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Electronic nose to differentiate between several drying techniques for *Origanum syriacum* leaves

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Abstract

Dried oregano (*Origanum syriacum* L.) is a common product in the Mediterranean diet and it has wide culinary applications. The quality and functional ingredients profile of oregano is highly affected by drying technology. This study was aimed to discriminate different quality traits of air, solar, and freeze-dried oregano by employing electronic nose (e-nose), chromameter, and sensory analysis. E-nose signals were analysed by using multivariate data analysis (MVDA). Our findings showed that the e-nose signal exhibited different clusters for all groups by using principal component analysis (PCA). Moreover, there were clear differences in the colour index (L*a*b*) between groups. Freeze-dried oregano exhibited significantly lower L*-values than air and solar-dried oregano. Sensory analysis showed that there were clear differences between solar and freeze-dried oregano. In this context, f-dried thyme had significantly lower values of colour acceptance (4.80 *vs.* 7.57, $p<0.05$), degree of freshness (5.57 *vs.* 7.14, $p<0.05$), taste acceptance (5.46 *vs.* 6.75, $p<0.05$), and overall acceptance (5.75 *vs.* 7.19, $p<0.05$) than solar-dried thyme, respectively. In conclusion, e-nose and chromameter were effective tools to discriminate between different types of dried oregano.

1. Introduction

Origanum syriacum is a well-known crop in Mediterranean regions. There are several common names for this herb such as "za'atar", Lebanese oregano, Syrian oregano, and Bible hyssop (The Herb Society of America, 2005). *Origanum syriacum* is classified as a perennial herb with 60-90 cm height, woody creeping roots, and branched woody hairy stems (Farhat *et al.*, 2012). Za'atar is one of the most common products in Mediterranean regions that is made from *Origanum syriacum*. Za'atar is a mix made from dried milled *O. syriacum* leaves, seeds, sesame, sumac berries (*Rhus coriaria*) and salt (Daouk *et al.*, 1995; The Herb Society of America, 2005). Za'atar has many culinary uses such as a mixture with olive oil consumed at breakfast (Daouk *et al.*, 1995; Mudalal *et al.*, 2020), spread on a pizza-like pastry called "Man'ousheh" in Arabic (United Nations, 2010), and as seasoning (Daouk *et al.*, 1995).

Za'atar has an intense and pleasant aroma due to the high content of essential oil (Daouk *et al.*, 1995). Oregano (*O. syriacum*) contains a high level of functional ingredients and aromatic compounds. Therefore, it was suggested as a treatment for several diseases such as respiratory disease: whooping cough, bronchitis and asthma (Salehi *et al.*, 2018). Oregano is rich in unique bioactive compounds such as carvazole and thymol (Xu, 2006). It also has a high content of folic acid, beta carotene, and vitamins (A, C, E, K) (Xu, 2006), zea-xanthin, pigenin, lutein, luteolin and thymonin (Hazzit *et al.*, 2006). Oregano (*O. syriacum*) exhibited anti-oxidizing, anti-bacterial, and anti-fungus properties (Eqbal *et al.*, 2017).

The most common and commercial drying technique that is usually employed to dry *O. syriacum* leaves is solar drying. Even solar drying is a clean process but it has many limitations because it is highly dependent on weather conditions. Besides, solar drying may produce an inconsistent product in quality and the risk of physical and microbiological contaminations are high (Douglas *et al.*, 2005). Other drying technologies such as freeze and air drying can be a good alternative for solar drying in order to avoid the quality defects in solar drying (Antal, 2015).

