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Instrumental and sensory characterisation of Solaris white wines in Denmark

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ABSTRACT

This study aimed to investigate the volatile and non-volatile compositions as well as sensory properties of the most common monovarietal white wine (*var. Solaris*) in Denmark. Using dynamic headspace sampling (DHS) coupled to gas chromatography–mass spectrometry (GC–MS), 79 volatile compounds were identified. Among the major non-volatile components glycerol, sulphite, sugars and organic acids were analysed. A primary sensory difference was observed among wine samples, half of which were characterised by floral and fruity flavours (peach/apricot, Muscat, melon, banana and strawberry) while the remainder were described by less pleasant flavours, such as chemical, wood and rooibos/smoke. Partial least squares regression (PLS) showed that acetates and ethyl esters of straight-chain fatty acids were associated with floral and fruity odours while ethyl esters of branched-chain fatty acids were less associated with them. The study also suggested that differences in vintage were less characteristic than differences caused due to sulphite management by producers.

1. Introduction

Denmark is an emerging wine-producing country with a rapidly growing commercial production since official approval by the EU in 2000. The Danish Vineyards Association has more than 1400 members and the number of commercial wine producers amounts to 90 (Becker & Toldam-Andersen, 2012). Although the cold climate and short growing season put certain restraints on the varieties of grapes that can be grown for wine production, some fast ripening "cold climate" varieties have been shown to provide sufficient oenological capabilities. The main grape varieties utilised at present are Rondo, Regent, Leon Millot, Solaris, Ortega and Orion (Becker & Toldam-Andersen, 2012; Lederer, Nielsen, Toldam-Andersen, Herrmann, & Arneborg, 2013). Among the varieties for white wine, Solaris has become the most dominantly cultivated. Originating in Germany, Solaris gained official varietal protection in 2001 and is now commonly planted in Europe's coldest wine regions like Scandinavia, due to its hardiness; it is formally listed as a *Vitis vinifera* cultivar but in fact it is not a pure *Vitis vinifera* since it contains several hybrid grapes in its pedigree (Gustafsson & Mårtensson, 2005; Winegrowers' supplies: vine variety information, 2008).

Numerous studies have been conducted to characterise the chemical composition of wines made from different varieties. These include Cabernet Sauvignon (Forde, Cox, Williams, & Boss, 2011; King et al., 2014), Sauvignon Blanc (Green, Parr, Breitmeyer, Valentin, & Sherlock, 2011; Parr, Schlich, Theobald, & Harsch, 2013), Malbec (Aruani et al., 2012; Fabani, Ravera, & Wunderlin, 2013), Godello (González Álvarez, González-Barreiro, Cancho-Grande, & Simal-Gándara, 2011; Losada, Andrés, Cacho, Revilla, & López, 2011) and others. However, little is known about the flavour properties and potential of wines produced from the "cold climate" grapes utilised in Denmark. No comparative characterisation studies have been reported on specific grape varieties or vinification practices. Systematic studies on the flavour characters will contribute to a rational development of wine styles, which are representative of the Danish and Nordic regions.

The characterisation of wines from a specific grape variety requires information about the composition of the volatile and non-volatile flavour components and their contribution to the sensory properties of the wine. Volatile compounds play an essential role for the flavour quality and several hundreds of volatiles from different chemical classes including alcohols, esters, acids, terpenes

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and aldehydes, etc., have been identified in wines. Also, the flavour profiles of wine depend on the balance between sugars and organic acids, which are among the major non-volatiles. To measure the volatile composition, headspace analysis followed by gas chromatograph–mass spectrometry (GC–MS) is commonly employed. The headspace contains many of the volatiles that are responsible for wine aroma while GC–MS provides an effective and precise tool for odorant separation and detection (Vilanova, Escudero, Graña, & Cacho, 2013). On the other hand, descriptive sensory analysis is considered as the primary measurement method for the sensory aspects of wine, typically with trained sensory panels examining the perceived attributes quantitatively.

The aim of the present study was to survey white wines from different commercial producers all of whom are utilising Solaris grapes for monovarietal wine production. This was a first step in obtaining an understanding of the kind of flavours present in Solaris wines and the volatiles and non-volatiles associated with them. This characterisation will serve for further studies into vinification methods and their influence on Solaris white wine flavours.

