# Towards odorless cosmetic ingredients: determination of fatty esters odor origin

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## Abstract

**Background**: The cosmetic industry is increasingly demanding towards the cosmetic ingredients used in formulations. Fatty esters are no exception; on top of their performance as emollients, emulsifiers, solubilizers, or dispersing agents, they are also expected to present the lowest possible olfactive intensity. This work aims to study volatile odorant compounds released by selected fatty esters to determine their odor origin.

**Methods**: Fatty ester samples underwent sensory analyses to assess their odor intensity thanks to an n-butanol in water reference intensity scale and their odor profile with the Langage des Nez® method. Volatile compounds were also extracted from their headspace by Solid-Phase MicroExtraction (SPME) and analyzed by Gas Chromatography coupled to Mass Spectrometry (GC-MS) to be identified and semi-quantified. Furthermore, Gas Chromatography coupled to Mass Spectrometry and Olfactometry (GC-MS-O) analyses were carried out on fatty esters to put forward odor-active compounds amongst previously detected volatiles.

**Results**: A substantial number of volatile compounds was put forward by SPME-GC-MS analyses: it could be as high as around 70 released volatiles found in trace amounts in fatty ester samples. Some of them have been established as odor-active compounds thanks to GC-MS-O analyses.

**Conclusion**: To conclude, this study allowed us to assess the olfactive impact of different fatty esters and to bring to light the odor-active compounds present in trace amounts in the esters. The residual odor origin has been clarified for the greater part. Thus, these results will guide further investigations to improve the fatty ester refining process, especially the deodorization step to develop odorless ingredients.

**Keywords:** Cosmetic ingredients; residual odor; volatile compounds; GC-MS-O

## Introduction

Fatty esters are obtained by the reaction of an acid and an alcohol that present carbon chains of varying lengths. This reaction is associated with the elimination of a water molecule. Fatty esters can be classified into three groups due to their origin: natural ones or those of synthetic and hemi synthetic origins [1]. Three main industrial ways are well-known to synthesize fatty esters: Fisher esterification, transesterification, and interesterification. High temperatures and acid or base catalysis are usually required for ester synthesis.

Fatty esters are commonly used in various fields of application thanks to their versatility; including cosmetics, health, industry and food. Cosmetic ingredients used in formulations have to meet increasingly demanding criteria. On top of fatty ester performance as emollients [2], emulsifiers, solubilizers or dispersing agents, they are expected to be multifunctional, eco-responsible, ethical, and also present the lowest olfactory sensory properties. Indeed, an undesirable odor from an ingredient in the formula could significantly impact the final odor of the cosmetic product.

The industrial synthesis of fatty esters follows several refining steps to take into consideration these specifications: esterification, neutralization, washing, drying, bleaching, filtration, and finally deodorization. This last step of deodorization is decisive to remove volatile compounds and oxidation products [3]. Nevertheless, some fatty esters may still emit a residual odor even after all these refining steps. Therefore, it is essential to establish the origin of the residual odor to improve industrial refining steps subsequently and propose fatty esters with lowest odor.

Volatile compounds of fatty esters have been barely studied in the literature. However, vegetable oils show chemical structural similarities as they are mainly constituted of triglycerides (ie glycerides with three hydroxyl groups esterified with fatty acids) [4]. Fatty acids from vegetable oils correspond in some cases with the ones at the origin of fatty esters. This is especially the case for olive oil and ethyl oleate. Indeed, olive oil is partly composed of oleic, linoleic, and linolenic fatty acids. These fatty acids' auto-oxidation leads to secondary volatile compounds [5]. Free radicals appear due to degradation by oxygen and they react on the double bond of unsaturated acids. This reaction, which is called primary oxidation, induces the generation of hydroperoxides, which decompose into secondary volatile compounds due to their instability. Besides odor-active volatile compounds that cause a sensory defect in olive oil have been determined [6]. The main chemical compounds responsible for odors are aldehydes (pentanal, hexanal, heptanal, octanal, (E)-2-heptenal, nonanal), acids (hexanoic acid), ketones (1-octen-3-one), esters (ethyl acetate) and alkanes (octane) [7].

