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DEVELOPMENT AND FORMATION FOR THE DESCRIPTIVE SENSORY EVALUATION PANEL OF YOUNG ALCOHOLIC SPIRIT BEVERAGES

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Development and formation for the descriptive sensory evaluation panel of young alcoholic spirit beverages.

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Abstract

One of the greatest hardships of the food and beverages industry is sensory evaluation of these products. In the case of young distilled beverages, it is also added the fact that the high alcohol content interferes to a greater extent with the organoleptic senses making this task even more difficult. In this current study a training methodology was tested to perform Quantitative Descriptive Analysis of young Muscat distillates. Also, descriptors were evaluated to find most significant characteristics easily assessed in quick and short trainings. Finally, a synergic and antagonist study was performed over four important compounds: Acetaldehyde, Linalool, Ethyl Hexanoate, and Ethyl Acetate. It was found that for the floral descriptor of Linalool it is inhibited by Ethyl Hexanoate and Ethyl Acetate. Also significant interactions between Ethyl Hexanoate-Acetaldehyde and Ethyl Hexanoate-Ethyl Acetate were shown to potentiate the fruity descriptor. Finally, Linalool and Ethyl Hexanoate have a masquerading effect over the Glue/Nail polish remover descriptor.

Una de las mayores dificultades de la industria de alimentaria es la correcta evaluación sensorial de estos productos. En el caso de las bebidas destiladas, se acrecienta la dificultad por el hecho de estas cuentan con un alto contenido de alcohol que interfiere en mayor medida con los sentidos organolépticos haciendo está labor un poco más complicada. En el presente estudio se evaluó una metodología de formación de panel de cata para llevar a cabo Análisis Descriptivo Cuantitativo de destilados Moscatel jóvenes. Además, se evaluaron los descriptores para encontrar características más importantes que puedan ser evaluados fácilmente por medio de entrenamientos rápidos y cortos. Finalmente, un estudio sinergia y antagonismo se realizó en cuatro compuestos importantes: Acetaldehído, Linalool, Hexanoato de Etilo, y Acetato de Etilo. Se encontró que el descriptor floral de Linalool es inhibido por Hexanoato de Etilo y el Acetato de Etilo. También se encontraron interacciones significativas que potencian el descriptor frutal entre Hexanoato de Etilo-Acetaldehído y Hexanoato de Etilo-Acetato de Etilo. Finalmente, se pudo concluir también que Linalool y Hexanoato de Etilo tienen un efecto de inhibidor sobre el descriptor de Pegamento.

1. Introduction

The sensory characterization of young distilled spirits can prove to be difficult and complex since it can provoke saturation or interference of the senses of panelists due to the high concentrations of ethanol present in the beverage. Both orthonasal and retronasal senses have shown to be hindered when performing extensive evaluations with multiple samples. Additionally further restricting sensory analysis, it has been shown that tasters have poor ability to describe red and white wine using olfactory attributes through retronasal sensory olfactory perception (Aubry, Etievant, Sauvageot, & Issanchou, 1998; Zamora & Guirao, 2002).

It is particularly important to be able to characterize young distilled spirits in the output stream before further treatments are applied because it may be an indicator of the outcome of the distillation process. Nevertheless, currently available score sheet for spirits tend to be too generalized like the OIV (2009), example shown in **Figure A-1**. Here no specific aroma or odor profiles have been specified, instead it describes the beverage based on its typicality or quality. Moreover, other researchers have evaluated distilled beverages using the Buxbaum model were only four attributes were evaluated on a 20 points base scale (Balcerek, Pielech-Przybylska, Patelski, Sapinska, & Ksiezopolska, 2013; Tešević et al., 2005). However, the attributes studied kept the same generalized features since the evaluation parameters were color, clearness, aroma, and taste.

Sensory evaluation and development of vocabulary for distilled beverages have been conducted and applied to a variety of distilled products (Donnell, Hulin-Bertaud, Sheehan, & Delahunty, 2001). However, unlike wine and beer, to an extent, distilled beverages have a plethora of primary aromas from its ranging amounts of raw materials. For example: rum is made from sugar cane, tequila from agave, brandy from grapes, whiskey from grains, vodka from grains or potato, or fruit spirits that can be made from berries, prunes, apple or pear. Furthermore, variation and complexity is added if the product is subjected to aging since it can vary multiple parameters such as: time, wood employed, primary use of the barrels (if previously used or if it is new), or treatment to the barrels to mention some. Characterization of a spirit is affected by the origin and treatment of the product; therefore, a solution fit all sizes would not describe adequately all products. Each product must be studied in order to perform an effective sensory descriptive analysis.

Nevertheless, Bordeu, Formas, & Agosin (2004), have developed an extensive aroma wheel that identifies odors, aromas and mouthfeels that appear to be more noticeable in Chilean Piscos that may be used as a source for the development of such attributes. The first tier of odor and aroma descriptors is classified as: oily, wood, sweet, fruity, flora, spicy, vegetative, chemical, oxidized, earthy and moldy.

In the interest of narrowing down the most significant attributes that are related to the distillation processes, it will be selected some characterizing compounds that are intrinsically linked to a part of the process. Pungent and Glue or nail polish remover will be used to describe the head fraction of the distillate. While, floral attributes will be used to describe for heart fraction which allows evaluating the distillation process based in the preservation of the raw materials characteristics. Also, fruity attribute will be used to describe the distillation process as well since it is a positive indicator. Finally, smoke is linked to the tail fraction and indicates the final cut and help evaluates the duration of the distillation. Peña y Lillo, Latrille et al., (2005) found that the most trackable compounds per fraction for Peruvian Pisco are as follow: head fraction is rich in esters and the heart fraction is rich in alcohols and terpenes, while the tail fraction (second heart) is rich in acids, alcohols and Furfural.

The main objective of this study is to develop a training methodology that relies on quick and simple trainings of rotating participants to identify important attributes oriented on young Muscat distillates. Additionally, it is intended to identify the most important and descriptive parameters or attributes that will reliably evaluate the prominent characteristics of young distilled spirit. Finally, it will be studied the synergic or antagonist effects that these compounds exert on each other.

2. Materials and Methods

Reagents and Samples

To prepare de samples in this study neutral spirit made from wine at 95 % concentration of alcohol by volume (ABV) was used. Concentration of 40 % ABV was achieved by via dilution with distilled water to inhibit any properties that may arise from components in mineral water. Samples were spiked with different compounds such as Acetaldehyde for pungent descriptor, Linalool and Geraniol for floral descriptor, Isoamyl Acetate, Ethyl Hexanoate and Ethyl 2-trans-4-cis-Decadienoate (Ethyl Decadienoate or Pear Ester) and Ethyl Decanoate for fruity descriptor, Ethyl Acetate for glue descriptor and, Furfural for smoke descriptor. Linalool, Geraniol, Isoamyl Acetate were kosher food grade quality (Sigma Aldrich, St. Louis, MO, USA), Ethyl Hexanoate and Furfural were food grade as well (Aldrich, Steinheim, Germany) and Acetaldehyde, Ethyl Decanoate, Ethyl 2-trans-4-cis-Decadienoate, and Ethyl Acetate were HPLC quality (Fluka, France, Switzerland and Germany).

Gas Chromatography

A Chromatograph Agilent Technologies 6890N equipped with a flame ionizing detector (GC-FID) was used to perform analysis on different samples. This was done to verify purity of raw materials and control of spiked samples. The capillary chromatographic column used was a polar MetaWAX of 60 meters in length, 0.25 mm internal diameter and 0.5 µm of phase thickness. Sample were prepared with 2-octanol at a 20 ppm concentration as an internal standard.