2. Materials and methods

2.1. Wine samples

Twelve white wines were selected for this study, among which eleven were commercial wines collected from representative Danish wine producers and one from an experimental production using commercial grapes. Details of these wine samples are shown in Table 1. The selection was preceded by a blind tasting of a wider range of Solaris wines by an expert panel in order to select Solaris wines spanning the range of flavours available on the market.

2.2. Oenochemical properties

Ethanol, pH, total acid, volatile acid and glycerol were measured using a WineScan FT 120 Fourier Transform Infrared Spectrophotometer (Foss, Hillerød, Denmark). Wine samples were analysed in quadruplicate and parameters were quantified using a high-input calibration file. Free and total sulphur dioxide (SO_2) were measured using a modified version of the Ripper iodine redox titration with a double titration, where the content of reductones other than sulphite were accounted for using a second titration with iodine after binding of free SO_2 with glyoxal (Tanner & Sandoz, 1972). Sugars and organic acids, including glucose, fructose, tartaric acid, succinic acid, malic acid and lactic acid, were measured by ion chromatography. A Metrohm ion-chromatography system (Metrohm AG, Herisau, Switzerland) equipped with an autosampler (919 IC Plus) ran parallel analyses, with a 20-μL injection to a 881 Compact IC Pro with a 896 Professional detector for the sugars and a 20-μL injection to a 883 Basic IC plus unit for the organic acids. The sugars were separated on a Metrosep CARB 1, 150/4.0 column at 35 °C with a 100 mM NaOH eluent (flow at 1.0 mL/min). The organic acids were separated on a Metrosep Organic Acid 250/7.8 column at room temperature with 0.4 mM H_2SO_4 + 120 mL acetone/L as eluent (flow at 0.38 mL/min). Both columns were installed with a Metrohm guard column.

2.3. Volatile composition analysis

Volatiles analysis was carried out using the laboratory's standard procedures which were performed by dynamic headspace sampling (DHS) followed by gas chromatography–mass spectrometry (GC–MS).

2.3.1. Dynamic headspace sampling (DHS)

Volatile compounds were collected in a dynamic headspace sampling (DHS) system. For each analysis, 20 mL wine were placed in a 100-mL gas washing flask equipped with a purge head, and 1.00 mL internal standard (50 μL/L 4-methyl-1-pentanol (Aldrich, Steinheim, Germany) in water) was added in each sample. A trap containing Tenax-TA (250 mg, mesh size 60/80; Buchem BV, Apeldoorn, The Netherlands) was attached to the purge head. The flask containing the sample was immersed in a laboratory water bath and held at 37 °C. Under magnetic stirring (200 rpm), the sample was then purged with nitrogen (100 mL/min) for 20 min to collect the volatiles. The traps were dry purged with nitrogen (100 mL/min) for 15 min to remove excess water trapped during purging. Sealed Tenax-TA traps with caps were kept at 5 °C before GC–MS analysis. The dynamic headspace collection was carried out in duplicate for all wines.

2.3.2. Gas chromatography–mass spectrometry (GC–MS)

The collected volatiles were thermally desorbed using an automatic thermal desorption unit (ATD 400; Perkin Elmer, Waltham, MA). The primary desorption was carried out at 250 °C (15 min) to a cold trap (30 mg Tenax TA, 5 °C), with a hydrogen flow of 50 mL/min. Volatiles were desorbed from the cold trap onto the GC column by heating to 300 °C for 4 min (secondary desorption), using a split ratio of 1:10. The volatiles were transferred through a heated transfer-line (225 °C) to a gas chromatograph–mass spectrometer (GC–MS, 7890A GC-system interfaced with a 5975C VL MSD with triple-axis detector from Agilent Technologies, Palo Alto, CA) equipped with a J&W Scientific DB-Wax column (30 m × 0.25 mm × 0.25 μm) and using helium as carrier gas (1 mL/min). The column temperature was kept at 40 °C for 10 min, increased at 8 °C/min to 240 °C, and kept isothermal for 5 min. The mass selective detector was in electron impact mode (70 eV). Mass spectra were obtained at a mass/charge (m/z) range between 15 and 300.

Table 1
List and details of the Solaris wines used in the study.