Volatile compounds released by olive oil can be analyzed with Gas Chromatography techniques to be quantified and/or identified [8]. These compounds can be perceived by olfaction if their concentration exceeds the detection threshold. To extract volatile compounds from olive oil headspace, different sample preparation methods have been considered. The static HeadSpace (SHS) technique is often used and does not involve a preconcentration of the sample [9,10]. However, a preconcentration step is sometimes necessary because volatile compounds are present in trace amounts and are thus under the detection limit of the GC-FID or GC-MS. Dynamic HeadSpace (DHS) [11] and HeadSpace with Solid Phase Micro-Extraction (HS-SPME) [12] are most frequently carried out to study olive oil samples.

Combining chemical and sensory analyses is crucial to understand the odor origin of a sample and especially determine odor-active compounds responsible for its residual odor. Indeed, chemical analysis allows for identifying volatile compounds released by the sample, but not all of them are odorant. Moreover, some odor-active compounds are not detected by chemical analysis because their concentration is below the detection limit of the GC-MS. In some cases, the nose remains a much more effective detector, hence the key role of sensory analysis. To limit the subjectivity of the panelists who assess odors, specific methodologies should be used, such as the Langage des Nez® [13] or the Field of odors [14]. GC-MS-O technique combines chemical and sensory analysis to identify odor-active compounds from diverse samples [15,16]. It has been developed in food flavor analysis [17,18] and it has particularly been implemented for vegetable oils studies [19,20].

Similar studies have not yet been conducted on fatty esters. Nonetheless, different articles consider the overall odor of fatty esters. Odor thresholds (ie the concentration at which point the odorant compound is perceived) of some fatty esters were evaluated in regards to their chemical structures: carbon chain length [21] and ester branching [22] were considered. These studies revealed that an increase in carbon chain length raises the detection threshold whereas branched esters show a higher detection threshold than linear ones considering the same carbon number.

Volatile compounds generated by thermal degradation of stigmasteryl esters were also investigated [23]. Different stigmasteryl esters as well as their original fatty acids and stigmasterol were heated at high temperature to determine the volatile compounds released by their thermal treatment. A substantial number of volatile compounds have been detected. There are mainly aldehydes, ketones, alcohols, and hydrocarbons and it is assumed that they are formed by the same auto-oxidation mechanism as noted for vegetable oils [24,25].

Nevertheless, the studies did not go further. This work aims to bridge the knowledge gap on odorant volatile compounds of fatty esters and understand the mechanism of their formation to optimize deodorization steps. This paper is especially focused on the study of ethyl oleate. This fatty ester is considered as an alternative to cyclopentasiloxane due to its comparable tactile sensory properties. However, while cyclopentasiloxane is an odorless compound, ethyl oleate tends to have a residual odor. Olfactory sensory evaluation, volatile compounds study with SPME-GC-MS, and the use of GC-MS-O led to the identification of odor-active compounds and their contribution to ethyl oleate overall residual odor. Further analyses have been run to compare odor-active compounds of ethyl oleate before and after the deodorization step.

## Materials and Methods

### *Samples*

One commercial fatty ester was considered for this study. It is mainly composed of ethyl oleate (ethyl (Z)-octadec-9-enoate) (≥ 75%). The 25% left of this ester composition corresponds to ethyl palmitate (ethyl hexadecanoate), ethyl stearate (ethyl octadecanoate), and ethyl linoleate (ethyl (9Z,12Z)-octadeca-9,12-dienoate). It is referred to as “ethyl oleate” thereafter. Raw materials of these fatty esters were also studied: olein, stearin, and ethanol (≥ 99%). Olein and stearin are mixtures of plant origin mainly composed of oleic acid ((Z)-octadec-9-enoic acid); and palmitic and stearic acids (hexadecanoic acid and octadecanoic acid), respectively. The ester and its raw materials were supplied by Stéarinerie Dubois (Scoury, France).