The method utilized to analyze samples have been taken form Matias-Guiu, Rodríguez-Bencomo, Orriols, Pérez-Correa, & López, (2016). For the major and most volatile compounds, an injection of 2 µL was done in split mode (1:5) and was performed with an oven temperature program of: 40 ºC (5 min), 7 ºC/min up to 100 ºC (15 min), 3 ºC/min up to 140 ºC and 2 ºC/min up to 200 ºC (5 min). The carrier gas used was helium with a column-head flow ramp of 0.5 mL/min (28 min) and 5 mL/min up to 1.1 mL/min (67 min). For heavier and minor compounds, the injection was done in undivided mode, with an oven temperature ramp of 40 ºC (7 min), 2 ºC/min up to 140 ºC and 6 ºC/min up to 220 ºC (20 min). The carrier gas for both was helium at a constant column-head flow of 1 mL/min.

Quantification was performed by interpolation into calibrations built with synthetic solution spiked with the analyses at different levels.

Tasting panel, training and evaluation

All sensory analysis was carried out in the tasting room at the faculty of Enology of Universitat Rovira i Virgili in compliance with standard NF V09-105 AFNOR (AFNOR, 1987). The sensory panel consisted of 51 rotating judges of ages between 18 and 45. Where 33 of the participants

were ages 18- 24, 13 were between 25-29 and 5 where above 30. All participants were undergraduate, graduate and staff at Universitat Rovira i Virgili. About three fifths were males and about the same amount of the total participants reported having experience in sensory analysis panels.

Training was performed during 6 sessions of 1 hour focusing on one characteristic at a time. In order to better accommodate all participants, 2 different times slots were made available each week summing a total of 12 training sessions. Samples were prepared by using the compounds and concentrations as shown in **Table A-1**.

Training was executed differently from one session to the other of the same characteristic. In one of the sessions, 3 sample scale were given to each judge at 2 different intensities of the characteristic being studied. While in the other session, a 5 sample scale was used where there were 4 different intensities of the odor being studied. Volume served in each sample were of 5 mL. All samples were covered with petri dishes and were prepared in advance to allow the volatile fraction to balance in the glass. To spike samples, as a reference, concentrations used were obtained from the characterization done previously on Chilean Pisco (Peña y Lillo, Agosin, Belancic, & Latrille, 2005; Peña y Lillo, Latrille, et al., 2005)

A brief explanation of the compound and its orthonasal and retronasal characteristic odor and aroma was given to assessors during training sessions. Additionally, participants were encouraged to participate in giving their views on characterization of the compound to enhance their understanding. Participants were given between 10 and 15 minutes to assimilate the intensity and characterization of the week training. Later an Alternative Force Choice (3-AFC) test was performed orthonasally and retronasally, in certain cases, to track and understand each participant's performance.

Pungent training session: The objective of this session was to evaluate this compound that is usually present in the fermented beverage since it is a by-product in the fermentation after the degradation of pyruvate (Christoph & Bauer-Christoph, 2007). Due to its low boiling point, it comes at high concentrations in the head fraction. Concentrations of Acetaldehyde used for these sessions started at 400 ppm up to 1000 ppm. This descriptor is important since it is an indicator for the head fraction cut performance.

Floral training session: The aim of this session was to evaluate terpene compounds that are present in the heart fraction of the distillation and come from the raw material that was used to ferment the beverage. In this case concentrations of the compounds used were between 0 and 5 and 10 ppm and 0 ppm to 2 ppm for Linalool and Geraniol respectively. In this session, sensory evaluation orthonasally and retronasally were performed. This descriptor can be an indicator of the performance of the distillation in general since it characterizes the raw materials.

Fruit training session: Compounds characterized as fruits generally are esters are synthesized in the alcoholic fermentation for the most part (Peña y Lillo, Agosin, et al., 2005). The objective was to train judges to be able to identify this aroma by itself. The samples for these training sessions were made from a combination of a distillate of pear and neutral spirit. Dilutions were performed from 0% pear dilution to 100% the sample of this distillate. Just like the floral attribute, this also is an indicator of the performance of the raw material.

Floral/fruity Session: This session was design to train judges to be able to distinguish between what should be categorized as floral and fruity. Both samples were presented to the

participants at the same time for training. Samples were ordered in a gradient that went from one attribute to the other, passing through the neutral spirit in the middle (avoiding spiking a sample with both descriptors). For the 3-AFC test, as previously done, two samples where the same and one was different; thus assessors had to find which sample was singled out and identify of it was floral or fruity.

Glue/Nail polish remover: Ethyl Acetate is usually the compound attributed as the aroma for this descriptor. As for the fruity compounds, this also comes as a result of the fermentation process. At high concentrations this compound may be unpleasant. The compound appears at the beginning of the distillation in the head fraction just after Acetaldehyde. The samples for this session were prepared at concentrations 0 to 300 ppm. As the pungent descriptor, this also indicates the performance of the head cut.

Smoke and almond: The characteristic compounds of this descriptor generally are formed during ageing, however, the smoke and almond notes may arise due to long exposure to heat at the end of the distillation process as part of the Mailliard reactions between amino compounds and reducing sugars (Mottram, 2007). Samples were prepared by spiking Furfural at ranging concentrations from 0 ppm to 300 ppm. This should indicate not only the tail cut but also distillation time, since these compounds become more apparent as distillation time increases.

To conclude with this section a Quantitative Descriptive Analysis (QDA) was performed in 4 sessions to further evaluate participants and descriptors. Nine samples were prepared with known amounts of compounds. In the interest of simplicity, all samples were evaluated orthonasally only and for 5 specific characteristics (floral, fruit, glue or nail polish remover, smoke and pungent). A training remainder was performed at the beginning of the session. The scale of evaluation of each characteristic was done on a scale from 0 to 9.

Synergic and Antagonistic Interactions

The aroma interactions between Linalool, Ethyl Hexanoate, Ethyl Acetate and Acetaldehyde were studied in 4 tasting sessions of 30 to 40 minutes each. There were 18 junior students in enology at Universitat Rovira i Virgili participating in this sensory analysis panel. The first session performed was a quick training session where participants were introduced to three-sample scales of each compound except Acetaldehyde.

In order to perform the training, it was instructed that the first sample was a neutral spirit with no odor compound. The second sample of each series would have represented a medium concentration of the compound (representing a 2 in an evaluation scale from 0 to 5). The last sample was the higher concentration and it was instructed that would be representative of a 4 in the evaluation scale. Since the study was designed to understand the synergic or antagonistic interactions, a margin was allowed in the scale so that in a case of a potentiation interaction between two compounds assessors would be able to evaluate it properly.

A face centered star points design was executed together with a design of tastings where each participant only tasted three samples per session. Prior to conducting the sensory evaluation participants were allowed to review the three scale training samples for floral descriptor (Linalool), fruity descriptor (Ethyl Hexanoate), and glue/nail polish remover descriptor (Ethyl Acetate). Spiking concentrations for each sample and the experimental design for tasting used can be seen in **Table A-2** and **Table A-3**.

Statistical Analysis

For the Statistical analysis two different software packages where used. The 3-AFC test data was processed using the binomial distribution analysis and Thurstone model in XLSTAT (Addisoft, Brooklyn, NY, USA). The assessors performance was evaluated through PanelCheck (Version V1.4.2, Nofima, Tromsø, Norway) using the workflow in Tomic et al., (2009). Significant attributes were identified by three-way ANOVA. Tucker plots and Statis weight of assessors were used to find consensus amongst participants. Taking into account only significant attributes and assessors in agreement, a Principal Component Analysis (PCA) bi-plot was performed to analyze samples and attributes. An ANOVA was used to performed in order to identify significant synergic and antagonistic interactions between compounds. Later, a Linear Modeling tool was used to understand how compounds affected attributes. Both were performed using XLSTAT (Addisoft, Brooklyn, NY, USA).

