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# Rapid sensory analysis using response surface methodology: application to the study of odour interactive effects in model spirits

**Pau Matias-Guiu, Eduardo Garza-Moreira, Juan José Rodríguez-Bencomo† and Francisco López***

**ABSTRACT: One of the major objectives of food industry is predicting the sensory profile of a product by chemical analysis. In the case of spirit drinks, their volatile compounds can exhibit odour interactive effects. This study investigated the odour perception of linalool, ethyl hexanoate, ethyl acetate and acetaldehyde with model solutions (in 40% *v/v* ethanol) on flowery, fruity and glue-like attributes. Design of experiments for sensory analysis and response surface methodology were used to evaluate the aroma interactions of these compounds by sniffing or orthonasal olfaction. Results showed that ethyl hexanoate masks flowery and glue-like descriptors. Acetaldehyde provides a low-intensity fruity odour, which is confused with ethyl hexanoate odour. High levels of ethyl acetate suppress and enhance the fruity descriptor at high and low levels of ethyl hexanoate, respectively. In summary, this sensory technique enables the rapid but consistent assessment of the interaction of aroma compounds in an alcoholic spirit. It is suggested that this approach may be a useful tool in the optimization and development of alcoholic products.**


**Keywords:** central composite design; odour mixtures; aroma modelling; aroma interaction

## Introduction

The quality of young Muscat brandy (Pisco) depends on its aromatic composition, where terpenic compounds (e.g. linalool) and low-boiling ethyl esters (e.g. ethyl hexanoate) have a positive odour effect, related to flowery and fruity descriptors, respectively. In contrast, high-volatility compounds such as acetaldehyde and ethyl acetate are considered off-flavours, with pungent and glue-like odour, respectively. To achieve the highest organoleptic quality and production yield, Piscos are usually produced by batch distillation, where the first fraction is discarded (heads) and the following fraction is the main product (hearts). This methodology allows producers to concentrate or avoid certain aroma compounds according to their physicochemical characteristics *(1)*.

The typical aroma of Pisco comes from linalool *(2)*, which is described as flowery and citrus *(1,3)*. This terpene alcohol has a low odour threshold in spirits (1 mg/L of ethanol 40% *v/v*) with respect to its concentration in commercial Piscos (0.17 to 10.4 mg/L 40% *v/v*) *(4)*. Linalool is formed during grape ripening and in wines can be found both free and as the glycoside precursor *(5)*. Owing to its physicochemical characteristics, linalool tends to distil in the early stages of distillation of the heart fraction.

Fruity odour in wine spirits mostly comes from low-boiling ethyl esters ($C_4$–$C_{10}$) with boiling points between 125.8 and 247.7°C *(6)*. Their origin is mostly microbiological, being produced during fermentation by yeasts and other microorganisms. Ethyl esters are highly volatile, and thus tend to distil in the head fraction, with low amounts are found in the product *(7)*. However, ethyl esters have an important aroma impact in Muscat spirits, as they have very low odour thresholds (0.005 to 0.26 mg/L 40% *v/v*) *(1)*. Furthermore, in our study, ethyl hexanoate was chosen as reference ethyl ester, since Peña y Lillo et al. *(8)* found that ethyl hexanoate was the ethyl ester with highest correlation in the volatile composition of the heart fraction. The aroma of ethyl hexanoate is described as apple, banana and violet *(1)*.

One of the most common defects in spirits of agricultural origin is from ethyl acetate, which contributes a glue-like aroma similar to the odour of nail polish remover. Ethyl acetate is produced by yeast and bacterial metabolism or formed through the chemical esterification of ethanol and acetic acid. In spirit drinks, ethanol is presented in very high concentrations (>30% *v/v*); therefore ethyl acetate formation is favoured. According to its high volatility, ethyl acetate distils in head fractions; however, high levels can be found in the heart fraction (4 to 800 mg/L 40% *v/v*) in relation to its odour threshold (7.5 mg/L 40% *v/v*) *(1)*.

Pungent odour is another spirit off-flavour, which comes from acetaldehyde. However, low levels can suggest positive odour in spirits, as sweetish, cut apple or nut notes *(9)*. Acetaldehyde is the most important carbonyl compound formed during the alcoholic fermentation. Since it is highly volatile, acetaldehyde distils

* Correspondence to: Francisco López, Departament d'Enginyeria Química, Facultat d'Enologia, Universitat Rovira I Virgili, Av. Països Catalans 26, 43007, Tarragona, Spain. E-mail: francisco.lopez@urv.cat

† Current address: Institut Charles Gerhardt, Université de Montpellier, Place Eugène Bataillon, Montpellier, France.

