity	Bitter	Pungent
OR1	1	0.42	5		2.25	2.3	2.25
OS1	2	0.56	5		1.8	2.4	2.3
ON1	3	0.34	5		1.7	2.35	2.65
OS2	4	0.34	5		2.4	1.75	3.35
OS3	5	0.31	5		0.95	2.35	3.1
OT1	6	0.42	5		2.7	1.6	2.7
OJ1	7	0.34	5		2	2.35	2.9
ON2	8	0.25	5		1.7	2.4	3.5
ON3	9	0.28	7		1.6	1.95	2.5
OS4	10	0.42	5		0.7	2.5	2.2
OS5	11	0.48	5.6		2.1	3	3.85
OS6	12	0.37	6.4		0.75	0.95	2.1
OS7	13	0.28	5		1.3	1.45	2.85
ON4	14	0.28	5		2.35	1.8	2.9
OS8	15	0.39	5		1.8	1.7	2.75
ON5	16	0.42	5		1.6	1.85	2.75
OT2	17	0.34	5		3.25	2.05	1.65
OT3	18	0.23	5		3.45	1.25	2
OT4	19	0.23	5		3.35	2.45	2.7
ON5	20	0.31	5		1.95	1.1	1.95
ON6	21	0.31	5		1.75	1.1	1.6
OS9	22	0.34	5		2.1	1.1	1.95
ON7	23	0.25	5		3.5	2.9	3.5

جامعة النّجاح الوطنيّة

كلية الدراسات العليا

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إشراف

أ. د. حسان ابو قاعود

قدمت هذه الأطروحة استكمالاً لمتطلبات الحصول على درجة الماجستير في الإنتاج النباتي بكليّة الدراسات العليا في جامعة النّجاح الوطنيّة في نابلس، فلسطين.

2020م

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الملخص

تهدف هذه الدراسة إلى بحث إمكانية تطبيق استخدام مجس كهروكيميائي يسمى اللسان الإلكتروني على زيت الزيتون وتحديدا في التمييز بين زيت الزيتون البكر الصالح للاستهلاك الآدمي وزيت الزيتون الغير صالح لذلك، وكذلك تقييم زيت الزيتون البكر الممتاز عالي الجودة من حيث الخصائص الحسية الإيجابية. وقدرة الجهاز الإلكتروني على تمييزها ودعم عمل الفريق الوطني للتذوق.

تم قياس وتحليل عينة من زيت الزيتون البكر الغير الصالح للاستهلاك الآدمي و23 عينة لزيت الزيتون البكر الممتاز. أخذت العينات من مؤسسة المواصفات والمقاييس الفلسطينية وذلك بعد تقييمها حسيا من قبل اللجنة الوطنية للتذوق. وهذه الثلاثة وعشرون عينة اجتازت بنجاح جميع الفحوصات الحسية والكيماوية النهائية الخاصة بمسابقة زيت الزيتون الذهبي. والتي تنظمها سنويا مؤسسة المواصفات والمقاييس الفلسطينية. حيث إن تحليل البيانات تم من خلال الجمع بين تقنية مجس اللسان الإلكتروني وتحليل البيانات المتعدد العوامل. وأظهرت نتائج تحليل الدراسة، أن اللسان الإلكتروني استطاع أن يتوقع الصفات الحسية الإيجابية الثلاث لزيت الزيتون البكر الممتاز. حيث إن الفاكهية هي الأعلى، يليها المرارة وبدرجة اقل الشعطة. وتشير النتائج إلى أن أداء اللسان الإلكتروني مرضٍ بشكل عام. وان هذه التكنولوجيا يمكن أن تساعد في قياس طعم وجودة زيت الزيتون.