3. Results and discussion

Training and Sensory Panel

As shown in **Table 3-1**, fifty-one judges participated in the study all together. Only ten participants made it through all of the training sessions (one of the participants participated on one of the training sessions twice summing a total of 7 sessions for that participant). The following 10 participants were able to attend 5 sessions. This means, that they failed to attend only one training session. Most participants were identified as having previous training or experience in sensory analysis for other products (mostly wine) although none had specific training or experience in distilled beverages.

Table 3-1 The number of participants that attended a given number of training sessions.

Amount of training performed	Number of Participants	Sex		Experience	
		Male	Female	No	Yes
1	23	12	11	12	11
2	5	4	1	3	2
3	2	2	0	1	1
4	1	1	0	0	1
5	10	4	6	3	7
6	9	6	3	3	6
7	1	1	0	0	1
Total	**51**	**30**	**21**	**22**	**29**

Alternative Force Choice Test Results

Six training sessions were done focusing in an attribute or the distinction between two attributes. In chronological order, training sessions were: pungent (Acetaldehyde), flowery (Linalool and Geraniol), fruity (Isoamyl Acetate, Ethyl Decanoate and Ethyl 2-trans-4-cis-Decadienoate), flowery and fruity, glue (Ethyl Acetate) and smoke or almond (Furfural). The samples were presented to participants after the short training and they were instructed to indicate which sample was different and how was it different. In all but one case, they only need to indicate if it was more or less of the attribute, in relation to the other two samples. In

the floral/fruity session they had to indicate what was the sample (fruity or floral). The data was analyzed using a binomial distribution and Thurstonian model for each 3-AFC (Angulo O. & O'Mahony, 2009). In **Table 3-2** are shown sessions conformation and the results of triangle test and 3-AFC.

Table 3-2 Participation, characteristics of the tasting panel, and the results obtained per session.

Session	Total Participants	Sex		Experienced	Orthonasal (Correct)		Retronasal (Correct)	
		Female	Male		Tringle Test	3-AFC	Tringle Test	3-AFC
Punget	29	11	18	17	8	6		
Floral	32	14	18	21	14	12	12	6
Fruity	27	11	16	19	25	23	18	17
Floral/Fruity	18	7	11	10	13	12	14	12
Glue/Nail Polish	24	9	15	16	17	15		
Smoke	23	9	14	16	21	19	15	12

The P-Value and the distance between the two normal distribution of the compound (d') were calculated for each session. The results can be observed in **Figure 3-1**. The pungent and floral sessions were found to have no significant results ($p>0.05$). Meaning that the ability of the participants to discriminate between samples was non-significant. However, when observing the floral/fruity combined session results, it can be noticed that the outcome is shown to be significant, and one may argue that it is a replicate for the fruity session since the floral discrimination attributes was shown to be non-significant. Nevertheless, when comparing both results through an ANOVA (fruity and floral/fruity), the probability of both being different is about 85%. To reasonably conclude on this subject further experiment should be performed.

Generally speaking, referring to **Figure 3-1,** it can be observed that there is a tendency of better discrimination capabilities or better performance of participants as sessions progressed. However, it cannot be inferred that undoubtedly participants experienced a learning advancement because more data is needed to assert this statement (at least another cycle of the same sessions). As sessions progressed, it can be observed that the P-value got smaller and that the d' (distance between two sample mean normal distribution) got larger. This is more obvious when reviewed by the probability of being the correct answer.

Furthermore, no significant results were obtained by increasing the number of samples for training. Meaning, individual results for all sessions were consistent with their respective duplicate. The fact that training had been done with 3 samples or 5 samples was found to be non-significant. Nevertheless, for the floral training sessions it was found that had different results for each session. One was non-significant while the other session was significant at a tolerance of $\alpha=5\%$. Yet this is probably not due to the fact that each session had different number of samples at training, but to some ulterior reason. In the case of the pungent session, it was found to be both sessions were non-significant as previously observed.

Additionally, the sessions that had retronasal training showed to have significant results as well. The floral/fruity and the fruity 3-AFC tests were found to be significant at a $\alpha=1\%$ tolerance. Likewise, the furfural 3-AFC for retronasal evaluation were found to be significant at a $\alpha= 5\%$ tolerance. Contrarily, the floral session results were non-significant for both sessions performed. To further reiterate results, following the training sessions a QDA was performed.

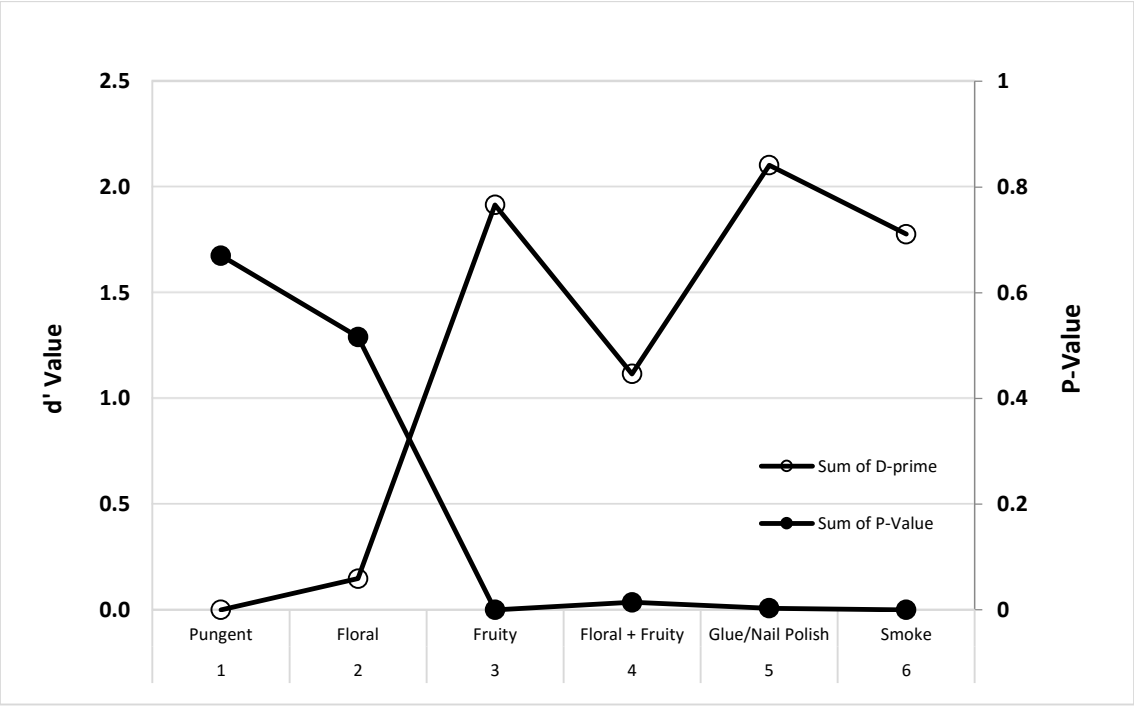

Figure 3-1 P-Values and d' through time or sessions.

Sensory evaluation tasting panel

In order to further evaluate the effectiveness of the training done it was performed 4 tasting session of 9 different distillates artificially spiked. Samples were prepared as shown in **Table 3-3**. When performing the tasting panel Latin Squares were used in order to minimize systematic error on results. A total of 25 participants out of the data base of 51 attended at least to one session. Yet, only 19 participants attended 2 sessions as required for replicates. Therefore, the analysis performed was only based on these 19 participants. Samples were evaluated only orthonasally due to the fact that some compounds used were not food grade.