Departament d'Enginyeria Química, Facultat d'Enologia, Universitat Rovira I Virgili, Av. Països Catalans 26, 43007, Tarragona, Spain

in the head fraction. Spirits can also present high concentrations in the heart if the head fraction is not well adjusted, this being a typical defect. In commercial products, acetaldehyde presents a wide range of concentrations (<2–160 mg/L 40% *v/v*) *(1)*.

A common way to estimate the odour intensities of volatile compounds is the calculation of odour activity values (OAV; the ratio between chemical concentration and odour threshold), in order to predict the sensory profile of food products by chemical analysis. Several studies have determined olfactory thresholds of many volatile compounds present in spirits; however, these experimental values do not take into account odour interactive effects between compounds, such as enhancement or suppression of odours *(10)*. There are some methods to evaluate aroma interactions, mostly comparing odour intensities in binary mixtures. A much more comprehensive explanation of odour interactive effects can be found in some reviews *(11,12)*.

A few studies have used factorial experiments to evaluate sensory interactive effects in complex mixtures *(13–15)*. However, no study has focused on interactive odour effects on spirits, where ethanol can produce olfactory fatigue *(16)*.

This work was planned with a dual purpose. The first objective was to perform a rapid sensory methodology to analyse a large number of samples of wine distillates using response surface methodology (RSM), especially designed to perform quick evaluation of distillation strategies to obtain products with specific aromatic profile. The second objective was to analyse the aroma interactive effects between volatile compounds in wine spirit drinks using model solutions.

## Materials and methods

### Samples

Ethyl alcohol of agricultural origin was the base spirit (Alcohol Suave, Bodegas y Destilerías Lehman S.A., Tortosa, Spain), with an alcohol degree of 40% (*v/v*). Ethanol content was checked with an electronic density meter (Anton Paar GmbH, Graz, Austria). According to the experimental design (Table 1), 26 aliquots with model solution were spiked at several levels with linalool, ethyl hexanoate, acetaldehyde and ethyl acetate (Sigma-Aldrich, St Louis, MO, USA) 12 h before the experiment. Samples were stored in sealed amber glass bottles at room temperature. Concentration levels were chosen according to the order of magnitude of common levels in commercial spirits *(1)*.

**Table 1.** Coded concentrations (±1) and odour thresholds of the compounds (factors) for the face centered central composite design with two center points (CP)[a]

| Sample number | Linalool | Ethyl hexanoate | Ethyl acetate | Acetaldehyde |
|---|---|---|---|---|
| 1 | −1 (0) | −1 (0) | −1 (0) | −1 (0) |
| 2 | −1 (0) | −1 (0) | −1 (0) | +1 (300) |
| 3 | −1 (0) | −1 (0) | +1 (300) | −1 (0) |
| 4 | −1 (0) | −1 (0) | +1 (300) | +1 (300) |
| 5 | −1 (0) | +1 (10) | −1 (0) | −1 (0) |
| 6 | −1 (0) | +1 (10) | −1 (0) | +1 (300) |
| 7 | −1 (0) | +1 (10) | +1 (300) | −1 (0) |
| 8 | −1 (0) | +1 (10) | +1 (300) | +1 (300) |
| 9 | +1 (10) | −1 (0) | −1 (0) | −1 (0) |
| 10 | +1 (10) | −1 (0) | −1 (0) | +1 (300) |
| 11 | +1 (10) | −1 (0) | +1 (300) | −1 (0) |
| 12 | +1 (10) | −1 (0) | +1 (300) | +1 (300) |
| 13 | +1 (10) | +1 (10) | −1 (0) | −1 (0) |
| 14 | +1 (10) | +1 (10) | −1 (0) | +1 (300) |
| 15 | +1 (10) | +1 (10) | +1 (300) | −1 (0) |
| 16 | +1 (10) | +1 (10) | +1 (300) | +1 (300) |
| 17 | −1 (0) | 0 (5) | 0 (150) | 0 (150) |
| 18 | +1 (10) | 0 (5) | 0 (150) | 0 (150) |
| 19 | 0 (5) | −1 (0) | 0 (150) | 0 (150) |
| 20 | 0 (5) | +1 (10) | 0 (150) | 0 (150) |
| 21 | 0 (5) | 0 (5) | −1 (0) | 0 (150) |
| 22 | 0 (5) | 0 (5) | +1 (300) | 0 (150) |
| 23 | 0 (5) | 0 (5) | 0 (150) | −1 (0) |
| 24 | 0 (5) | 0 (5) | 0 (150) | +1 (300) |
| 25 (CP) | 0 (5) | 0 (5) | 0 (150) | 0 (150) |
| 26 (CP) | 0 (5) | 0 (5) | 0 (150) | 0 (150) |
| Odour threshold (mg/L 40% *v/v*) | 1.00[b] | 0.005[c] | 7.50[c] | 10.0[c] |

[a]Numbers in brackets are the experimental concentrations of factors, expressed in mg/L ethanol 40% *v/v*.
[b]Cacho et al. *(4)*.
[c]Christoph and Bauer-Christoph *(1)*.