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There are limited studies that evaluated the effect different of different drying techniques on the quality traits (sensory, aromatic and flavour profile, microbiological, and physical properties) of *O. syriacum*. It was found that air-drying exhibited more total phenols (TP) and antioxidant activity if compared to vacuum ovens and freeze-drying (Hossain *et al.*, 2010). Hanna Wakim *et al.* (2013) showed that drying temperature had a great effect on chlorophyll and carotenoid contents in *O. syriacum*. Atallah *et al.* (2010) found that *O. syriacum* dried by an air-drying exhibited higher intensity of flavour and aroma than *O. syriacum* dried by the oven and freeze-drying. The same authors showed that there was more loss in carvacrol during the oven and freeze-drying than in air drying. The effect of different drying techniques (solar, shade, and forced air hot artificial air drying) on the quality traits of two types of Lebanese *O. syriacum* L has been investigated. The study showed that there was a higher loss in chlorophyll content at high temperature (45°C) if compared to low temperature, similar results were also found for thymol and carvacrol loss (Hanna Wakim *et al.*, 2013).

Za'atar (*O. syriacum*) sector in Palestine is considered a very important part of the agricultural business. It is a promising and growing sector even there are several challenges. The planted area with za'atar (*O. syriacum*) in Palestine is more than 6000 dunams. About 85% of produced za'atar is locally sold while the remaining part is exported (The Palestinian Za'atar Sector, 2017). There is a need to improve drying conditions in order to expand the export market.

Drying conditions (temperature, relative humidity, drying time, and composition of food) affects greatly on the quality of dried products (Fudholi *et al.*, 2010). Increasing the drying temperature accelerated the chemical and physical deterioration reactions. Freeze-drying is a non-thermal method based on the sublimation process. Freeze drying had a mild adverse effect on the quality of food if compared to thermal drying methods (Erbay and Icier, 2010). Accordingly, it is usually employed to dry delicate biological and highly heat-sensitive products (Chakraborty and Dey, 2016). Moreover, there is a very limited loss in flavour, aroma, and vitamins (Antal, 2015).

Electronic nose (e-nose) is a chemical sensor that mimics the olfaction system. It has been used for many applications. It has several advantages such as fast, fewer prices than other analytical devices, ease to use and small size.

Metal oxide semiconductors (MOS) are the most common materials that e-noses are made of, they are sensitive and have a long life (Oates *et al.,* 2020). E-nose signals are complex, and for revealing them they are needed to be analysed using multivariate data analysis (MVDA) (Qneibi *et al.*, 2018; Abu-Khalaf *et al.*, 2018, Abu-Khalaf *et al.*, 2019; Al Ramahi *et al.*, 2019; Abu-Khalaf and Abu Rumaila, 2020).

The goal of this research is to investigate the possibility of using e-nose and chromameter as a tool to distinguish between three groups of oregano that were dried in three different methods, air-dried, freeze-dried and solar-dried.

2. Materials and methods

2.1 Sample selection and preparation

Za'atar (*O. syriacum*) was harvested as stems from a field near Tulkarm city, Palestine. The leaves were separated manually from stems. Any abnormal leaves (aged or infected leaves) were excluded from the sample. About 1600 g of fresh green leaves of *O. syriacum* were obtained. The obtained quantity was divided into three treatments (400 g/treatment). In the first treatment, *O. syriacum* leaves were dried by the solar method. In the second treatment, *O. syriacum* leaves were dried for one day by freeze dryer (Millrock) at temperature -80°C and pressure 50 – 200 mbar. In the third treatment, *O. syriacum* leaves were dried by air oven (Blnder, Germany) at 105°C for 16 hrs. All samples were dried until moisture content was about 15%.

2.2 Quality traits measurement

2.2.1 Colour index measurement

Colour index (L*a*b*) was measured by Minolta Chroma Meter CR-400 reflectance colorimeter the instrument was calibrated by a white ceramic tile reference at (Y = 93.9, x = 0.3130 and y = 0.3190). From each treatment, 5 samples were selected to measure colour index. Each sample was filled in a transparent plastic dish with a lid. The colour index was measured in triplicates, the results were expressed as mean values for three measurements.

2.2.2 Sensory analysis

Sensory traits were evaluated by a consumer test. More than 41 assessors (age: 18-25 years) were involved in the sensory analysis Table 1. For each assessor, a plastic dish containing two samples (one freeze-dried leaf and one solar-dried leaf) was offered to carry out a sensory analysis. The sensory analysis was conducted in a laboratory equipped with booths at the Agriculture and Veterinary Medicine Faculty at An-Najah National University in Tulkarm.