Table 3-3 Samples composition in parts per million (ppm) of sensory panel

Descriptor	Floral	Fruity		Glue	Smoke	Punget
Compound	Linalool	Pear Ester	Ethyl Decanoate	Ethyl Acetate	Furufural	Acetaldehyde
A	0	0	0	0	0	0
B	4	2.8	0.7	75	25	75
C	8	5.6	1.4	150	50	150
D	8	0	0	75	0	150
E	0	5.6	1.4	75	50	0
F	4	5.6	1.4	0	50	75
G	4	0	0	150	0	75
H	8	2.8	0.7	0	25	150
I	0	2.8	0.7	150	25	0

In **Figure 3-2**, it can be observed that out of 19 participants 13 completed at least 5 out of 6 trainings available. Furthermore, one of the participants repeated the first training session as previously mentioned. In addition, the weight of participants is a measure of the degree of correspondence between data matrices and it is used to lower the emphasis on assessors that

perform different from the group (Næs, Brockhoff, & Tomic, 2010). In this case, it is a measure of consensus among participants. Meaning that the higher the weight, higher evaluation grades used by the participant, and/or had better agreement with the group on the characteristic attributes of the samples. In **Figure 3-2** and **Figure 3-3** it can be seen that there is no relationship between better overall performance after completing a 6 session training. In fact, only 3 out of 8 participants that performed all the training sessions showed to have better weight than the average of the group. Likewise, participants that did not perform all the training sessions had better consensus with the group and performed evaluation more aggressively in comparison.

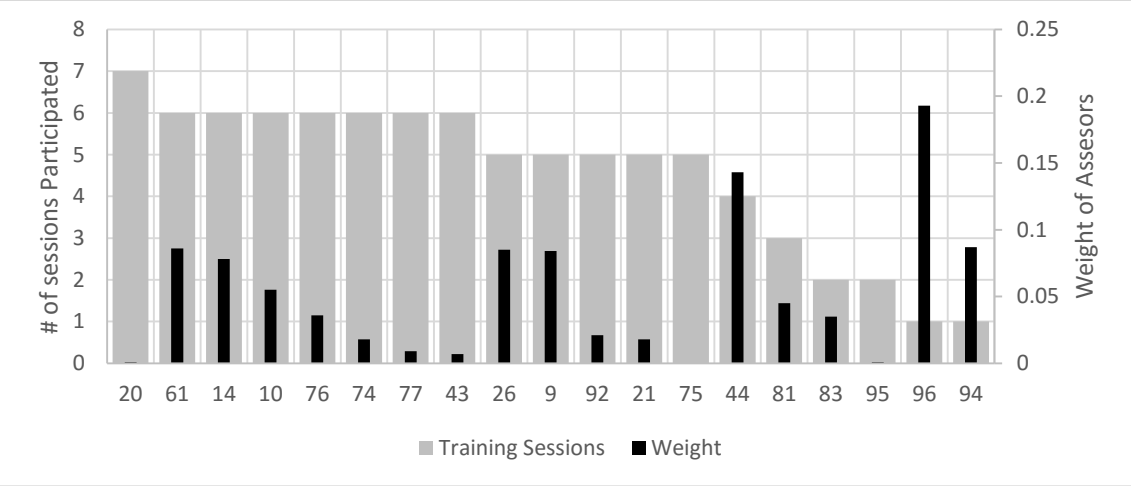

Figure 3-2 Relationship between sessions participated and weight on panel

As observed in the 3-AFC tests, in **Figure 3-4a** is shown that Acetaldehyde (pungent) had no significance to participants. In agreement with previous studies, Acetaldehyde was not perceived as a characteristic of Pisco at high concentrations (Bordeu et al., 2004). This means that they were not able to identify the attribute correctly in the mixed samples. Upon visual inspection of the Tucker-1 plots (**Figure A-2**) aided by the weight calculated to each assessor, 5 were eliminated based on their weigh and lack of consensus with their peers. Hence, it can be observed that the smoke attribute also becomes non-significant (see **Figure 3-4b**).

Overall consensus within participants seems to indicate that they are able to identify floral, fruity, and glue/nail polish remover. This can be observed not only by **Figure 3-4** but also **Figure 3-5** since these three attributes are located in different regions of the Principal Component Analysis (PCA). From this point onward, all performed analysis excludes the 5 lowest ranking participants.

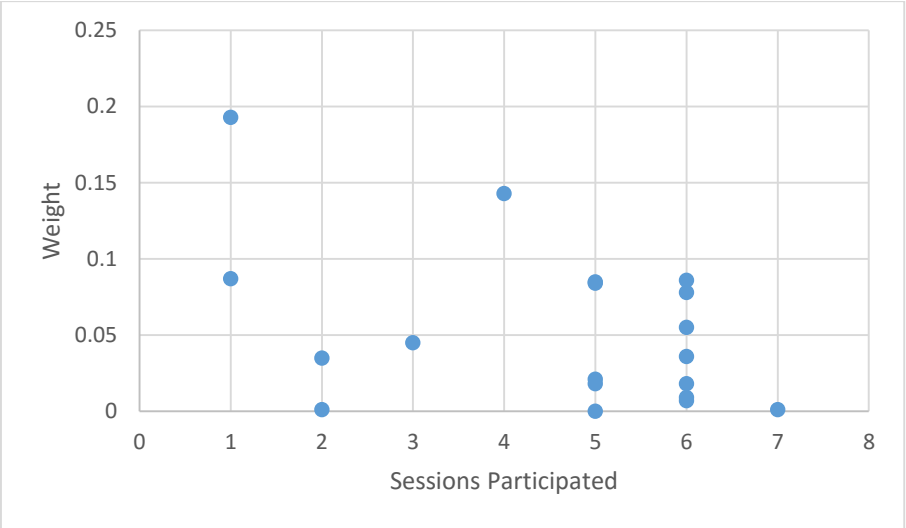

Figure 3-3 Number of training sessions participated and weight each assessor had on evaluation

Further comparison of the results observed in the 3-AFC tests and in the QDA, only fruity and glue/nail polish remover descriptors seem to be significant in both cases. The pungent descriptor or Acetaldehyde has shown difficulty of correctly being evaluated in both cases. However, floral in this case has shown to be highly significant to the 14 participants displaying a P-Value of at least 0.001. Perhaps arising the question whether the 3-AFC test for the floral descriptor might have had an erroneous result due to the ill preparation of the samples, or some improbable event that prevented participants from appropriately identifying the correct answer. In fact, when analyzing individual sessions, one showed to be non-significant (which had slightly more participants) while the other showed to be significant at a tolerance value of α=5%.

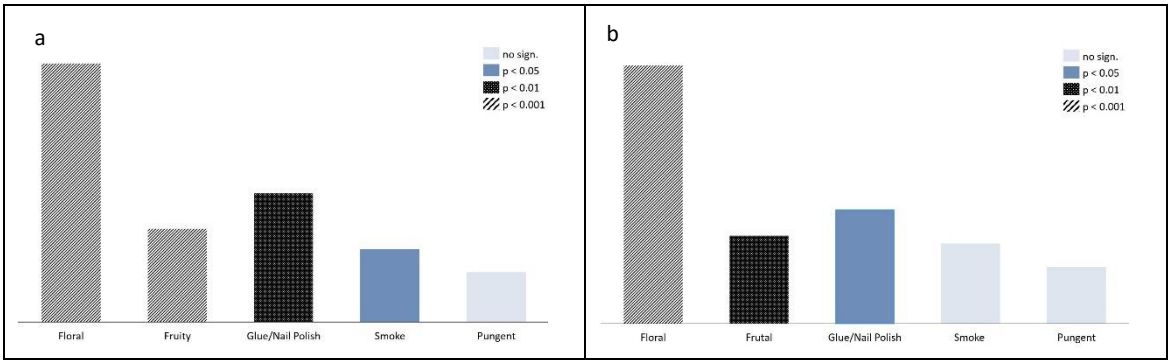

Figure 3-4 F-values and significance of attributes to assessors including (a) all assessors and (b) reducing the 5 with the lowest score weights.

To better understand the performance of the participants based on what it is known from the samples it can be compared the results of the PCA shown in **Figure 3-5** to the values in **Table 3-3**. It can be observed that samples with higher concentrations of Linalool (C, D and H) are further away from the Floral descriptor than the samples that contained half of this quantity (B, F, and G). Coincidentally, these samples closer to the floral descriptor had medium

concentrations of Acetaldehyde and the samples further away had higher concentration of Acetaldehyde.