### Experimental design

**Central composite design.** Central composite design (CCD) is an experimental design that allows the estimation of linear and quadratic effects on a response variable with a minimum number of experiments. CCD is built with two-level fractional factorial design (FFD) points, a centre point and axial points. Two-level factorial design studies all possible combinations of two or more factors at two levels. The centre point is an experimental run whose factor values are the median of the two levels of the factors establish in the FFD. Centre point is normally replicated to determine the variance of the system. Axial points have the same factor values as centre point, except for one factor whose value is at an $\alpha$ distance from the centre point. For processing data, independent variables (factors) are coded as follows to construct the design: FFD points = ±1; centre point = 0; axial points = $\pm\alpha$ (for one factor) and 0 (for other factors).

For the study, a three-level–four-factor CCD with face-centred axial points ($\alpha = \pm 1$) and two centre points was designed (26 runs). Factors were the selected compounds: linalool, ethyl hexanoate, ethyl acetate and acetaldehyde, spiked in a model spirit. Table 1 shows all of the experiments in the standard order of runs with coded (−1, 0 and +1) and experimental (spiked concentrations) factor values.

**Response surface methodology.** RSM, introduced by Box and Wilson *(17)*, aims to screen, model and optimize an experimental design by studying the relationships between the independent variables (spiked compounds) and each response (odour descriptors). Therefore, after the CCD is performed, RSM estimates a second-degree polynomial model with all of the compiled data.

In this study, ANOVA has been used to observe the significance of linear and quadratic main factor effects and two-way interaction



  

factor effects. Sum-of-squares type III was used to calculate the error terms for statistical signification. Non-significant effects ($p > 0.05$) were ignored to obtain more accurate estimation models. A lack-of-fit test ($p > 0.05$) was performed to check the adequacy of each model. The RSM estimated response for each compound was calculated using eqn (1), a second-degree polynomial function with four variables:

$$\hat{Y} = b_0 + b_1.X_1 + b_2.X_2 + b_3.X_3 + b_4.X_4 + b_{1,1}.{X_1}^2 + b_{2,2}.{X_2}^2 + b_{3,3}.{X_3}^2 + b_{4,4}.{X_4}^2 + b_{1,2}.X_1.X_2 + b_{1,3}.X_1.X_3 + b_{1,4}.X_1.X_4 + b_{2,3}.X_2.X_3 + b_{2,4}.X_2.X_3 + b_{3,4}.X_3.X_4 \quad (1)$$

where $\hat{Y}$ is the estimated sensory response (flowery, fruity or glue-like descriptor), $b_0$ is the regression coefficient for the intercept, $b_1$, $b_2$, $b_3$ and $b_4$ are the coefficients of the main effects, $b_{1,1}$, $b_{2,2}$, $b_{3,3}$ and $b_{4,4}$ are the coefficients of the quadratic effects, and $b_{1,2}$, $b_{1,3}$, $b_{1,4}$, $b_{2,3}$, $b_{2,4}$ and $b_{3,4}$ are the coefficients of two-way interactions. Terms $X_1$, $X_2$, $X_3$ and $X_4$ refer to the coded concentrations of variables (±1). During this study, subscripts stand for the following terms: 0 means intercept; 1 means linalool; 2 means ethyl hexanoate; 3 means ethyl acetate; and 4 means acetaldehyde.

### Rapid Sensory Analysis

The taste panel was composed of 18 third-year students of oenology (with experience in wine tasting) at the Universitat Rovira i Virgili during five sessions on different days of 30–40 min each. All sensory analyses were carried out in the tasting room at the Faculty of Enology of the Universitat Rovira i Virgili in compliance with standard NF V09-105 *(18)*. The first session performed was a training session to determine assessors' sensory capabilities, and during the other four sessions the CCD was completed.

The selected descriptors were named as flowery, fruity and glue-like, which describe the aromas of linalool, ethyl hexanoate and ethyl acetate, respectively. Preliminary studies found that acetaldehyde was not well discriminated by semi-trained assessors (data not shown); however it has been reported that it possesses the ability to change the perception of other compounds *(14)*. For both reasons, it was introduced in the experimental design as a factor, but not as an odour response. Samples were analysed by orthonasal olfaction, since is more discriminant than the retronasal route in Muscat spirits *(19)*.

