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# A Comparative Study on the Volatile Profile of Virgin Olive Oils (cv. Koroneiki)

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**Abstract**

The aroma of virgin olive oil (VOO) is derived from volatile compounds. We utilized GC/MS to examine these compounds and discovered both quantitative and qualitative variations in the volatile profiles of the VOO samples we analyzed. The differences were attributed to enzymatic processes in the olive fruits before oil extraction or changes during VOO storage. Odor activity values (OAVs) were used to identify the primary volatiles and their impact on the aroma in each VOO. In the extra virgin olive oil (EVOO) sample, the highest OAV was E-2-Hexenal, followed by Hexanol, Hexanal, and Z-3-Hexenyl acetate. Conversely, Z-3-hexenyl acetate, E-2-nonenal, E-2-decenal, Nonanoic, propanoic, and Butanoic acids were responsible for the fusty VOO sample. The main contributors to the rancid defect were E-2-decenal, nonanal, hexanal, and E-2-nonenal. While, E-2-decenal, butanoic acid, acetic acid, and nonanal were the primary volatiles responsible for the vinegary defect. Lastly, the findings indicated that hexanal, 2,4-heptadienal, and nonanoic acids were responsible for the musty defect.

***Keywords:*** Virgin olive oils, Koroneiki, EVOO, Fusty, musty, vinegary, rancid, volatiles

## 1. Introduction

Extra virgin olive oil (EVOO) is obtained from fully ripened fruits of the Olea europaea L. tree using appropriate extraction methods. It has a unique flavor that sets it apart from other vegetable oils and is valued for its nutritional, sensory, and health properties. EVOO is widely used around the world for various purposes. The olives used to make EVOO produce distinct volatile components that contribute to its highly prized taste and aroma (Tomé-Rodríguez et al., 2021). Sensorial analysis is primarily used to classify grades of olive oils, such as extra virgin olive oil (EVOO), virgin olive oil (VOO), ordinary virgin olive oil (OVOO), or lampante virgin olive oil (LVOO), based on sensory attributes (positive and negative) (Frangipane et al., 2023). The volatile components play a crucial role in determining the sensory properties of VOOs and are essential for evaluating the overall quality of the oil and meeting consumer preferences. These compounds are closely linked to the sensory perception of olive oil. For example, C6 aldehydes and alcohols contribute to the freshness and aroma of EVOO, while esters are responsible for its fruity smell. Conversely, carboxylic acids are associated with pungency and sour perceptions, and are linked to negative attributes in VOOs, such as rancidity (Genovese et al., 2021).

Virgin olive oils grades contain approximately 180 different compounds, including aldehydes, alcohols, esters, ethers, ketones, and hydrocarbons. The positive and negative flavors of these oils are the result of various mechanisms, such as enzymatic processes involving polyunsaturated fatty acids through the Lipoxygenases (LOX) pathway, sugar fermentation or amino acid transformation, enzymatic activity of molds and fungi, or the autoxidation process (Žaneti´c et al., 2021). The sensory

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assessment of VOO is standardized by EU regulation and IOC standards. Trained panelists evaluate sensory attributes, which are categorized into positive and negative attributes. The defects mainly include musty, fusty, muddy sediment, vinegary, and rancid. These defects are primarily described by volatile components, which are the result of pathways involving living organisms in olives, over-ripening fruit, fatty acid oxidation, and infection by fungi and bacteria. The volatile components of the vinegary defect are generated by acetic acid bacteria, which develop during olive storage. The main substances of this defect are acetic acid and ethyl acetate. Furthermore, the rancidity defect is related to the oxidation of unsaturated fatty acids, which produce peroxides. Additionally, the fusty defect develops when olive fruits are left in piles for a long time. It is explained by the content of acids, alcohols, and aldehydes, such as propionic, butanoic, pentanoic, and hexanoic acids (Cecchi et al., 2021). The sensory defects in VOO are linked to the volatile composition of VOOs and are associated with oxidation, whether chemical or enzymatic. Chemical oxidation creates off-flavor compounds such as pent-2-enal and hept-2-enal. These off-flavor volatiles are responsible for unpleasant flavors like fusty, musty, winey-vinegary, rancid, and muddy sediments (Neugebauer et al., 2021). Musty is a specific flavor found in oil extracted from olives that have been attacked by molds, fungi, and yeasts due to improper storage. It is associated with the presence of alcohols, ketones, C8 volatile components, and short-chain fatty acids (Neugebauer et al., 2021). Fusty oil is a specific type of oil extracted from olives that have been stored in piles and bags and then subjected to advanced anaerobic fermentation. This process results in the presence of ethyl butanoate, propanoic, and butanoic acids. Additionally, winey and vinegary defects are related to olive fermentation and the presence of acetic acid, ethanol, ethyl acetate, and 3-methylbutan-1-ol. Rancid oil is the result of oil oxidation, characterized by the absence of C6 aldehydes, alcohols, and esters, and the presence of numerous aldehydes with a low odor threshold (Neugebauer et al., 2021). The presence of pent-1-en-3-one is positively correlated with bitterness and pungent attributes, while hexanal is negatively correlated with these attributes. Z-Hex-3-en-1-ol and E-hex-2-enal are negatively correlated with bitterness and pungency, respectively. Lampante virgin olive oil (LVOO) shows significant changes in its sensory attributes, including a decrease or absence of fruity, bitter, and pungent attributes (Averbuch et al., 2023).