N-butanol (≥ 99.9%) was obtained from Honeywell (Seelze, Germany). It was diluted at different concentrations in ultrapure water to get olfactive intensity levels for the reference scale used for sensory analysis.

### *Sensory analysis*

The jury was formed by 15 expert assessors; 12 women and 3 men aged from 22 to 39. The assessment took place in a laboratory dedicated to sensory evaluation, in which panelists were seated in individual booths. Fatty ester samples were designated with a three-digit number and were introduced to each assessor in a different random order on an odorless sniffing paper stick after dipping 1cm in a solution of pure fatty ester.

The odor intensity was assessed with a reference intensity scale of ten levels of concentration of n-butanol in water: from 0 to 9 at respectively 0, $10^{-3}$, $10^{-2}$, $5.10^{-2}$, $10^{-1}$, $5.10^{-1}$, 1, 5, 10 and 50 g/L.

Furthermore, assessors had to define each fatty ester odor with a maximum of five odorant referents from the Langage des Nez® method. This technique can be used to describe objectively all types of odors, after regular training of the panelists. Assessors have to select the odorant referents with the closest odor nature to the studied sample. Langage des Nez® is composed of 45 well-defined pure odorant compounds which have been diluted in an odorless solvent. These referents are grouped into 7 poles: terpenic, sulfurous, phenolic/pyrogenic, aromatic, ester, amine, and alkyl.

Thus, olfactive intensity and profile were evaluated thanks to odorant references to reduce variability and subjectivity among assessors' ratings. An odor profile graph regroups all these data to display referents from Langage des Nez® and its cumulative intensity. This intensity includes the number of times odorant referents have been mentioned (at least two times) by the assessors as well as the intensity assigned to each referent on the n-butanol scale.

### *Headspace study of fatty esters with SPME-GC-MS*

Fatty esters samples were analyzed in triplicate. A MultiPurpose Sampler (MPS, Gerstel) was used to extract volatiles from the samples with an SPME module. 1mL of the sample in a 20 mL glass vial, capped with Silicone/PTFE septum, was heated at 50°C for 30 min until volatile compounds reached an equilibrium in the headspace. An SPME fiber (Divinylbenzene/Carboxen/Polydimethylsiloxane, needle size 23 ga, StableFlex, 2 cm length, 50/30 µm film thickness, Supelco) previously conditioned at 270°C for 30 min was then introduced into the vial headspace for 10 min at 50°C to extract the volatile compounds.

The SPME fiber was then introduced into the injection port of a gas chromatograph GC-2010 Plus (Shimadzu) at 250°C for 5 min in splitless mode to thermally desorb volatile compounds.

The GC device was coupled to a quadripolar mass spectrometer GCMS-QP2010 SE (Shimadzu) equipped with an SLB-5MS column (5% diphenyl/95% dimethylpolysiloxane phase, 30 m x 0.25 mm i.d., 0.25 µm film thickness, Supelco). Helium was used as carrier gas at a constant flow rate of 1 mL/min. The oven temperature was held at 50°C for 5 min and programmed to rise to 250°C at 5°C/min. The GC-MS interface and the MS source temperature were 250°C and 200°C, respectively. The electron impact energy was set at 70 eV and data were collected in the range of 35-350 atomic mass units. Mean relative peak areas were monitored to compare samples. Linear retention indexes (LRI) were calculated for each volatile thanks to a homologous series of C5 to C24 n-alkanes. Volatile compounds were identified by comparing mass spectral data and retention index with those of the NIST MS Search 2.2 database.