**Training session.** Assessors were taught to relate the odours of the compounds linalool, ethyl hexanoate and ethyl acetate with the descriptors flowery, fruity or glue-like, respectively, on a six-point scale from 0 to 5. Three samples at different levels of each compound were used as a training scale (nine samples), prepared with the concentrations of the coded factor levels (−1, 0 and +1) of the CCD (Table 1). The −1, 0 and +1 coded concentrations were related to 0, 2 and 4 values on the six-point scale, respectively. The value 0 of the six-point scale was included to differentiate the aroma of ethanol with respect to the aroma of the studied compounds, and the value 5 of the six-point scale was not trained but considered in case of odour enhancement between compounds at high concentrations.

**Design of experiments for sensory analysis.** Spirits contain a large amount of ethanol. Therefore the olfactory capabilities of the assessors may decrease. In preliminary studies, it has been observed that it is not suitable to evaluate more than six samples in a continuous session, a break being needed every three samples (data not shown). However, our CCD consisted of 24 samples plus two centre points, and just 18 assessors.

Design of experiments for sensory analysis ensures acceptable statistical results when a small group of judges evaluate a large set of products. With this methodology, products are analysed with an overall frequency as homogeneous as possible during all of the experimentation and during each session, and with a random order to avoid bias. The design of experiments for sensory analysis used in this study is shown in Table 2, where each sample had eight or nine replicates.

**CCD orthonasal sensory procedure.** In the four sessions, three samples of 5 mL were placed in front of each panellist in transparent glasses covered with empty plastic petri dishes to ensure a homogeneous head-space and to prevent evaporation. Samples were ordered from left to right side according to Table 2. Panellists scored the three descriptors by orthonasal olfaction with the six-point scale.

### Statistical analysis

CCD, ANOVA, lack-of-fit test and regressions models for RSM were performed with STATISTICA 7.0 statistical package. Design of experiments for sensory analysis was performed with XLSTAT 2016 statistical ad-in for Microsoft Office.

## Results and discussion

The purpose of this work was to study the interactions between compounds and their aromas, which were determined by regression models containing main, quadratic and two-way interaction coefficients. To avoid confusion between interaction terms throughout the article, the term 'two-way interaction effect' has been used to describe significant statistical two-way interactions, and the term 'odour-interactive effect' to describe sensory interactions. Therefore, an odour-interactive effect indicates that a compound enhances or reduces the perception of another compound. On the other hand, a two-way interaction effect shows how two compounds produce an odour variation when both are present (which can or cannot produce a sensory interactive effect on the studied odour).

### Regression model

Table 3 shows the regression results of the sensory evaluation of the CCD samples, calculated using eqn (1). As can be seen, panellists could identify and match the three descriptors with their respective trained compound: flowery with linalool ($b_1$ coefficient), fruity with ethyl hexanoate ($b_2$) and glue-like with ethyl acetate ($b_3$), indicating the reliability of the training session. Moreover, odour-interactive effects between compounds were also observed in all descriptors, and will be discussed in the following sections.

The concentration of the selected compounds in the CCD samples were much higher than their odour thresholds (Table 1). However, the relation between concentration and odour intensity differs in each compound, and that is why OAV should not be compared between compounds. Regardless, concentration and odour intensity relationships are usually modelled with a sigmoidal function *(20)*. In contrast, in this study no quadratic effects were found for any descriptor. Therefore, it has been assumed that the intermediate concentration used in the CCD was located in the logarithmic