This study aims to identify and differentiate the volatile profiles of extra virgin olive oil (EVOO) and lampante virgin olive oils (LVOOs).

## 2. Materials and methods:

### Materials:

Olive fruits (c.v. Koroneiki) were harvested from Giza-Egypt during season 2021-2022. Fruits were divided into different proportions as mentioned below:

Olive fruits were directly transported to the extraction of VOO (Sample No.1)

Olive fruits were accumulated or stocked in anaerobic conditions to produce a fusty flavor from anaerobic fermentation (Sample No.2).

Olive fruits were stored in wet conditions and developed more fungi and yeasts to obtain a musty flavor (Sample No.3).

Olive fruits were stored and undergone aerobic fermentation to produce a vinegary flavor (Sample No.4).

VOO has an intense oxidation process to produce a rancid flavor (Sample No.5).

### Olive oils extraction

Virgin olive oil (VOO) was obtained from olive fruits using a two-phase continuous extraction system. Sample No.1 represents VOO extracted from healthy fruits.

### Quality parameters

The free fatty acids (%as oleic acid), peroxide value (meq O2 /kg oil), and UV absorbance at 232 and 270 nm were measured according to IOC methods **(IOC, 2022).**

### Sensory analysis

The sensory analysis of VOOs was conducted following the IOC method **(IOC, 2018).** The assessments were conducted by 8 trained panelists at Food Technology Research Institute, Giza, Egypt.

### Volatile profile analysis

The VOO (5 ml) was heated to 40 °C for 15 minutes in a 10.0 mL flask to create headspace. The vapor phase of components was extracted using solid-phase microextraction with a PDMS/DVD fiber. After 15 minutes, the fiber was injected into a gas chromatograph/mass spectrometer (GCMS-QP2010 plus; Shimadzu, Kyoto, Japan). The injection was made in splitless mode with helium as the carrier gas at a flow rate of 1mL/min. The injector temperature was set at 220°C. A fused-silica capillary column was used for compound separation. The oven temperature was initially set at 35°C for 2 minutes and then ramped up to 250°C at a rate of 5°C/min, and maintained at 250°C for 1 minute. The mass spectrometer was operated with electron ionization (70 eV) and a mass scan range from 40 to 600 Da. The temperatures of the ion source and the GC-MS interface were 200 °C and 230 °C, respectively. Compounds were identified by comparing their mass spectra with GC/MS libraries (**Nunes *et al*., 2013).**

### Odour Activity Values (OAVs)

The OAVs were calculated by dividing the concentrations of compounds by their sensory thresholds. OAVs greater than 1 contribute to the aroma of VOO (**Luna *et al*., 2006**).

### Statistical analysis

Statistical analyses were performed using SPSS version 16.0.

## 3. Results and discussions

### Quality parameters of VOOs

The primary indicator of VOO quality is the level of free fatty acids (FFA), which reflects the health of the fruits, the quality of the oil extraction process, and the conditions of oil storage. FFA values are used as a classification criterion for different grades of VOOs **(Elsorady*et al*., 2015)**. The data in Table 1 shows that Sample No.1 had the lowest FFA content (0.31% as oleic acid) and was classified as extra virgin olive oil (EVOO). This finding was agreed with **Elsorady*et al.* (2017).** In contrast, the other samples had higher FFA values ranging from 3.45 to 4.21% (as oleic acid). This was likely due to the storage conditions of the olive fruits before oil extraction and the conditions of oil oxidation after extraction. These VOOs were classified as lampante virgin olive oils (LVOOs). The FFA contents were consistent with the limits of 0.8% for EVOO and more than 3.3% for LVOO, as established by the International Olive Council **(IOC, 2022).**

**Table (1): quality parameters of VOOs**

| Characteristics | VOOs | | | | |
|---|---|---|---|---|---|
| | Sample No.1 | Sample No.2 | Sample No.3 | Sample No.4 | Sample No.5 |
| FFA % (as Oleic acid) | 0.31±0.01[a] | 3.68±0.03[c] | 4.21±0.06[e] | 3.45±0.06[b] | 3.78±0.07[d] |
| PV (meq $O_2$ / kg oil) | 8.45±0.2 [a] | 21.13±0.28[c] | 21.28±0.06[c] | 20.09±0.41[b] | 24.42±0.11[d] |
| $K_{232}$ | 1.34±0.03[a] | 2.84±0.02[b] | 2.82±0.02[b] | 2.79±0.01[b] | 2.90±0.04[c] |
| $K_{270}$ | 0.05±0.00[a] | 0.35±0.02[b] | 0.34±0.02[b] | 0.34±0.01[b] | 0.40±0.01[c] |