### *Determination of odor-active compounds by GC-MS-O*

Samples were automatically prepared with an SPME fiber following the same conditions mentioned above. They were then analyzed using a gas chromatograph GC-2010 Plus (Shimadzu) coupled with a mass spectrometer GCMS-QP2010 SE (Shimadzu) coupled to an Olfactory Detection Port (ODP, Gerstel) with an SLB-5MS column (30 m x 0.25 mm i.d., 0.25 µm film thickness, Supelco). Helium was used as the carrier gas with a constant flow rate of 2 mL/min. The column effluent split was regulated at 1:2 respectively to the mass detector and the ODP. Other chromatographic conditions were the same as those used in simple GC-MS as detailed above.

Three trained panelists assessed twice each sample with Gerstel ODP recorder software. They could monitor the odor-active areas of the eluate as well as the odor intensity on a four-level scale using a remote control. Referents from Langage des Nez were also vocally registered to characterize each perceived odor. These data were plotted into a cumulative olfactogram with Gerstel Olfactory Data Interpreter. It represents the cumulative intensity of the six analyses for each odorant perception. This cumulative intensity includes the number of times an odor has been detected as well as its rated intensity on the four-level scale. An odor-active compound has to have been detected at least twice on the six analyses to be displayed on the cumulative olfactogram.

## Results

### *Sensory analysis*

Assessors evaluated the olfactive intensity of ethyl oleate on the n-butanol reference intensity scale. The mean score of the 15 ratings of panelists reached 7.8 ± 0.9. The odor profile of ethyl oleate was also assessed by referring to odorant compounds from Langage des Nez® and assigning an olfactive intensity to these odorant referents thanks to the n-butanol reference scale (**Figure 1**). Four different poles of Langage des Nez® emerge from this study: alkyl, ester, aromatic and phenolic. Alkyl and ester poles stand out due to the high cumulative intensity of their referents. Diacetyl (creamy, buttery notes) reaches 32 and ethyl isobutyrate (apple, fruity note) 21 in terms of cumulative intensity. The ester odor is thus mainly described by fatty and fruity notes.

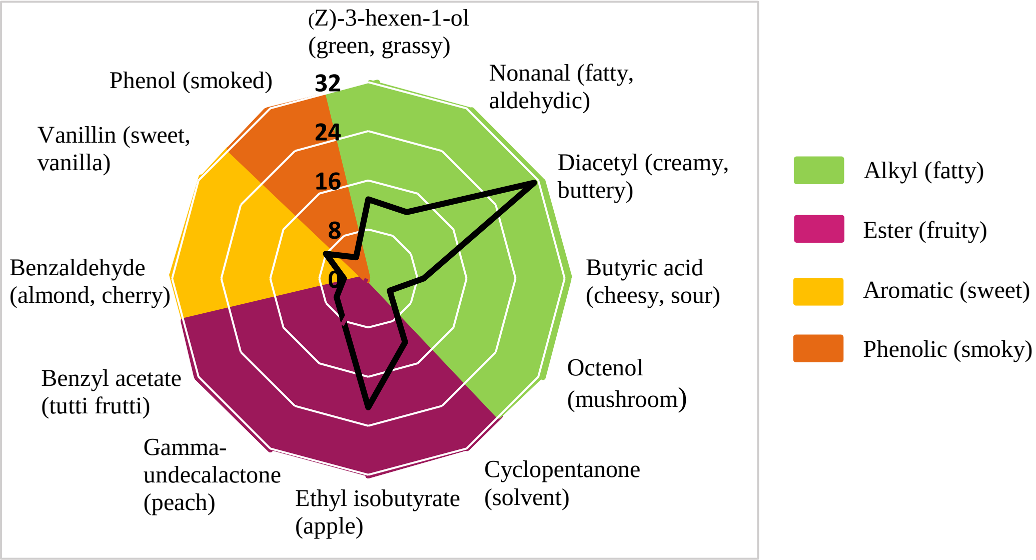

**Figure 1** – Olfactive profile of ethyl oleate with the cumulative intensity of odor referents from Langage des Nez quoted at least two times by assessors