**Table 2.** Design of experiment for sensory analysis for 18 assessors and 26 samples

| Tasting order Assessor number | Sample number (Table 1) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | First session | | | Second session | | | Third session | | | Fourth session | | |
| | 1st | 2nd | 3rd | 1st | 2nd | 3rd | 1st | 2nd | 3rd | 1st | 2nd | 3rd |
| 1 | 3 | 18 | 1 | 2 | 21 | 4 | 10 | 26 | 24 | 15 | 14 | 11 |
| 2 | 8 | 24 | 11 | 25 | 13 | 21 | 4 | 5 | 22 | 16 | 12 | 14 |
| 3 | 5 | 20 | 15 | 22 | 8 | 16 | 20 | 9 | 23 | 3 | 4 | 2 |
| 4 | 21 | 17 | 22 | 4 | 1 | 2 | 14 | 8 | 3 | 11 | 24 | 15 |
| 5 | 7 | 26 | 16 | 10 | 1 | 5 | 17 | 25 | 2 | 18 | 24 | 20 |
| 6 | 12 | 9 | 13 | 2 | 26 | 25 | 6 | 1 | 12 | 15 | 25 | 16 |
| 7 | 25 | 6 | 19 | 24 | 22 | 23 | 16 | 19 | 13 | 5 | 3 | 21 |
| 8 | 10 | 23 | 14 | 6 | 4 | 5 | 18 | 21 | 7 | 19 | 11 | 20 |
| 9 | 14 | 8 | 12 | 3 | 2 | 1 | 7 | 4 | 6 | 10 | 15 | 24 |
| 10 | 17 | 9 | 24 | 15 | 3 | 6 | 8 | 1 | 5 | 23 | 10 | 19 |
| 11 | 19 | 13 | 26 | 20 | 11 | 9 | 13 | 12 | 25 | 9 | 22 | 1 |
| 12 | 23 | 6 | 7 | 4 | 2 | 3 | 21 | 19 | 17 | 11 | 15 | 10 |
| 13 | 1 | 4 | 3 | 26 | 14 | 17 | 24 | 11 | 10 | 25 | 7 | 8 |
| 14 | 11 | 25 | 15 | 24 | 7 | 18 | 22 | 16 | 23 | 5 | 17 | 26 |
| 15 | 22 | 18 | 20 | 23 | 12 | 19 | 3 | 26 | 9 | 21 | 6 | 13 |
| 16 | 16 | 5 | 10 | 9 | 7 | 8 | 2 | 20 | 18 | 1 | 17 | 4 |
| 17 | 12 | 10 | 11 | 21 | 19 | 20 | 6 | 18 | 22 | 14 | 13 | 9 |
| 18 | 13 | 15 | 14 | 18 | 16 | 17 | 12 | 23 | 7 | 26 | 2 | 8 |

**Table 3.** Response surface methodology estimate effects of flowery, fruity and glue-like orthonasal responses, calculated with coded concentration values (±1) of linalool ($X_1$), ethyl hexanoate ($X_2$), ethyl acetate ($X_3$) and acetaldehyde ($X_4$)[a]

| Orthonasal response | Coefficient name | Regression coefficient | Standard error | *p*-Value (ANOVA) |
|---|---|---|---|---|
| Flowery | $b_0$ | 1.56 | 0.097 | <0.001 |
| | $b_1$ | 0.373 | 0.116 | 0.002 |
| | $b_2$ | −0.261 | 0.116 | 0.026 |
| Glue−like | $b_0$ | 1.8 | 0.105 | <0.001 |
| | $b_2$ | −0.552 | 0.125 | <0.001 |
| | $b_3$ | 0.396 | 0.125 | 0.002 |
| Fruity | $b_0$ | 2.01 | 0.086 | <0.001 |
| | $b_2$ | 0.928 | 0.103 | <0.001 |
| | $b_4$ | 0.233 | 0.103 | 0.025 |
| | $b_{2,3}$ | −0.246 | 0.109 | 0.025 |
| | $b_{2,4}$ | −0.353 | 0.109 | 0.001 |

[a]Coefficients are related to eqn (1) for each response.

phase of the theoretical odour intensity function and the maximum concentration on the plateau of the sigmoidal function.

In short, the models of this study should be interpreted as a quick technique to evaluate potential odour interactive effects between volatile compounds, but not as accurate odour intensity estimation models.

### Flowery and glue-like descriptors

Flowery and glue-like descriptors are discussed together as both presented similar odour interactive effects with ethyl hexanoate (Table 3). Regarding to the flowery descriptor, the regression coefficient of linalool had a positive value ($b_1$ coefficient), as can be expected since it was the trained compound. However, ethyl hexanoate appeared to negatively affect the flowery perception ($b_2$). Flowery perception decreased with high concentrations of this ester. This effect is also shown in Fig. 1, where the highest score of flowery descriptor occurred with high concentrations of linalool and low concentrations of the ethyl hexanoate. Peña y Lillo et al. *(8)* studied the correlations between volatile compounds, distillation fraction, blending process and main sensory attributes of Pisco by principal component analysis. This study showed that ethyl hexanoate and linalool were the highest correlated chemical markers of the blended Pisco heart fraction, where linalool odour