For the fruity descriptor similar results may be observed, since samples F, C, and H are relatively close to fruity descriptor and all have some Acetaldehyde. Yet, sample D does not contain the fruit compounds intended for this study, however, concentrations of Acetaldehyde and Ethyl Acetate at intermediate concentrations could potentially contribute to a fruity character (Odello, Giomo, Versini, & Zironi, 1997).

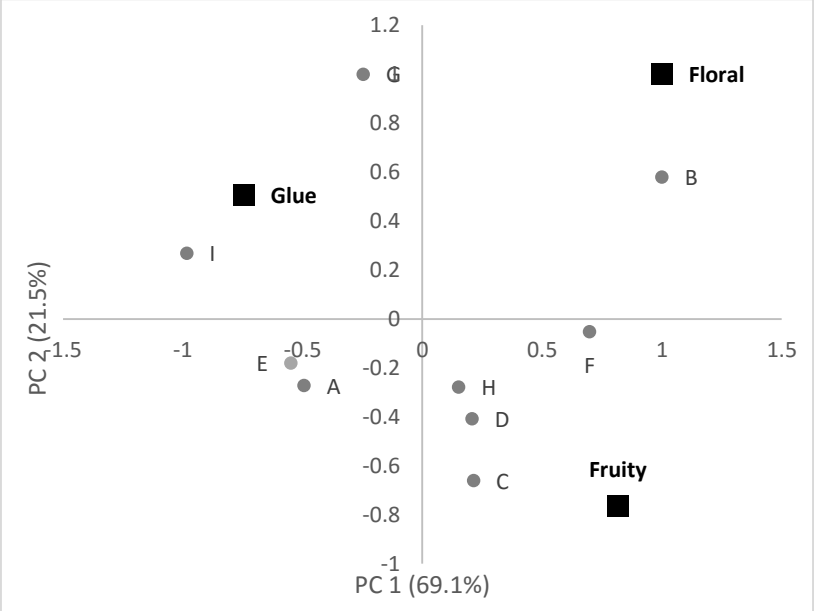

Figure 3-5 Principal Component Analysis (PCA) bi-plot of attributes a samples studied.

Finally, for Glue/Nail polish remover descriptor, as expected, the closest samples are I and G. However, sample C has a higher concentration of Ethyl Acetate but at the same time also the maximum concentration of all the other compounds and it appeared to have more of a fruity character. This could also be potentially explained by the contribution that the other compounds could have on a fruity character. On the remaining samples interactions could not be ruled out, but also these samples are not clearly explained. However, to a certain extent, the training performed can be accredited for the positive results obtained while performing the QDA since the three significant attributes were well evaluated by the panel.

Undoubtedly, some interactions may be observed in these results and further understanding is essential for properly evaluate samples. According to Coetzee et al., (2016), Acetaldehyde in wine at higher concentrations may suppress green pepper aromas and at lower concentrations may contribute to fruity aroma. Given that the experiment was not intended nor designed to evaluate such interactions, and based on the data collected, it cannot be drawn an irrefutable conclusion that these interactions are significant, an experiment was design to study interactions based on what was learned so far.

Synergic and Antagonistic Interactions

Based on the results from the previous section and qualitative information learned during the process, an experiment was design in order to better understand how different compounds interact. Given that the interactions of Acetaldehyde are poorly understood and since it was

shown to be statistically non-significant as a descriptive characteristic, it was included as a spiking agent, but was excluded as an evaluation parameter. The evaluation parameters used were reduced to: floral, fruity and glue/nail polish remover. Furfural and smoke attribute was completely excluded in the interest of simplification of the design of experiments. Additionally, due to the fact that furfural was found to be non-significant as a descriptor in earlier results, and that the smoke characteristic on young distillates is a *coupage* of different compounds and not solely represented by this one compound (Christoph & Bauer-Christoph, 2007). Another experiment could be design to study these specific interactions that would have its own values.

In the interest of time and mitigating the fatigue or saturation of the senses that assessors might suffer, the amount of samples tasted per assessor per session was reduced from nine in the QDA to three in this study. A design of experiments for sensory analysis was performed to lessen the systematic error. Details on the design of experiments for samples and for the study performed are available in **Table A-2** and **Table A-3**.

Table 3-4 P-Values form ANOVA results for each compound and interactions. The results are p<5% or a tolerance of α=5%[a], and bold yellow p<10% or a tolerance of α=10%[b].

Compunds and interactions	Floral	Fruity	Glue/Nail Polish
Linalool (1)	**0.000[a]** +	0.158	**0.025[a]** −
Ethyl Hexanoate (2)	**0.009[a]** −	**0.000[a]** +	**0.000[a]** −
Ethyl Acetate (3)	**0.003[a]** −	0.209	**0.011[a]** +
Acetaldehyde (4)	0.263	**0.0801[b]** +	0.415
(1)•(2)	0.865	0.572	0.189
(1)•(3)	0.714	0.581	0.961
(1)•(4)	0.575	0.636	0.315
(2)•(3)	0.301	**0.026[a]** +	0.459
(2)•(4)	0.361	**0.001[a]** +	0.381
(3)•(4)	0.360	0.718	0.374

In **Table 3-4** P-values from the ANOVA results may be observed. Here as expected, it can be noticed that all the compounds had linear significant relationships with their respective characteristic (i.e. Linalool to Floral, Ethyl Hexanoate to Fruity, Ethyl Acetate to Glue/Nail polish remover). No higher power polynomial relationships were found to be significant in this analysis. Both facts support the idea that assessors and training has performed in conformity with expected and reasonable results. For the floral descriptor, it was found that Ethyl Hexanoate and Ethyl Acetate have significant influence. However, no effect of Acetaldehyde was found over the floral descriptor.

Similarly, it can be observed that there is a significant positive influence of Acetaldehyde at a α=0.10 tolerance over the fruity descriptor. Additionally, both interactions of Ethyl Hexanoate - Ethyl Acetate (2•3) and Ethyl Hexanoate - Acetaldehyde (2•4) are observed to be significant. These interactions have positive correlation with the fruity descriptor at the intermediate concentration of both, Ethyl Acetate and Acetaldehyde. Confirming that it is also true that for distilled beverages an intermediate concentration of Acetaldehyde contributes to the fruity descriptor as observed in white wine by Coetzee et al., (2016). However, no suppression interactions by Acetaldehyde are observable in this experiment. Perhaps, higher concentrations of this compound must be used in order to observe suppression interactions.

Finally, in the Glue/Nail polish remover descriptor: both Linalool and Ethyl Hexanoate had some negative influence over this descriptor. Which affirm that these compounds help masquerading negative attributes in distilled beverages.

4. Conclusions

In the current study it has been performed a training methodology to evaluate young distillate beverages. Training was evaluated through two methods, Alternative Forced Choice test (3-AFC) and through a Quantitative Descriptive Analysis. For the first one, results show some improvement in time. Though, to assert conclusive results regarding the learning tendency observed, a second cycle of training sessions or a repetition of the training to a new panel must be performed. Nevertheless, it may be argued that the training executed may have had a positive effect on the results in the QDA since some attributes were clearly identified by assessors. However, some adverse results were explained by compound interactions that not necessarily correlate to the training done.

Furthermore, it was also found that the compounds used have interactions that can potentiate or hinder odors and aromas. For the case of the floral descriptor of Linalool: it was found that suppression interactions are observed by Ethyl Hexanoate and Ethyl Acetate. Additionally, both interactions of Acetaldehyde and Ethyl Acetate with Ethyl Hexanoate have shown to potentiate the fruity descriptor. In other words, both interactions increase the perception of fruits in a distillate. Finally, it was also found that Linalool and Ethyl Hexanoate suppresses the Glue/Nail polish remover descriptor. Some effects provided in this study may show no significance at high tolerances, however it cannot be ruled out that Linalool or Ethyl Acetate may have some effect over the fruity descriptor since the probability of it having effect is about 80% for both. The same can be said about the effect that Acetaldehyde may over the floral descriptor given its high probability.