### *Headspace composition of ethyl oleate*

The volatile compounds from ethyl oleate headspace were studied thanks to the SPME extraction technique. They were analyzed after extraction by GC-MS and the generated chromatogram is presented in **Figure 2**. 72 different volatile compounds were put forward and amongst them, 57 have been identified (23 esters, 13 aldehydes, 10 hydrocarbons, 8 ketones, 2 alcohols, and 1 furan). More than 70% of ethyl oleate headspace is constituted of 9 main volatiles (7 esters, 1 alcohol, and 1 aldehyde). The other volatile compounds were semi-quantified below 2.5%.

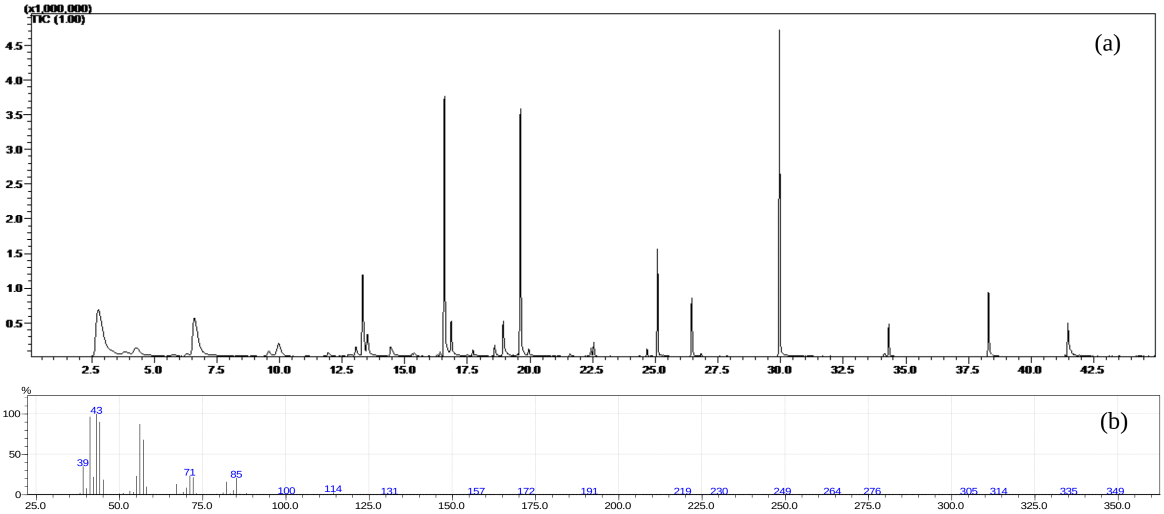

**Figure 2** – Chromatogram of the headspace of ethyl oleate (a) with each chromatographic peak associated with volatile compounds using mass spectra comparison (b)

*Determination of odor-active compounds by GC-MS-O*

The cumulative olfactogram of ethyl oleate is represented in **Figure 3**. Each peak of the olfactogram matches an odor perceived at least two times by the assessors. Olfactive cumulative intensity is assigned to each peak; it includes the number of times the odorant perception was detected by the assessors as well as the intensity they rated. The odor-active area corresponds to the first 25 minutes of the GC analysis, after which the panelists had no odorant perceptions. The assessors perceived on the whole area 23 odorant compounds. LRI comparison made it possible to link some odorant perceptions with chromatographic peaks and their identification was achieved by comparing experimental mass spectra and LRI to the NIST library. Thus, 14 odor-active compounds have been identified; they are predominantly esters as well as saturated and unsaturated aldehydes. The rest of the odorant perceptions do not match with any of the chromatographic peaks, which is frequent for GC-MS-O analysis and could be explained by a low detection threshold.

The Langage des Nez® referents quoted by the panelists to characterize the 23 odorant perceptions are mostly from alkyl (11 referents) and ester poles (7 referents). Furthermore, compounds with the highest cumulative intensities on the olfactogram are designed by alkyl referents. It explains the notes mainly perceived in the overall odor of ethyl oleate as seen in **Figure 1**.