The assessor tasks were to evaluate through hedonic scale different sensory traits using 10 points-scale (0 =

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Table. 1 Age distribution of assessors that conducted the sensory analysis

Mean	N	Std. Deviation	Std. Error of Mean	Minimum	Maximum	Range
20.361	40	3.0961	0.3649	18	32	14

disliked extremely/weak, 5 = neither liked nor disliked/ neutral, and 10 = liked extremely). The sensory traits employed in the analysis were colour (greenness, yellowness, and acceptance), odour (dried grass, woody, and acceptance), freshness, taste acceptance, earthy aroma, flavour acceptance, and overall acceptance.

2.2.3 Electric noise analysis

Eight metal oxide semiconductors (MOS) (MQ 2-9 and MQ 135) (Hanwei Electronics Co., Ltd., China) was used for building a prototype of e-nose. This type of sensor was used in several applications (Oates *et al.,* 2020). Three groups of oregano (air-dried, freeze-dried and solar-dried) were measured. About 50 g of five samples from each group were randomly analysed using e-nose. The time for each measurement was about 5 mins.

2.2.4 Statistical analysis

The Unscrambler program (version 9.7, CAMO Software AS, Oslo, Norway) was used to make a principal component analysis (PCA) to investigate the possibility of classifying three dried groups of oregano. The differences in colour index and sensory traits between treatments were determined by a one-way ANOVA test. Tukey's test was used and ($p \leq 0.05$) was considered significant.

3. Results and discussion

The coefficients of variation of signals for measured samples were between 0-7.8%, 0-8.7% and 0-6.8% for air-dried, freeze-dried and solar-dried oregano, respectively. Figure 1 shows the scores plot of principal component scores of e-nose's signals of the three groups. Two principal components (PCs) explained 87% of the total variation of the data.

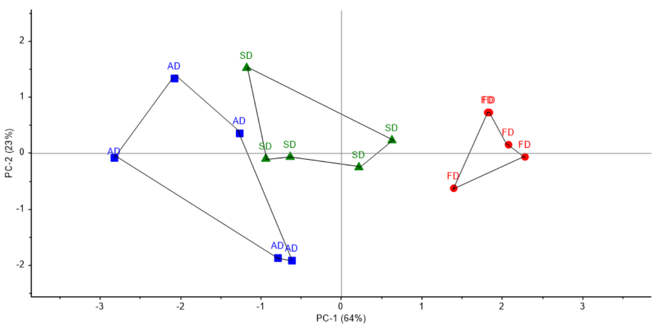

Figure 1. Scores plot of the three dried groups of thyme (AD: air-dried, SD: solar-dried and FD: freeze-dried). Two principal components explained 87% of the total variation. Clear grouping between groups is shown.

There was a clear grouping for the measured samples. The relation between the groups themselves is also clear in Figure 1. The air-dried samples are closer to solar-dried samples than freeze-dried samples. However, freeze-dried is closer to solar-dried than air-dried samples.

The results can help in quality control for following up the drying process or for classifying new samples and to identify their groups. The results further support the idea of using e-noses with thyme. E-noses were successfully used for distinguishing between different thyme cultivars (Vouillamoz *et al.*, 2009) and for evaluating the effect of thyme oil on bacterial activity on textile (Asadi Fard *et al.,* 2018).

The results of the colour index are shown in Figure 2 for lightness (L*), Figure 3 for redness index (a*), and Figure 4 for yellowness index (b*). Solar dried oregano exhibited significantly higher L*-values than freeze and air-dried oregano. Moreover, air-dried oregano had a significantly higher L*-value than freeze-dried oregano. Air-dried oregano exhibited the highest a*-values if compared to solar and freeze-dried oregano. Besides, freeze-dried oregano exhibited significantly lower a*-values than solar-dried oregano. For the yellowness index (b*), solar-dried oregano had significantly higher b*-values than freeze-dried oregano, while there was a moderate difference between air and solar-dried oregano. Generally, the most differences in the colour index can be attributed due to the breakdown of chlorophyll and anthocyanin (Rayaguru and Routray, 2010). The differences in lightness index (L*) between air/solar and freeze-dried oregano can be attributed due to the thermal