5. Recommendations

Further analysis could be performed in order to identify if the order of attributes on the list may alter the evaluation results.

Throughout the training sessions: performing QDA may enhance the learning process of participants with proper feedback.

Further evaluate characteristics on real samples instead of spiked samples in a QDA and, as this is being performed limit the amount of samples that participant evaluate per session.

Find ways to mitigate scale error attempting to encourage participants to evaluate samples at similar magnitudes.

Further experimentation must be done with the 3-AFC tests to better understand if there is a learning process or if some attributes are not as perceptible as others.

6. Acknowledgment

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A. Supplementary Material

Table A-1 Sample preparation for training sessions

Session	Samples in training	Units of measurements	Concentrations	Observations
Pungent	3	ppm (mg/L)	400, 600, 1000	High concentrations of Acetaldehyde were spiked because the neutral spirit used for these sessions started at 400 ppm.
	5	ppm (mg/L)	400, 500, 600, 600,1000	
Floral	3	ppm (mg/L)	0,5,10[a] & 0, 1, 2[b]	Linalool and Geraniol were spiked using the same gradient.[a]Concentrations use for Linalool and [b]concentrations used for Geraniol
	5	ppm (mg/L)	0, 2.5,5,8,10[a] & 0,0.5,1,1.05,2[b]	
Fruity	3	% v/v	0, 50%, 100%	A commercial pear distillate was diluted with neutral spirit to reduce the pear odor and aroma.
	5	% v/v	0, 25%,50%,75%, 100%	
Flora + Fruity	3	ppm (mg/L)& %v/v	10[a], 0, 100%[e]	Samples were presented in counter-gradient from one (no sample had two spikes). Only [a]Linalool and the [d]commercial pear distillate were used as spiking agents.
	5	ppm (mg/L)& %v/v	10, 5[a], 0, 50%, 100%[e]	
Glue/Nail Polish Remover	3	ppm (mg/L)	0, 150, 300	Ethyl Acetate was used to spike samples.
	5	ppm (mg/L)	0, 75, 150, 225, 300	
Smoke	3	ppm (mg/L)	0, 150, 300	Furfural was used to spike samples.
	5	ppm (mg/L)	0, 75, 150, 225, 300	

Table A-2 Face centered 4 factors 3 levels design of experiments for synergic and antagonist interactions

Sample	Floral	Fruit	Glue	Spicy
1	0	0	0	0
2	0	0	0	300
3	0	0	300	0
4	0	0	300	300
5	0	10	0	0
6	0	10	0	300
7	0	10	300	0
8	0	10	300	300
9	10	0	0	0
10	10	0	0	300
11	10	0	300	0
12	10	0	300	300
13	10	10	0	0
14	10	10	0	300
15	10	10	300	0
16	10	10	300	300
17	0	5	150	150
18	10	5	150	150
19	5	0	150	150
20	5	10	150	150
21	5	5	0	150
22	5	5	300	150
23	5	5	150	0
24	5	5	150	300
25 (c)	5	5	150	150
26 (c)	5	5	150	150

Table A-3 Design of experiments for sensory analysis

	Samples											
Participant	First Sesion First Experiment			Second Session First Experiment			First Session Second Experiment			Second Session Second experiment		
J1	P3	P18	P1	P2	P21	P4	P10	P26	P24	P15	P14	P11
J2	P8	P24	P11	P25	P13	P21	P4	P5	P22	P16	P12	P14
J3	P5	P20	P15	P22	P8	P16	P20	P9	P23	P3	P4	P2
J4	P21	P17	P22	P4	P1	P2	P14	P8	P3	P11	P24	P15
J5	P7	P26	P16	P10	P1	P5	P17	P25	P2	P18	P24	P20
J6	P12	P9	P13	P2	P26	P25	P6	P1	P12	P15	P25	P16
J7	P25	P6	P19	P24	P22	P23	P16	P19	P13	P5	P3	P21
J8	P10	P23	P14	P6	P4	P5	P18	P21	P7	P19	P11	P20
J9	P14	P8	P12	P3	P2	P1	P7	P4	P6	P10	P15	P24
J10	P17	P9	P24	P15	P3	P6	P8	P1	P5	P23	P10	P19
J11	P19	P13	P26	P20	P11	P9	P13	P12	P25	P9	P22	P1
J12	P23	P6	P7	P4	P2	P3	P21	P19	P17	P11	P15	P10
J13	P1	P4	P3	P26	P14	P17	P24	P11	P10	P25	P7	P8
J14	P11	P25	P15	P24	P7	P18	P22	P16	P23	P5	P17	P26
J15	P22	P18	P20	P23	P12	P19	P3	P26	P9	P21	P6	P13
J16	P16	P5	P10	P9	P7	P8	P2	P20	P18	P1	P17	P4
J17	P12	P10	P11	P21	P19	P20	P6	P18	P22	P14	P13	P9
J18	P13	P15	P14	P18	P16	P17	P12	P23	P7	P26	P2	P8

OIV Organisation Internationale de la Vigne et du Vin

SCORE SHEET	SPIRITUOUS BEVERAGES OF VITIVINICULTURAL ORIGIN

Jury	N°	Sample	N°	Category	N°

		Excellent +				Inadequat -		Observations
Visual	Limpidity	☐ (5)	☐ (4)	☐ (3)	☐ (2)	☐ (1)		
	Colour	☐ (5)	☐ (4)	☐ (3)	☐ (2)	☐ (1)		
Nose	Typicality*	☐ (6)	☐ (5)	☐ (4)	☐ (3)	☐ (2)		
	Quality	☐ (15)	☐ (13)	☐ (11)	☐ (9)	☐ (7)		
	Positive intensity	☐ (9)	☐ (7)	☐ (5)	☐ (3)	☐ (1)		
Taste	Typicality*	☐ (8)	☐ (7)	☐ (6)	☐ (5)	☐ (4)		
	Quality	☐ (20)	☐ (18)	☐ (14)	☐ (10)	☐ (6)		
	Harmonious persistence	☐ (12)	☐ (10)	☐ (8)	☐ (6)	☐ (4)		
Harmony – Overall judgement		☐ (20)	☐ (18)	☐ (14)	☐ (10)	☐ (6)		

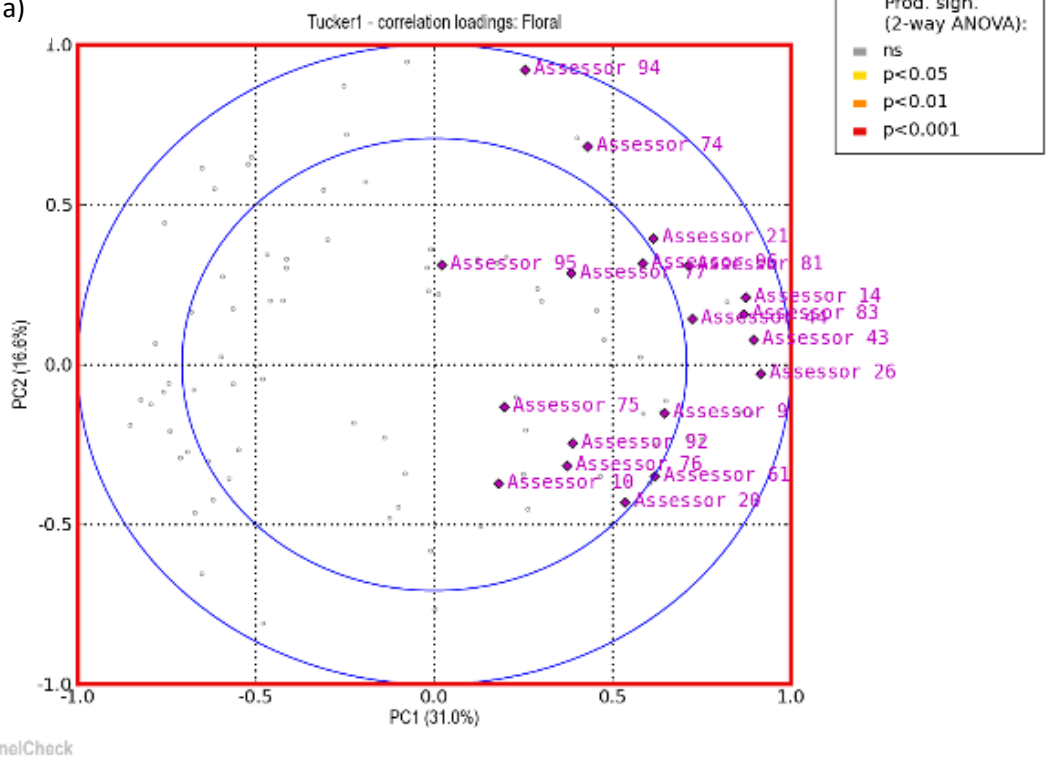
Total	+	+	+	+	=	
Eliminated due to major defect						0