*different superscripts indicate significant differences ($p < 0.05$)

The results showed that VOO No.1 had the lowest peroxide value (PV) of 8.45 meq O2 / kg oil (Table 1), while VOOs No.2, 3, 4, and 5 had higher values, exceeding the established PV limits for virgin oils (20 meq O2 / kg oil) (**Codex, 1999**). The PV is an indicator of the total peroxide content in VOO, with VOO No.5 having the highest content at 24.42 meq O2 / kg oil. Additionally, the ultraviolet region at specific wavelengths of 232 and 270 nm was used to measure conjugated diene and triene in VOO (**Elsorady*et al*. 2015**). For sample No.1, the absorbance at 232 and 270 nm did not exceed the established limits of 2.5 and 0.22, respectively, for EVOO (**IOC**, **2022**), while the other samples exceeded these limits. **Rotondi *et al*. (2021)** reported that factors such as olive fly attacks or improper harvest systems, transport, and storage of olives can have a significant effect on quality parameters.

The sensory analysis revealed that VOO no.1 is free from any defects and is considered to be extra virgin olive oil (EVOO), while the other samples had different defects, such as a fusty flavor in sample no.2, musty in sample no.3, winey in sample no.4, and rancid in sample no.5. The median scores for these defects ranged from 6.25 to 7.25, and the samples were graded as lampante virgin olive oils (LVOOs) (Table 2). Figure 1 shows the sensory profile of EVOO (sample no.1), which has apple, grass, and tomato flavors, as well as bitter and pungent attributes.

**Table (2) Sensory analysis of VOOs**

| Attributes | VOOs | | | | |
|---|---|---|---|---|---|
| | Sample No.1 | Sample No.2 | Sample No.3 | Sample No.4 | Sample No.5 |
| Fruity | 4.75 | ND* | ND | ND | ND |
| Bitter | 3.50 | ND | ND | ND | ND |
| Pungent | 3.00 | ND | ND | ND | ND |
| defects | ND | 7.0 | 6.5 | 6.25 | 7.25 |
| Grades | EVOO | LVOO Fusty | LVOO Musty | LVOO Vinegary | LVOO Rancid |

*ND: Not detected

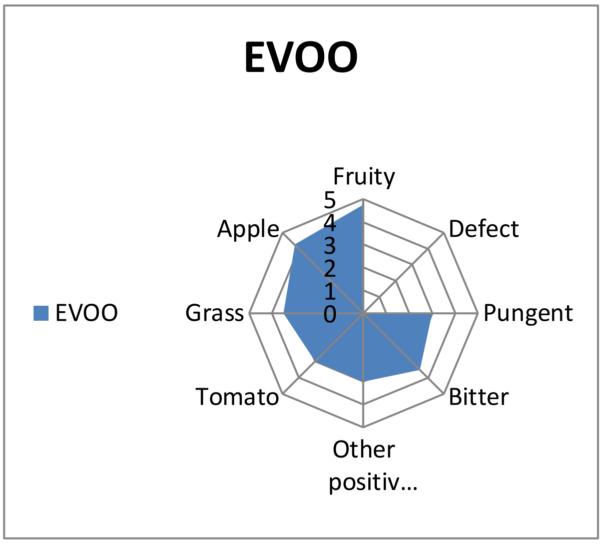


**Figure (1): Sensory profile of Koroneiki cv. EVOO.**

The volatiles profile, 20 compounds from 4 chemical classes (aldehydes, alcohols, ketones and esters) were detected in EVOO cv. Koroneiki (Table 3, Figure 2). Aldehydes were the most abundant chemical class in EVOO followed by alcohols. It was in accordance with **Bajoub*et al.* (2015)** and **Baccouri*et al.* (2008).** Results revealed that E-2-Hexenal is the most abundant component (38.5%), followed by Z-3-hexen-1-ol (13.54%), E-2-hexen-1-ol (7.14%), E-3-Hexenal (5.32%),1-Penten-3-one (4.31%), Hexanal (2.98) and 2,4 Hexadienal (2.52%). These results agreed with **Baccouri*et al.* (2008), Peres *et al.* (2013) and HachichaHbaieb*et al.* (2016).**