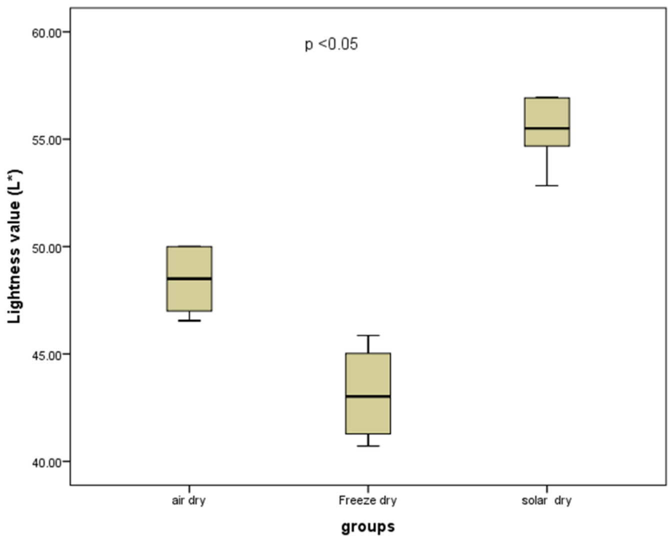

Figure 2. Lightness index (L*) for air, freeze, and solar dried oregano. $p<0.05$ indicates that there were significant differences. Different letters indicate significant differences ($p<0.05$).

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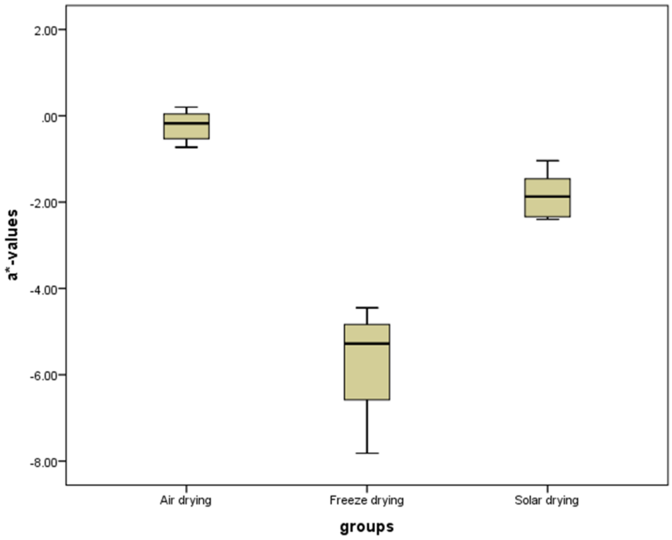

Figure 3. Redness index (a*) for air, freeze, and solar dried thyme. p<0.05 indicates that there were significant differences. Different letters indicate significant differences (p<0.05).

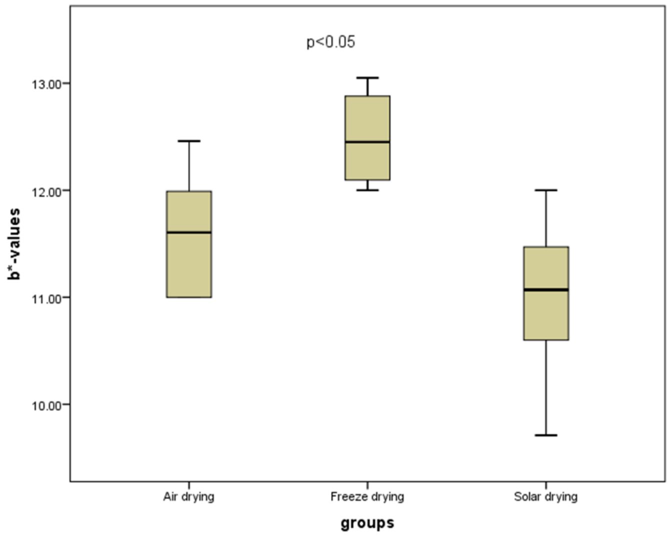

Figure 4. Yellowness index (b*) for air, freeze, and solar dried oregano. p<0.05 indicates that there were significant differences. Different letters indicate significant differences (p<0.05).