Signature of juror

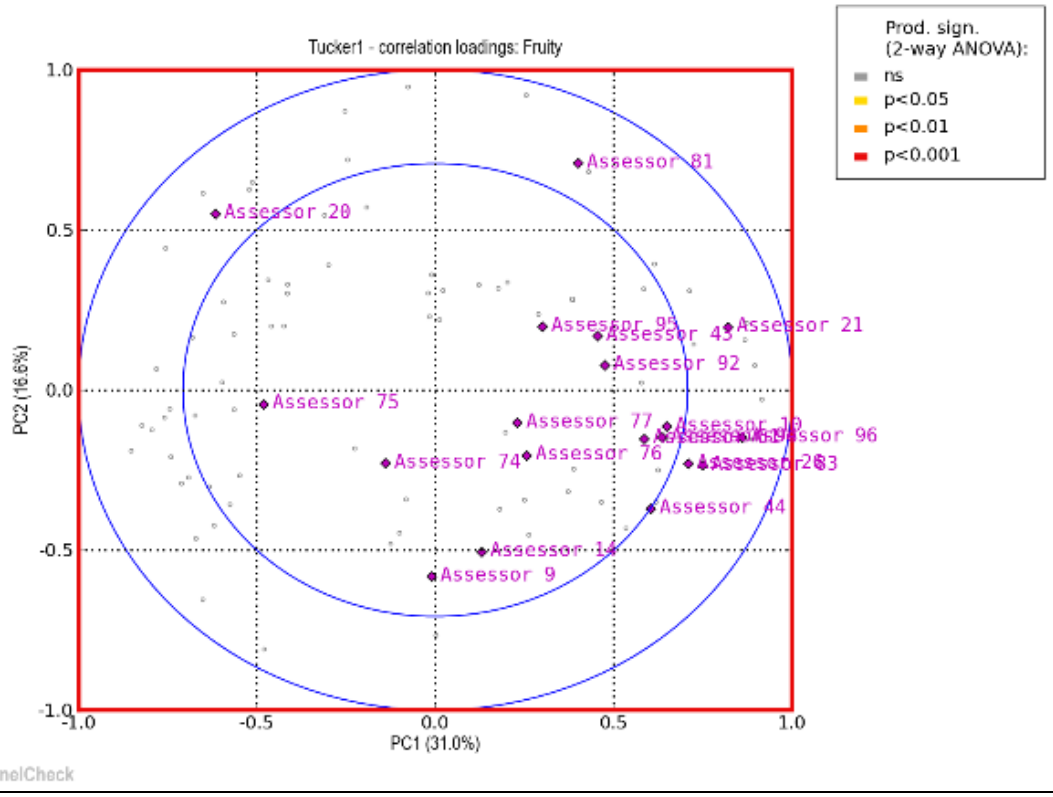
Signature of the President of the Jury

Figure A-1 OIV Spirituous beverages of Vitivinicultural Evaluation Sheet

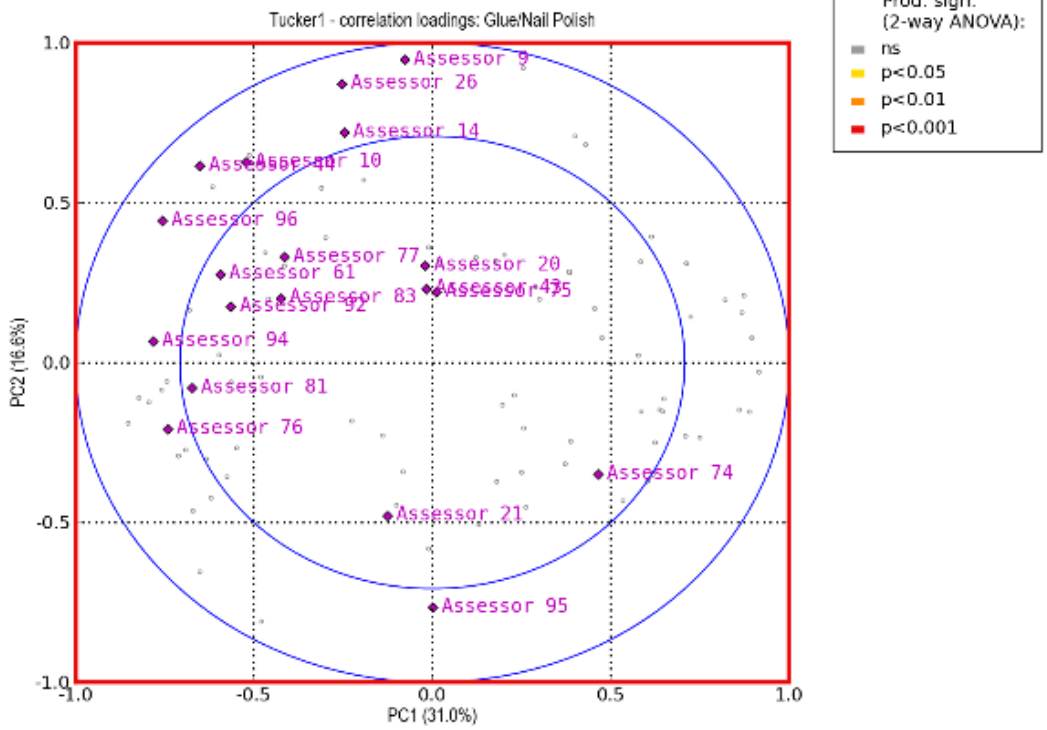
a)


Tucker1 - correlation loadings: Floral
Prod. sign. (2-way ANOVA):
ns
p<0.05
p<0.01
p<0.001
Assessor 94
Assessor 74
Assessor 21
Assessor 95
Assessor 77
Assessor 81
Assessor 14
Assessor 83
Assessor 43
Assessor 26
Assessor 75
Assessor 9
Assessor 92
Assessor 76
Assessor 10
Assessor 61
Assessor 20
PC2 (16.6%)
PC1 (31.0%)
1.0
0.5
0.0
-0.5
-1.0
PanelCheck