**Table (3) Volatile profile of EVOO *cv.* Koroneiki**

| Volatile compound | Concentration (%) | Odour threshold in oil* (μL/L) | Odour Activity Values (OAVs) | Aroma sensory descriptor* |
|---|---|---|---|---|
| Butyl acetate | 0.11±0.02 | NA | NA | Fruity, apple. |
| 3-Methyl butylacetate | 1.20±0.03 | NA | NA | Banana, citrus |
| Hexyl acetate | 0.30±0.01 | 0.31623 | 0.948 | Fruity |
| Z-3-Hexenyl acetate | 0.20±0.02 | 0.0018 | 111.111 | Banana, green |
| Pentan-3-one | 1.65±0.03 | NA | NA | Green |
| 1-Penten-3-one | 4.31±0.04 | NA | NA | Spicy |
| 3-Methylbutan-1-al | 0.04±0.00 | NA | NA | Fruity |
| Hexanal | 2.98±0.01 | 0.01380 | 215.942 | Green apple, grass |
| E-2-Hexenal | 38.50±0.02 | 0.03162 | 1122.707 | Bitter almond, green, banana, apple |
| E-3-Hexenal | 5.32±0.04 | NA | NA | Apple, green |
| Z-3-Hexenal | 1.56±0.02 | NA | NA | Apple, green |
| 2,4 Hexadienal | 2.52±0.04 | NA | NA | Fruity, green |

| | | | | |
|---|---|---|---|---|
| 1-Penten-3-ol | 1.10±0.02 | NA | NA | Mouldy |
| 1-Pentanol | 0.11±0.01 | NA | NA | Fruity |
| E-2-Penten-1-ol | 0.75±0.02 | NA | NA | Mushroom |
| Z-2-Penten-1-ol | 1.50±0.04 | NA | NA | Green |
| Hexanol | 16.35±0.05 | 0.04365 | 374.570 | Pungent, green |
| E-2-hexen-1-ol | 7.14±0.03 | NA | NA | Ripe, green soft |
| E-3-hexen-1-ol | 0.82±0.01 | NA | NA | Grass, green |
| Z-3-hexen-1-ol | 13.54±0.08 | NA | NA | Grass, green |

**NA: not Available; * Devos *et al.* (1990) and Morales *et al.* (2005).**

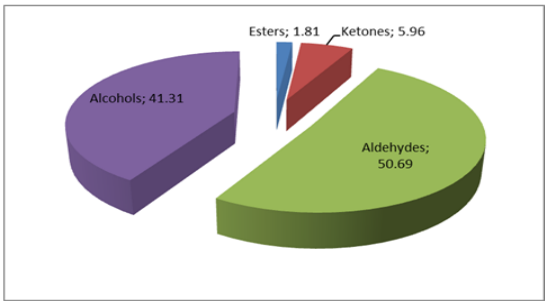


**Figure (2): Chemical classes of volatile profile in EVOO *cv.* Koroneiki**

The main volatile components of extra virgin olive oil (EVOO) are C6 aldehydes, alcohols, and esters, such as hexanal, Z-3-hexenal, E-2-hexenal, hexanol, Z-3-hexenol, E-2-hexenol, hexyl acetate, and Z-3-hexen-1-yl acetate **(Žanetić*et al*., 2021).** According to **Garcia-Oliveira *et al*. (2021),** these components make up about 60-80% of the total fraction. These components, known as green volatiles, are formed through the lipoxygenase (LOX) pathway, where enzymes modify linoleic and linolenic acids to produce aldehydes, alcohols, and esters that are considered crucial for the sensory quality of EVOO **(Tomé-Rodríguez *et al*., 2021).** The fruity, green, and sweet notes of EVOO are attributed to C6-aldehydes **(Tomé-Rodríguez *et al*., 2021)**. The intensity of stimuli evoked by volatile components is generally linked to their quantity. Chemical and external characteristics, such as hydrophobicity, volatility, shape, size, and stereochemistry of volatile molecules, and the position and type of functional groups, as well as matrix effects, have been shown to influence odor contributions more than their concentrations. This is related to the effect of chemical characteristics on taste and smell receptors **(Aprea, 2020).** The odor activity value (OAV) is determined by the ratio between the concentration of a volatile component and its odor threshold. It is considered a useful parameter for identifying the key contributors to the aroma of virgin olive oil (VOO) (**Aprea, 2020).** An OAV greater than 1 indicates a direct contribution of a volatile component to the flavor of VOO (Table 3). The volatile compound with the highest OAV in the extra virgin olive oil (EVOO) sample was E-2-Hexenal (1122.70), followed by Hexanol (374.57), Hexanal (215.94), and Z-3-Hexenyl acetate (111.11). Hexanal provides green apple and green grass sensory notes (**Olmo-Cunillera *et al*., 2022**). The fruity attribute is associated with E-2-hexenal (the most plentiful volatile substance) and Z-3-hexenal, which are related to the lipoxygenase (LOX) pathway. C6 aldehydes are responsible for the green smell **(Žanetić*et al*., 2021).**