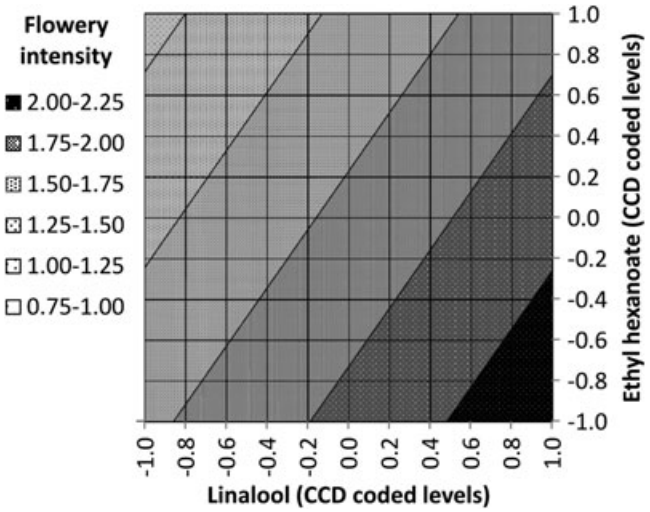


**Figure 1.** Response surface contour plot of flowery sensory response with respect to linalool and ethyl hexanoate coded concentrations (±1.00). Concentrations of ethyl acetate and acetaldehyde were both fixed at −1.00 coded level. Figure is reproduced in colour in online version.

was the main sensory attribute. However, that relationship might be due to the common aroma of hearts of Muscat spirits, since it has been observed that ethyl hexanoate decreased the intensity of linalool odour at all levels of our concentration range.

The glue-like descriptor, as expected, was associated with ethyl acetate with positive regression coefficient ($b_3$), since it was the trained compound for glue-like perception. However, ethyl hexanoate decreased the intensity of the glue-like descriptor at all tested levels (Fig. 2) as a flowery descriptor. Their chemical-structure likeness (structure–odour relationship) could lead to confusion of both esters' odour perception *(21)*.

These results allow us to establish that ethyl hexanoate has a masking effect on linalool and ethyl acetate odours and consequently on flowery and glue-like descriptors.

### Fruity descriptor

The fruity descriptor was the odour response more affected by the studied volatile compounds. As expected, ethyl hexanoate had a positive regression coefficient ($b_2$), since it was the trained compound for the fruity descriptor. However, there was an important odour-interactive effect of acetaldehyde, with a linear positive regression coefficient ($b_4$). Furthermore, a negative regression coefficient of the two-way interaction between acetaldehyde and ethyl hexanoate also influenced the fruity perception ($b_{2,4}$). As can be seen in Fig. 3, acetaldehyde increased the fruity descriptor at low concentrations of ethyl hexanoate, but without providing high odour intensity values. Several studies have shown that acetaldehyde has a fruity odour *(22,23)*. Coetzee et al. *(14)* found that acetaldehyde had an odour-interactive effect with the fruity descriptor using wine model solutions, showing enhancement at low concentrations and suppression at high concentrations. However, an enhancement of the ethyl hexanoate perception by acetaldehyde should be discarded, since the increase in fruity descriptor by acetaldehyde was much higher in samples without ethyl hexanoate. Thus, in our study assessors could perceive slight fruity notes of acetaldehyde which, in turn, were confused with the trained fruity odour of ethyl hexanoate. In addition, a slight masking effect on fruity odour can be observed in Fig. 3 at high levels of ethyl hexanoate.

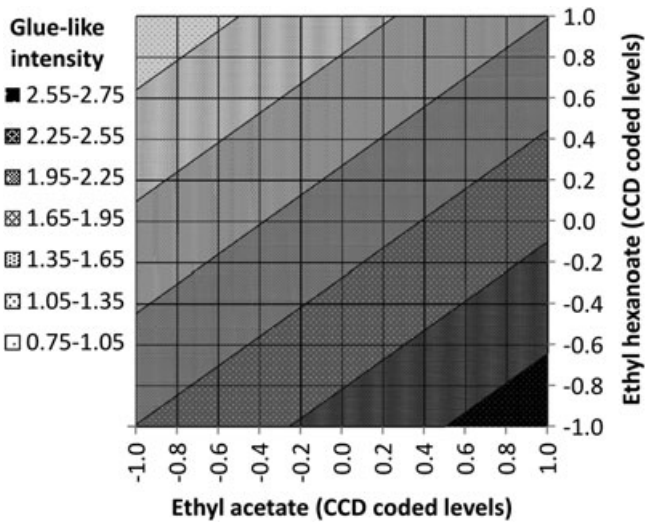


**Figure 2**. Response surface contour plot of glue-like sensory response with respect to ethyl acetate and ethyl hexanoate coded concentrations (±1.00). Concentrations of linalool and acetaldehyde were both fixed at −1.00 coded level. Figure is reproduced in colour in online version.