effect; it documented that thermal drying increased lightness (Doymaz *et al.*, 2006). In this context, it was found that freeze-dried oregano had higher L*-values than solar dried. Zhang *et al.* (2015) found that increasing temperature has led to an increase in L* and a*- values. When a*-value is low, this indicates a more green colour. This fact can explain why freeze-dried thyme exhibited lower a*-values than solar-dried oregano. Lafeuille *et al.* (2014) found that the freeze-drying technique was better in the preservation of chlorophyll content than solar drying. Pheophytinisation was considered one of the main contributing factors in the loss of chlorophyll (Hanna Wakim *et al.*, 2012).

The result of the sensory analysis is shown in Table 2. Freeze-dried oregano exhibited significantly lower values of greenness preferences (5.18 *vs.* 6.46, p<0.05), colon acceptance (4.80 *vs.* 7.57, p<0.05), degree of freshness (5.57 *vs.* 7.14, p<0.05), odour acceptance (5.83 *vs.* 7.54, p<0.05), taste acceptance (5.46 *vs.* 6.75, p<0.05), and overall acceptance (5.75 *vs.* 7.19, p<0.05) than solar-dried oregano, respectively. On another hand, there were no significant differences in yellowness preference, dried grass odour, woody odour, earthy aroma, and flavour acceptance between solar dried and freeze-dried oregano. Overall results indicated that even freeze-drying is considered a novel technology if compared to solar, but the sensory perception was lowered. Education of consumers about the added value behind this technology and associated image can improve the acceptance of the product (Sajdakowska *et al.*, 2018). The high sensory perception of solar-dried oregano may be attributed to different factors. One of the most important factors is the induction of Millard reactions by solar heat. Maillard reactions produce several compounds that are a precursor for many aromas and flavours (Cardelle-Cobas *et al.*, 2005). Generally, our findings were in agreement with previous results. In this context, it was found that freeze dried-oregano exhibited a higher score of sensory traits if compared to vacuum–microwave dried oregano (Sárosi *et al.*, 2013; Calin-Sanchez *et al.*, 2013). On another hand, freeze-dried oregano exhibited a lower score of aroma and

Table 2. Sensory analysis using 10 point-hedonic scale for freeze and solar dried thyme

Sensory traits	Freeze-dried thyme	Solar-dried thyme	P-value*
	Mean ±SEM	Mean ±SEM	
Greenness preference	5.18±1.99	6.46±2.42	<0.05
Yellowness preference	4.66±2.48	4.22±2.33	0.42
Colour acceptance	4.80±2.30	7.57±1.91	<0.05
Dried grass odour	5.55±2.87	5.50±2.77	0.94
Degree of freshness	5.57±2.35	7.14± 2.39	<0.05
Woody odour	4.05±2.89	3.4±2.16	0.25
Earthy aroma	4.98±2.79	3.91±2.69	0.08
Odour acceptance	5.83±2.38	7.54±2.13	<0.05
flavour acceptance	5.44±2.55	6.44±2.39	0.07
Taste acceptance	5.46±2.82	6.75±2.64	<0.05
Overall acceptance	5.75±2.69	7.19±2.49	<0.05

Values are presented as mean±standard error of mean. *Significant differences are considered when p<0.05.

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flavour (characterized by earthy and musty flavour) when compared with other drying techniques (Sárosi *et al.*, 2013).

4. Conclusion

In conclusion, e-nose was an effective tool to discriminate between air, solar, and freeze-dried oregano. It was clear that the differences in sensory attributes between solar and freeze-dried oregano are very sharp. Moreover, it was possible to discriminate between different drying techniques for oregano by using the colour index.

Conflict of interest

The authors declare no conflict of interest.

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