Table 4 shows the volatile fractions in a fusty sample of VOO. The components detected in the fusty VOO sample included acids, esters, alcohols, aldehydes, ketones, and alkanes (Fig. 3). The highest fraction of total volatile classes was acids (36.74%), while the lowest fraction was alkanes (0.75%). The fusty defect is caused by bacteria genera such as Escherichia and Aerobacter at the beginning of storage, and Clostridium and Pseudomonas at long-term storage under anaerobic conditions (**Karanth *et al*., 2023).** This storage can produce aldehydes, alcohols, acetic, propionic, butanoic, pentanoic, and hexanoic acids **(Genovese *et al*., 2021).**

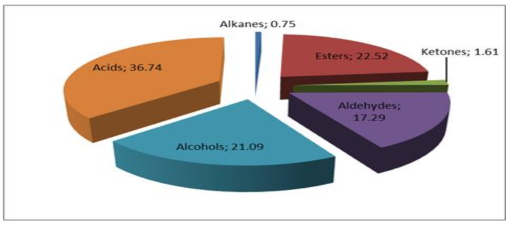


**Figure (3): Chemical classes of volatile profile in fusty VOO sample**

**Table (4) Volatile profile of fusty VOO sample**

| Volatile compound | Concentration (%) | Odour threshold in oil (μL/L)* | Odour Activity Values (OAVs) | Aroma sensory descriptor* |
|---|---|---|---|---|
| Octane | 0.75±0.03 | 5.75440 | 0.13 | Sweet, alkane |
| Ethyl acetate | 3.67±0.09 | 2.63027 | 1.39 | Sticky, sweet |
| Hexyl acetate | 5.25±0.06 | 0.31623 | 16.60 | Fruit, green |
| Z-3-hexenyl acetate | 10.14±0.09 | 0.00180 | 5633.33 | Banana, green |
| Octyl acetate | 3.46±0.03 | 0.00398 | 869.34 | Fruit |
| Butan-2-one | 1.10±0.04 | 7.76247 | 0.14 | Ethereal, fruit |
| 6-methyl-5-heptene-2-one | 0.53±0.02 | 0.05623 | 9.42 | Pungent, oil |
| Hexanal | 7.79±0.07 | 0.01330 | 585.71 | Green apple, grass |
| E-2-Hexenal | 5.53±0.03 | 0.03162 | 174.88 | Bitter almond, green |
| E-2-heptenal | 0.77±0.02 | 0.01349 | 57.07 | Fat, soap, almond |
| Octanal | 0.53±0.04 | 0.00135 | 392.59 | Fat, lemon, soap, green |
| Nonanal | 1.36±0.02 | 0.00224 | 607.14 | Fat, citrus, green |
| E-2-nonenal | 0.30±0.02 | 0.00015 | 2000.00 | Fat, cucumber, green |
| Decanal | 0.29±0.01 | 0.00089 | 325.84 | Vinegary |
| E-2-decenal | 0.72±0.02 | 0.00036 | 2000.00 | Tallow |
| Ethanol | 3.10±0.05 | 28.84032 | 0.10 | Alcohol |
| 2-Methyl-butan-1-ol | 2.15±0.04 | NA | NA | Fusty, vinegary |
| 3-Methyl-butan-1-ol | 2.63±0.03 | 0.45774 | 5.74 | Fusty, vinegary |
| 1-Hexanol | 5.37±0.02 | 0.04365 | 123.02 | Pungent, green |
| 2-hexen-1-ol | 5.13±0.04 | NA | NA | Sticky, balsamic, strong |
| Z-3-hexen-1-ol | 2.71±0.02 | NA | NA | Grass, green |
| Acetic acid | 6.23±0.07 | 0.14454 | 43.10 | Sour, vinegar |
| Propanoic acid | 25.72±0.09 | 0.29512 | 87.15 | Pungent, sour |
| Butanoic acid | 0.35±0.01 | 0.00389 | 89.97 | Fusty, strong, cheese |
| Penatonic acid | 0.26±0.02 | 0.00479 | 54.27 | Putrid, pungent |
| Hexanoic acid | 0.18±0.02 | 0.01259 | 14.29 | Sharp, rancid |
| Heptanoic acid | 0.45±0.02 | NA | NA | Rancid, fat |
| Octanoic acid | 0.25±0.02 | NA | NA | Rancid, fat |
| Nonanoic acid | 3.29±0.03 | 0.00191 | 1722.51 | Rancid, fat |

**NA: Not Available; * Devos *et al.* (1990) and Morales *et al*. (2005).**

Table 4 displays the volatile profile of the fusty VOO sample. It is worth noting that this sample contains a higher concentration of ester components compared to the EVOO sample. This may be attributed to the activity of microorganism enzymes and the fermentation of overripe fruits. Specifically, the concentrations of Z-3-hexenyl acetate, hexyl acetate, and hexanal in the fusty VOO sample were 10.14%, 5.25%, and 7.79%, respectively, which are higher than the concentrations in the EVOO sample, which were 0.2%, 0.3%, and 2.98%, respectively (see Table 3). It is important to note that hexanal can be produced through the LOX pathway and the oxidation of fatty acids **(Hashem*et al*., 2022)**