Raw materials of ethyl oleate were analyzed by SPME-GC-MS to investigate the ester residual odor origin. 7 of the 14 identified odor-active compounds were also detected in the raw materials headspace. A part of the odorant volatiles is thus present upstream of the esterification process. As for the others, they seem to appear during the synthesis probably due to side reactions promoted by high temperature.

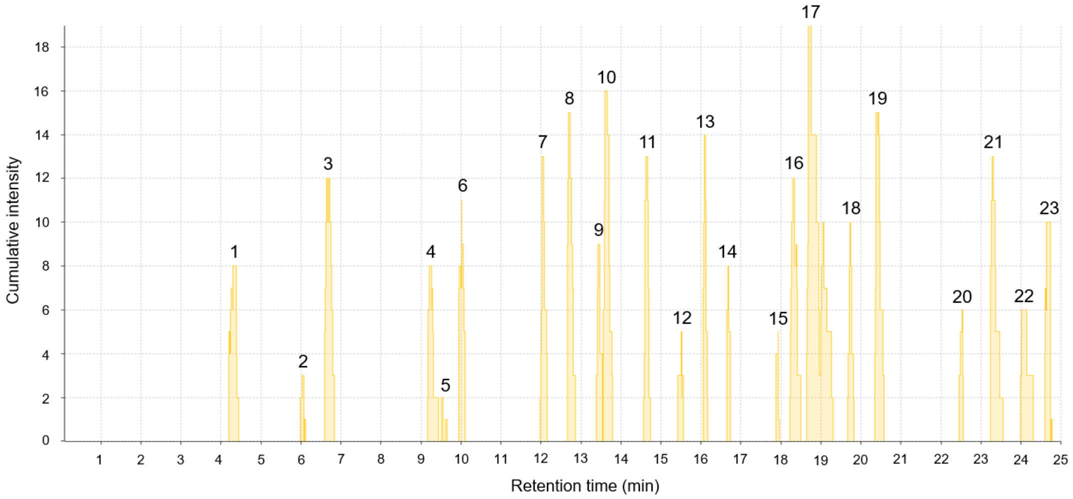

**Figure 3** – Cumulative olfactogram of ethyl oleate (zoom on odorant period) with each odorant perception detected at least two times by assessors

*Impact of deodorization step*

A sample of ethyl oleate underwent deodorization to be compared to non-deodorized ethyl oleate. Volatiles from the deodorized sample were extracted with SPME fiber and then analyzed by GC-MS-O two times by each of the three panelists. The six analyses are summed on the cumulative olfactogram of **Figure 4b)**. After deodorization, the number of odor-active compounds of ethyl oleate plummets down from 23 to 12. This comparison of olfactograms shows the direct impact of the deodorization step on odor-active compounds of ethyl oleate. The deodorization is particularly effective on the most volatile compounds with a retention time lower than 17 min.