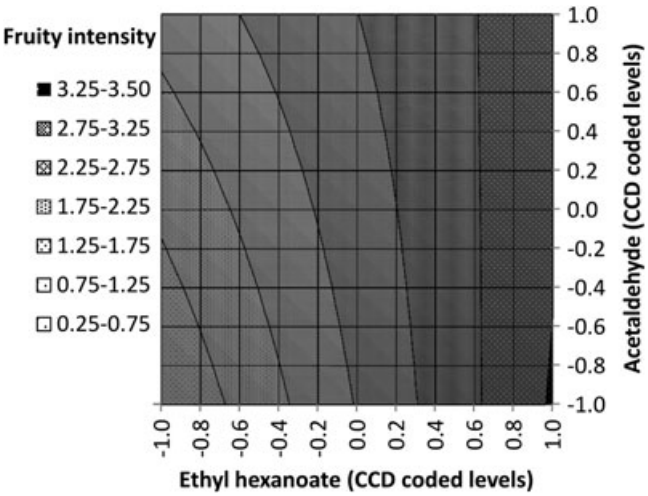


**Figure 3**. Response surface contour plot of fruity sensory response with respect to ethyl hexanoate and acetaldehyde coded concentrations (±1.00). Concentrations of linalool and ethyl acetate were both fixed at −1.00 coded level. Figure is reproduced in colour in online version.

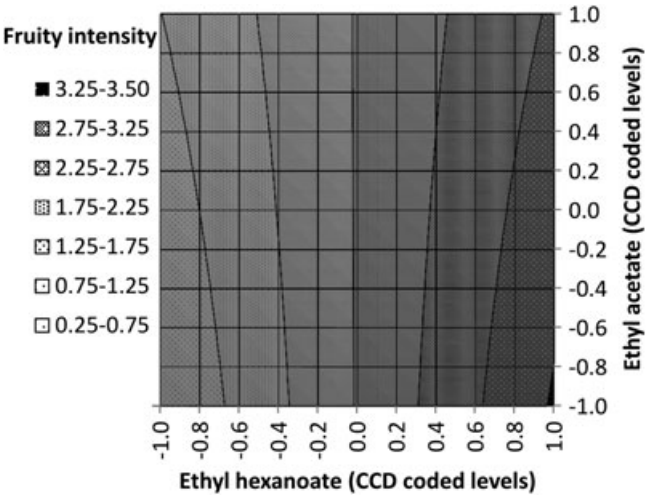


**Figure 4**. Response surface contour plot of fruity sensory response with respect to ethyl hexanoate and ethyl acetate coded concentrations (±1.00). Concentrations of linalool and acetaldehyde were both fixed at −1.00 coded level. Figure is reproduced in colour in online version.

A similar effect can be observed in Fig. 4 with a two-way interaction effect between ethyl hexanoate and ethyl acetate ($b_{2,3}$ coefficient). High levels of ethyl acetate enhanced the fruity perception at low levels of ethyl hexanoate, since ethyl acetate could provide a fruity odour at low concentrations *(3,23)*. Otherwise, high levels of ethyl acetate suppressed the fruity perception at high concentrations of ethyl hexanoate, suggesting a masking odour effect. Thus, as previously mentioned, their structure–odour relationship could lead to confusion of both odour perceptions *(21)*. Finally, linalool could also provide a fruity odour *(22,23)*; however odour interactive effects were not found in this study.

## Conclusions

In this work, RSM has shown odour interactive effects between linalool, ethyl hexanoate, ethyl acetate and acetaldehyde on flowery,

fruity and glue-like descriptors. Ethyl hexanoate showed a sensory masking effect on linalool and ethyl acetate, in relation to flowery and glue-like odour perception. Acetaldehyde and ethyl acetate increased fruity perception when samples had low ethyl hexanoate levels. High levels of ethyl acetate showed a masking effect on the fruity descriptor at high levels of ethyl hexanoate. Finally, it should be highlighted that RSM allowed easily evaluation of odour-interactive effects between volatile compounds in a medium that produces high olfactory fatigue after setting a single training session, showing the tool to be innovative and easy for future sensory multi-interaction studies.