On the other hand, the concentration of E-2-Hexenal (5.53%) was lower than that detected in the EVOO sample (38.5%). The high concentration of Propanoic acid (25.72%) was related to metabolites of Propionibacterium and Clostridium species **(Morales *et al*., 2005**). According to the OAVs in Table 4, the volatiles of the fusty VOO sample consisted of esters, aldehydes, and acids such as Z-3-hexenyl acetate, E-2-nonenal, E-2-decenal, Nonanoic, propanoic, and Butanoic acids. These results were consistent with those of **Morales *et al*. (2005).** Fusty is associated with the presence of ethyl butanoate, butanoic, and propanoic acids (**Žanetić*et al*., 2021**).

Rancidity is a common defect in VOO and is caused by the oxidation of unsaturated fatty acids. Figure 4 shows the chemical classes of volatile compounds in rancid VOO samples. The highest concentration of compounds was aldehydes, which are produced as a result of the oxidation of unsaturated fatty acids, and then further oxidized to form acids **(Morales *et al*., 2005**).

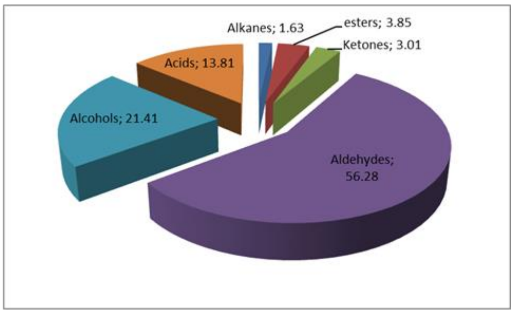


**Figure (4): Chemical classes of volatile profile in rancid VOO sample**

Table 5 displays the volatile profile of a rancid VOO sample. The highest concentration was found in Hexanal (36.52%), followed by 1-penten-3-ol (18.37%), Nonanal (7.54%), and Hexanoic acid (6.46%). The results also indicated that aldehydes were the main contributors to the rancid defect, with E-2-decenal having the highest OAV (4138.88), followed by Nonanal (3366.07), Hexanal (2745.86), and E-2-nonenal (2666.66). These findings are consistent with those of **Morales *et al*. (2005**). **Bendini*et al*. (2009)** also noted that Nonanal, E-2-decenal, and E-2-heptenal are associated with VOO oxidation during storage. Rancidity is the most common negative quality associated with improper storage of oil, leading to severe oxidation of the oils. Oxidation begins after the extraction of virgin olive oil (VOO) and increases with prolonged storage in unsuitable conditions. The levels of C6 aldehydes, particularly Z-2-hexenal, and C6 alcohols decrease, while the levels of C5-C11 aldehydes increase; these aldehydes are mainly responsible for the unpleasant odor (**Yan*et al*., 2020**).

**Nunes *et al*. (2013**) noted a significant increase in the formation of aldehydes, especially nonanal, and carboxylic acids, which had a negative impact on sensory attributes. Oil oxidation is commonly assessed by the formation of nonanal and hexanal**(Grebenteuch*et al*., 2021).**

**Table (5) Volatile profile of rancid VOO sample**

| Volatile compound | Concentration (%) | Odour threshold in oil* (μL/L) | Odour Activity Values (OAVs) | Aroma sensory descriptor* |
|---|---|---|---|---|
| Octane | 1.63±0.04 | 5.75440 | 0.28 | Sweet, alkane |
| Ethyl acetate | 3.85±0.03 | 2.63027 | 1.46 | Sticky, sweet |
| Butan-2-one | 0.88±0.02 | 7.76247 | 0.11 | Ethereal, fruit |
| 2-methyl-5-hepten-2-one | 2.13±0.02 | 0.05623 | 37.88 | Pungent, oil |
| Propanal | 3.10±0.04 | 0.02692 | 115.15 | Apple, green |
| E-2-pentenal | 0.65±0.02 | NA | NA | Green, apple |
| Hexanal | 36.52±0.15 | 0.01330 | 2745.86 | Green apple, grass |
| E-2-Hexenal | 1.52±0.02 | 0.03162 | 48.07 | Bitter almond, green |
| 4-heptenal | 1.65±0.05 | 0.01349 | 122.31 | Cream, biscute |
| E-2-heptenal | 1.05±0.03 | 0.01349 | 77.83 | Fat, soap, almond |
| E-2-octenal | 1.91±0.06 | 0.00204 | 936.27 | Fat, nut, green |
| Nonanal | 7.54±0.01 | 0.00224 | 3366.07 | Fat, citrus, green |
| E-2- | 0.40±0.02 | 0.00015 | 2666.66 | Fat, cucumber, |