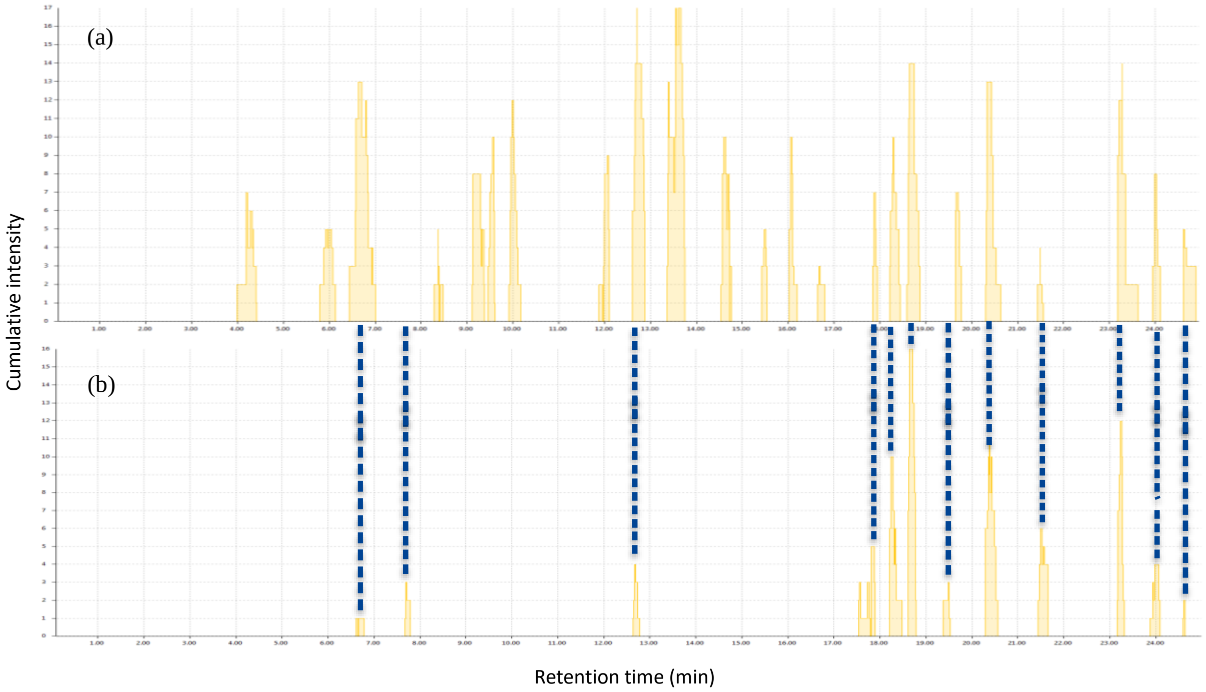

**Figure 4 -** Comparison of cumulative olfactograms of (a) non-deodorized and (b) deodorized ethyl oleate (zoom on odorant period)

## Discussion

Although low olfactive intensity of cosmetic ingredients is becoming a real stake, research on fatty ester odor remains poorly documented in the literature. The present work brought to light 70 different volatile compounds that are released by ethyl oleate. Only 14 of these have been matched with an odorant perception thanks to GC-MS-O analyses. Nevertheless, 9 odorant perceptions could not be assigned to any of the peaks of ethyl oleate chromatogram and thus could not be identified. These results point out the importance of combining chemical and sensory analyses. Comparison of deodorized with non-deodorized ethyl oleate made it possible to follow the removal of odor-active compounds from ethyl oleate headspace as well as their impact on the overall fatty ester residual odor. This comparison was especially crucial to study the odor-active volatiles that have not been detected nor identified by GC-MS analyses because of their low detection threshold. This illustrates that the human nose is still the most effective detector and that sensory analysis plays a key role in this type of study.

## Conclusion

A commercial fatty ester, ethyl oleate, commonly used in cosmetics was studied to determine its residual odor origin. The study combined both sensory and chemical analyses to elucidate its trace odor and identify its odor-active compounds. First, its odor intensity and profile were evaluated by 15 trained assessors thanks to objective methodologies, respectively n-butanol intensity reference scale and Langage des Nez®. GC-MS analyses were performed after volatile compounds extraction from ethyl oleate headspace with SPME fiber. Over 70 volatiles were detected in ethyl oleate headspace; they were mainly esters, aldehydes, hydrocarbons, and ketones. GC-MS-O analyses revealed 23 odor-active compounds, of which 14 have been identified. Odorant compounds of ethyl oleate are mainly esters as well as saturated and unsaturated aldehydes. The study of deodorized ethyl oleate demonstrated deodorization efficiency on odorant compounds with low retention times. The present results will steer further optimizations of fatty ester deodorization by selecting adapted methods to remove remaining odor-active compounds.

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**Conflict of Interest Statement**

NONE

**References**.

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