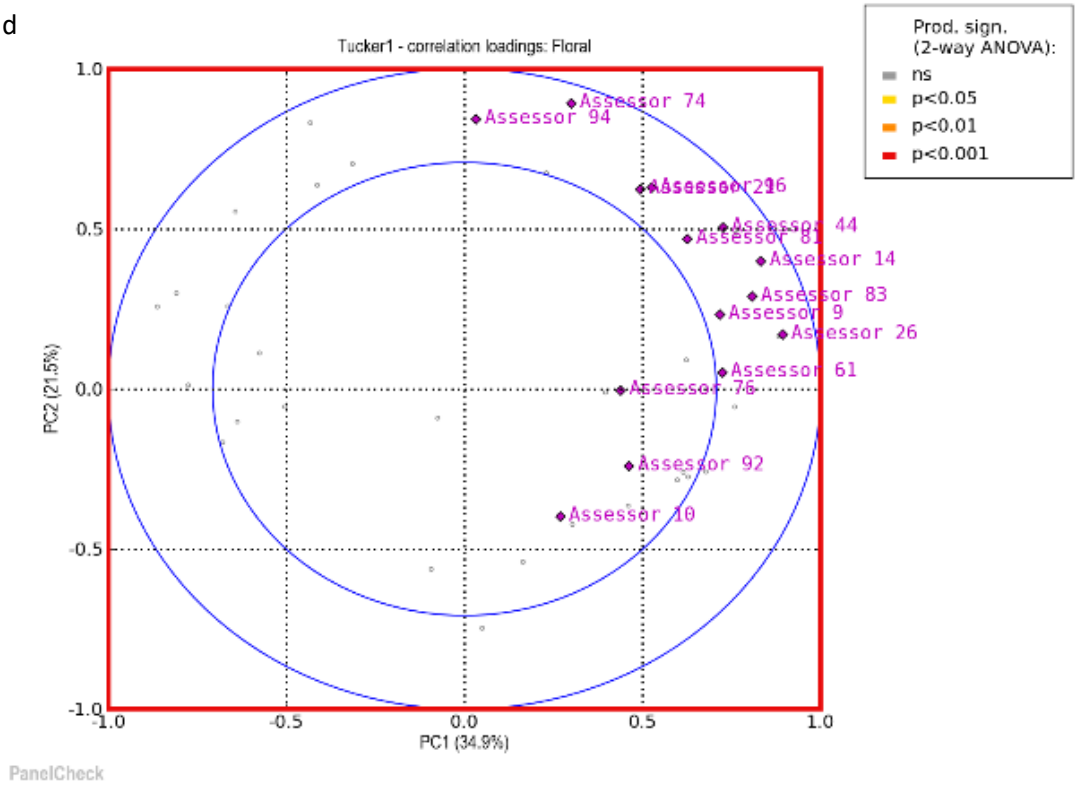
b)


Tucker1 - correlation loadings: Fruity
Prod. sign. (2-way ANOVA):
ns
p<0.05
p<0.01
p<0.001
Assessor 81
Assessor 20
Assessor 95
Assessor 43
Assessor 21
Assessor 92
Assessor 75
Assessor 77
Assessor 96
Assessor 74
Assessor 76
Assessor 44
Assessor 14
Assessor 9
PC2 (16.6%)
PC1 (31.0%)
1.0
0.5
0.0
-0.5
-1.0
PanelCheck

c


Tucker1 - correlation loadings: Glue/Nail Polish
Prod. sign. (2-way ANOVA):
ns
p<0.05
p<0.01
p<0.001
Assessor 9
Assessor 26
Assessor 14
Assessor 10
Assessor 96
Assessor 77
Assessor 20
Assessor 61
Assessor 83
Assessor 92
Assessor 94
Assessor 81
Assessor 76
Assessor 74
Assessor 21
Assessor 95
PC1 (31.0%)
PC2 (16.8%)
PanelCheck

d


Tucker1 - correlation loadings: Floral
Prod. sign. (2-way ANOVA):
ns
p<0.05
p<0.01
p<0.001
Assessor 74
Assessor 94
Assessor 14
Assessor 83
Assessor 9
Assessor 26
Assessor 61
Assessor 92
Assessor 10
PC1 (34.9%)
PC2 (21.5%)
PanelCheck

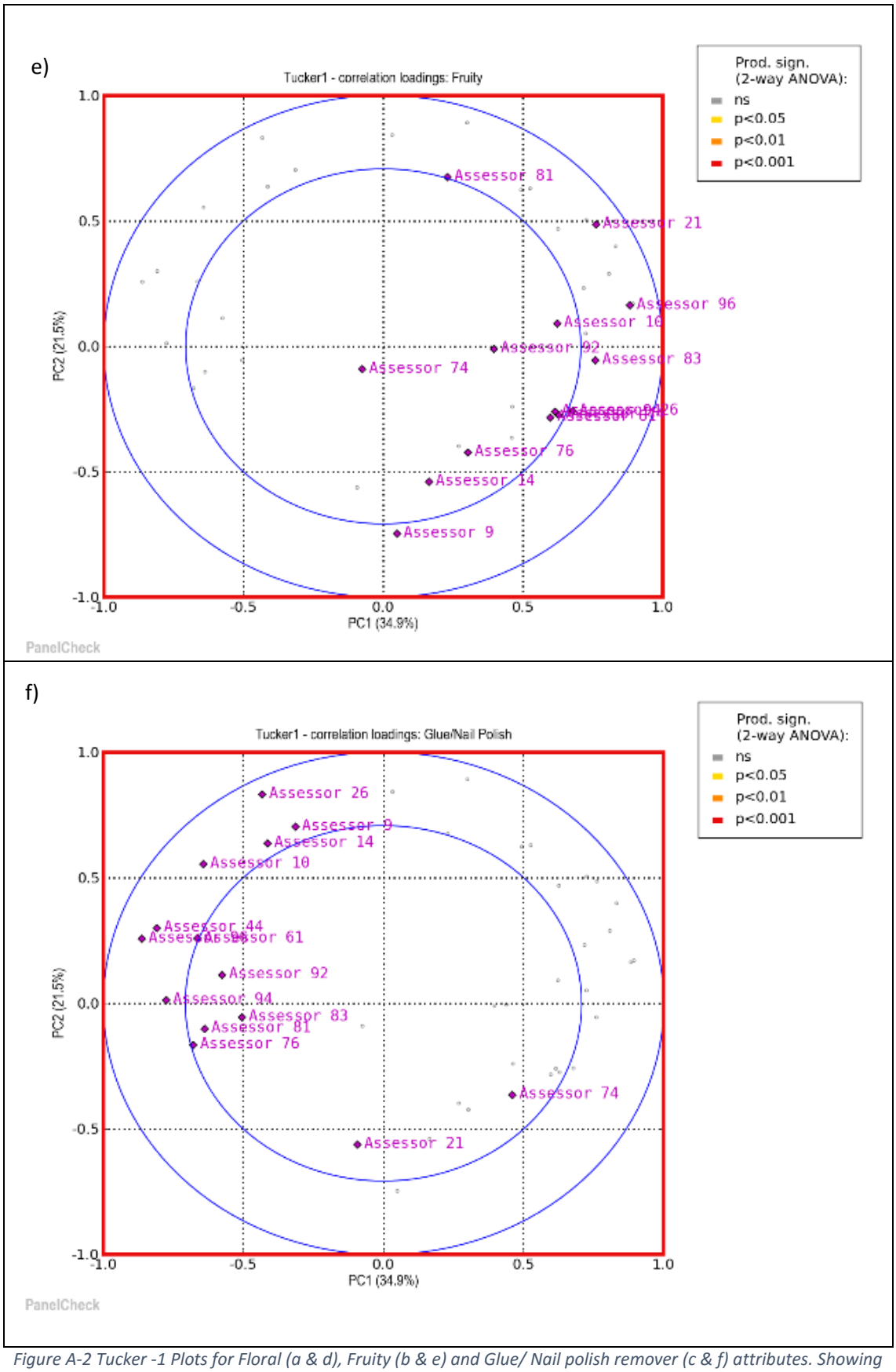


Figure A-2 Tucker -1 Plots for Floral (a & d), Fruity (b & e) and Glue/ Nail polish remover (c & f) attributes. Showing all participants (a, b, c) and showing only participants used for analysis (d, e, f).