| | | | | |
|---|---|---|---|---|
| nonenal | | | | green |
| Decanal | 0.45±0.04 | 0.00089 | 505.61 | Tallow, soap, orange peel |
| E-2-decenal | 1.49±0.01 | 0.00036 | 4138.88 | Tallow |
| 1-penten-3-ol | 18.37±0.12 | 0.40738 | 45.09 | Green, grass |
| 2-hexen-1-ol | 0.89±0.02 | NA | NA | Sticky, balsamic, strong |
| Z-3-hexen-1-ol | 0.39±0.02 | NA | NA | Grass, green |
| 2-octenol | 0.81±0.02 | 0.00302 | 268.21 | Soap, plastic |
| Nonanol | 0.97±0.02 | NA | NA | Fat |
| Acetic acid | 3.66±0.08 | 0.14454 | 25.32 | Sour, vinegar |
| Butanoic acid | 1.92±0.03 | 0.00389 | 493.57 | Fusty, strong, cheese |
| Hexanoic acid | 6.46±0.60 | 0.01259 | 513.10 | Sharp, rancid |
| Heptanoic acid | 1.43±0.02 | NA | NA | Rancid, fat |

**NA: Not Available; * Devos *et al.* (1990) and Morales *et al*. (2005).**

Figure (5) shows the chemical classes of the volatile profile in the vinegary VOO sample. The main class is acids (81.01%). This defect is related to the fermentation process by lactic acid (Lactobacillus) and acetic acid bacteria, which have been detected on olives (**Morales *et al*., 2005**). Acetic acid is the main volatile responsible for this defect (Table 6). The highest OAVs were 750.00, 650.38, 514.80, and 406.25 for E-2-decenal, butanoic acid, acetic acid, and nonanal, respectively.

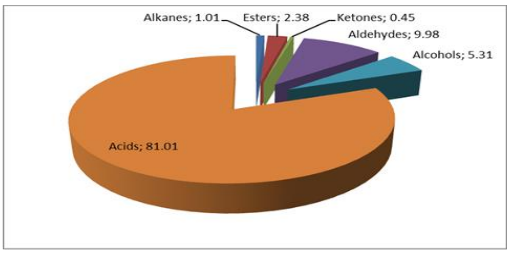


**Figure (5): Chemical classes of volatile profile in vinegary VOO sample**

**Table (6): Volatile profile of vinegary VOO sample**

| Volatile compound | Concentration (%) | Odour threshold in oil* (µL/L) | Odour Activity Values (OAVs) | Aroma sensory descriptor* |
|---|---|---|---|---|
| Octane | 1.01±0.03 | 5.75440 | 0.17 | Sweet, alkane |
| Ethyl acetate | 2.38±0.04 | 2.63027 | 0.90 | Sticky, sweet |
| 6-methyl-5-heptene-2-one | 0.45±0.02 | 0.05623 | 8.00 | Pungent, oil |
| Hexanal | 2.10±0.06 | 0.01330 | 157.89 | Green apple, grass |
| E-2-Hexenal | 6.10±0.15 | 0.03162 | 192.91 | Bitter almond, green |
| E-2-octenal | 0.36±0.02 | 0.00204 | 176.47 | Fat, green, nut |
| Nonanal | 0.91±0.01 | 0.00224 | 406.25 | Fat, citrus, green |
| Decanal | 0.24±0.02 | 0.00089 | 269.66 | Vinegary |
| E-2-decenal | 0.27±0.03 | 0.00036 | 750.00 | Tallow |
| Ethanol | 0.25±0.02 | 28.84032 | 0.008 | Alcohol |
| 2-Methyl-butan-1-ol | 1.62±0.01 | NA | NA | Spice, wine |
| 3-Methyl-butan-1-ol | 1.90±0.05 | 0.45774 | 4.15 | Wood, sweet |
| 1-hexanol | 0.83±0.02 | 0.04365 | 19.01 | Pungent, green |
| Z-3-hexen-1-ol | 0.71±0.03 | NA | NA | Grass, green |
| Acetic acid | 74.41±0.64 | 0.14454 | 514.80 | Sour, vinegar |
| Propanoic acid | 0.31±0.02 | 0.29512 | 1.05 | Pungent, sour |

| | | | | |
|---|---|---|---|---|
| Butanoic acid | 2.53±0.07 | 0.00389 | 650.38 | Fusty, strong, rancid, cheese |
| Penatonic acid | 1.07±0.01 | 0.00479 | 223.38 | Putrid, unpleasant, pungent |
| Hexanoic acid | 2.73±0.05 | 0.01259 | 216.83 | Pungent, rancid |
| Nonanoic acid | 0.15±0.02 | 0.00191 | 78.53 | Rancid, fat |