**Acknowledgements**

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## References

1. Christoph, N., and Bauer-Christoph, C. (2007) Flavour of spirit drinks: Raw materials, fermentation, distillation, and ageing, in *Flavours and Fragrances: Chemistry, Bioprocessing and Sustainability*, (Berger, R. G. Ed.), pp. 219–238, Springer, Berlin.
2. Cacho, J., Moncayo, L., Palma, J. C., Ferreira, V., and Culleré, L. (2013) Comparison of the aromatic profile of three aromatic varieties of Peruvian pisco (Albilla, Muscat and Torontel) by chemical analysis and gas chromatography–olfactometry, *Flavour Fragr. J. 28*, 340–352.
3. Buettner, A., and Schieberle, P. (2001) Evaluation of aroma differences between hand-squeezed juices from valencia late and navel oranges by quantitation of key odorants and flavor reconstitution experiments, *J. Agric. Food Chem. 49*, 2387–2394.
4. Cacho, J., Moncayo, L., Palma, J. C., Ferreira, V., and Culleré, L. (2012) Characterization of the aromatic profile of the Italia variety of Peruvian pisco by gas chromatography–olfactometry and gas chromatography coupled with flame ionization and mass spectrometry detection systems, *Food Res. Int. 49*, 117–125.
5. Agosin, E., Belancic, A., Ibacache, A., Baumes, R., and Bordeu, E. (2000) Aromatic potential of certain muscat grape varieties important for pisco production in Chile, *Am. J. Enol. Vitic. 51*, 404–408.
6. US Environmental Protection Agency (2012) Estimation Program Interface (EPI Suite, version 4.1.1).
7. Matias-Guiu, P., Rodríguez-Bencomo, J. J., Orriols, I., Pérez-Correa, J. R., and López, F. (2016) Floral aroma improvement of Muscat spirits by packed column distillation with variable internal reflux, *Food Chem. 213*, 40–48.
8. Peña y Lillo, M., Agosin, E., Andrea, B., and Latrille, E. (2005) Chemical markers for tracking the sensory contribution of production stages in muscat wine distillates, *J. Food Sci. 70*, S432–S441.
9. Apostolopoulou, A. A., Flouros, A. I., Demertzis, P. G., and Akrida-Demertzi, K. (2005) Differences in concentration of principal volatile constituents in traditional Greek distillates, *Food Control 16*, 157–164.
10. Francis, I. L., and Newton, J. L. (2005) Determining wine aroma from compositional data, *Aust. J. Grape Wine Res. 11*, 114–126.
11. Ferreira, V. (2012) Revisiting psychophysical work on the quantitative and qualitative odour properties of simple odour mixtures: A flavour chemistry view. Part 2: qualitative aspects. A review, *Flavour Fragr. J. 27*, 201–215.
12. Ferreira, V. (2012) Revisiting psychophysical work on the quantitative and qualitative odour properties of simple odour mixtures: A flavour chemistry view. Part 1: intensity and detectability. A review, *Flavour Fragr. J. 27*, 124–140.
13. Niimi, J., Overington, A. R., Silcock, P., Bremer, P. J., and Delahunty, C. M. (2016) Cross-modal taste and aroma interactions: Cheese flavour perception and changes in flavour character in multicomponent mixtures, *Food Qual. Prefer. 48*, 70–80.
14. Coetzee, C., Brand, J., Jacobson, D., and Du Toit, W. J. (2016) Sensory effect of acetaldehyde on the perception of 3-mercaptohexan-1-ol and 3-isobutyl-2-methoxypyrazine, *Aust. J. Grape Wine Res. 22*, 197–204.
15. Syarifuddin, A., Septier, C., Salles, C., and Thomas-Danguin, T. (2016) Reducing salt and fat while maintaining taste: An approach on a model food system, *Food Qual. Prefer. 48*, 59–69.
16. Jack, F. (2003) Development of guidelines for the preparation and handling of sensory samples in the Scotch whisky industry, *J. Inst. Brew. 109*, 114–119.
17. Box, G. E. P., and Wilson, K. B. (1951) On the experimental attainment of optimum conditions, *J. R. Stat. Soc. 13*, 1–45.
18. AFNOR (1987) NF V09-105, in *Analyse sensorielle – Directives générales pour l'implantation de locaux destinés à l'analyse sensorielle*, Association française de normalisation, Paris, pp. 1–13.
19. Peña y Lillo, M., Latrille, E., Casaubon, G., Agosin, E., Bordeu, E., and Martin, N. (2005) Comparison between odour and aroma profiles of Chilean Pisco spirit, *Food Qual. Prefer. 16*, 59–70.
20. Chastrette, M., Thomas-Danguin, T., and Rallet, E. (1998) Modelling the human olfactory stimulus–response function, *Chem. Senses 23*, 181–196.
21. Rossiter, K. J. (1996) Structure—odor relationships, *Chem. Rev. 96*(8), 3201–3240.
22. Rouseff, R., and Perez-Cacho, P. R. (2007) Citrus flavour, in *Flavours and Fragrances; Chemistry, Bioprocessing and sustainability*, (Berger, R. G. Ed.), pp. 117–134, Springer, Berlin.
23. Rega, B., Fournier, N., and Guichard, E. (2003) Solid phase microextraction (SPME) of orange juice flavor: Odor representativeness by direct gas chromatography olfactometry (D-GC-O), *Agric. Food Chem. 51*(24), 7092–7099.