**NA: Not Available; * Devos *et al.* (1990) and Morales *et al.* (2005).**

Figure 6 illustrates the chemical classes of volatile compounds in a musty VOO sample. Aldehydes make up the majority of the classes, accounting for approximately 58%. Table 7 presents the concentrations of detectable volatiles in the musty VOO sample, with a notably high concentration of hexanal at 41.18% and the highest Odor Activity Value (OAV) of 3096.24. This could be attributed to the enzymatic action of fungi during the lipoxygenase (LOX) pathway (**Morales *et al*., 2005**). C5 alcohols, such as 1-penten-3-ol and 2-methyl-1-butanol, are associated with rancid and musty defects **(Cecchi *et al*., 2021).**

The musty flavor is correlated with the presence of C8 volatiles, such as oct-1-en-3-ol and oct-1-en-3-one (**Morales *et al*., 2005**), which is induced by infected olive fruits with fungi and yeasts due to improper storage conditions.

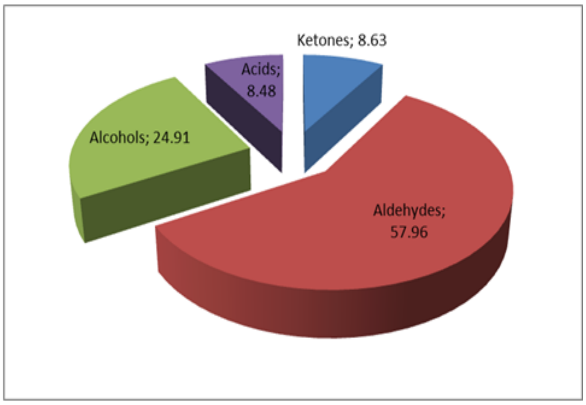


**Figure (6): Chemical classes of volatile profile in musty VOO sample**

**Table (7): Volatile profile of musty VOO sample**

| Volatile compound | Concentration (%) | Odour threshold in oil* (µL/L) | Odour Activity Values (OAVs) | Aroma sensory descriptor* |
|---|---|---|---|---|
| 6-methyl-5-heptene-2-one | 4.65±0.08 | 0.05623 | 82.69 | Pungent, oil |
| 1-octen-3-one | 2.66±0.04 | **NA** | NA | Mould, pungent |
| 2-nonanone | 1.30±0.23 | **NA** | NA | Green, weedy, earthy |
| Pentenal | 5.22±0.06 | NA | NA | Pungent, green, apple, tomato |
| Hexanal | 41.18±0.14 | 0.01330 | 3096.24 | Green apple, grass |
| E-2-Hexenal | 3.09±0.07 | 0.03162 | 97.72 | Bitter almond, green |
| E-2-heptenal | 6.04±0.05 | 0.01349 | 447.73 | Fat, soap, almond |
| 2,4 Heptadienal | 1.02±0.02 | 0.00055 | 1854.54 | Nutty, fatty |
| Nonanal | 1.41±0.05 | **0.00224** | **629.46** | Fatty, waxy, pungent |
| 2-Methyl-butan-1-ol | 1.30±0.05 | NA | NA | Fusty, vinegary |
| 3-Methyl-butan-1-ol | 7.57±0.10 | 0.45774 | 16.53 | Fusty, vinegary |
| 2-Methyl-5-hepten-3-ol | 1.31±0.03 | NA | NA | Herbs, pungent |
| 1 Penten-3-ol | 4.90±0.04 | 0.40738 | 12.02 | Wet earth-vegetable water |

| | | | | |
|---|---|---|---|---|
| Heptan-2-ol | 2.31±0.06 | NA | NA | Earthy, sweet |
| Octan-2-ol | 0.42±0.03 | 0.00417 | 100.71 | Earthy, fatty |
| 1-octen-3-ol | 4.55±0.08 | NA | NA | Earthy, mould |
| Nonan-1-ol | 2.53±0.09 | **NA** | NA | Dusty, wet, oily, ethereal |
| Propanoic acid | 2.23±0.04 | 0.29512 | 7.55 | Pungent, sour |
| Butanoic acid | 1.86±0.04 | 0.00389 | 478.14 | Fusty, strong, cheese |
| Penatonic acid | 1.23±0.05 | 0.00479 | 256.78 | Putrid, pungent |
| Hexanoic acid | 1.26±0.03 | 0.01259 | 100.07 | Sharp, rancid |
| Heptanoic acid | 0.19±0.02 | NA | NA | Rancid, fat |
| Octanoic acid | 0.32±0.01 | NA | NA | Rancid, fat |
| Nonanoic acid | 1.38±0.02 | 0.00191 | 722.51 | Cheese, Fatty, Waxy |

**NA: Not Available; * Devos *et al.* (1990) and Morales *et al*. (2005).**

Finally, the volatile profiles of EVOO and LVOOs samples are characterized by sensory attributes that are related to the perceptions of fruity or off-flavor attributes. The detected volatile compounds are responsible for the perception of both positive and negative attributes.

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