Wine code	Winery	Vintage	Winery location	Note
SKA_12	Skærsøgaard Vin	2012	South-east of Jutland	–
GAL_11	Galsgaard Vin	2011	South of Zealand	–
DYR_12	Dyrehøj Vingaard	2012	West coast of Zealand	–
ORN_11	Ørnberg	2011	West coast of Zealand	–
ORN_12	Ørnberg	2012	West coast of Zealand	–
DKU_11	Experimental wine	2011	North coast of Zealand	Grapes were from Domain Aalsgaard, and the wines were made in the experimental winery at the University of Copenhagen (KU)
DEG_08	Degnemosegaard	2008	Central Zealand	–
DEG_10	Degnemosegaard	2010	Central Zealand	–
VAN_10	Vexebo Vin, Annisse Winery	2010	Northern Zealand	Organic grapes were from Vexebo Vin, and the wines were made in Annisse Winery
VAN_11	Vexebo Vin, Annisse Winery	2011	Northern Zealand	Organic grapes were from Vexebo Vin, and the wines were made in Annisse Winery
MEO_11	Meonia	2011	Island of Møn	Organic producer
MEO_12	Meonia	2012	Island of Møn	Organic producer

GC–MS data processing was carried out using MSD Chemstation G1701EA software (Version E.01.00.237, Agilent Technologies). Volatile compounds were identified by verifying their mass spectra with those in a reference database (Wiley275.L, G1035A, Agilent Technologies, Inc.). Additionally, linear retention indices of all identified volatiles were calculated as the retention time of the volatile normalised to the retention times of adjacently eluting *n*-alkanes, and compared to reported retention indices to further support the identifications. All identified compounds were semi-quantified as peak areas in the total ion chromatogram (TIC).

2.4. Sensory evaluation

2.4.1. Panel

The sensory profiling was performed in the sensory laboratory of Sensory and Consumer Science section, University of Copenhagen. The panel was composed of seven females and three males aged 24-42 years (mean age = 28). All of them had at least two years prior experience in the sensory evaluation of foods. The sensory assessors were paid for their participation.

2.4.2. Panel training

Four 2-h specific training sessions were performed. In the first session, the twelve wines as well as a list of attributes based on Noble's aroma wheel and Jackson's "Wine Tasting" were provided to assessors (Jackson, 2009; Noble et al., 1987). They were asked to describe the wines by selecting attributes from the list or they could generate new ones. A new list of 103 attributes, including all attributes mentioned by the assessors, was thus generated. In the second session, an aroma kit was first presented to the panel in order to help them to become familiar with their attributes. Subsequently, the assessors judged the twelve wines by rating the attributes of the list from 0 (not present) to 5 (very intense). The data were processed with the formula proposed by Dravnieks (1985) to calculate the "geometric means" (*GM*) for each attribute; $GM = (F \times I)^{0.5}$, where *F* (%) is the detection frequency of an attribute expressed as percentage of total number of assessors ($n = 10$) and *I* (%) is the average intensity expressed as percentage of the maximum intensity. The *GM* (%) parameter, which can range from 0 to 100, makes it possible to take into account aroma attributes which were rarely mentioned but very important in terms of the perceived intensity, and also the attributes with a low perceived intensity but which were mentioned often by the assessors. A reduced list of 34 attributes was then produced by comparison of GM values and a further attribute refining was carried out in the third training session, resulting in a final list of 24 attributes. In the fourth session, different reference standards prepared based on Noble's method (1987) were presented to the panel, discussed, and modified to fit the group's idea of the odours and tastes evoked by these attributes. The list of attributes along with their reference compositions is presented in Table 2. The assessors then judged the wines using an unstructured, 15-cm linear scale, anchored "not perceived" at the left end and "very intense" at the right end.

2.4.3. Descriptive analysis

The twelve wines were evaluated in triplicate in three formal sessions that were held on different days. Each evaluation was conducted in individual tasting booths at room temperature (22 ± 1 °C). In each case, a volume of 30 mL was served at 12 ± 1 °C in standardised wineglasses (ISO-3591, 1977), which were coded with 3-digit numbers and covered by glass Petri dishes. In order to minimise first-order effects, carry-over effects and memory biases, all wine samples were presented in a different order specific to each assessor according to a Williams Latin-square arrangement generated by FIZZ software (Biosystems, Courtenon, France). The assessors scored each attribute for all the wine samples using the 15-cm linear scale. Water and plain crackers were provided for rinsing between wine tasting. Two 10-min breaks were enforced in the process of each session to limit fatigue. New bottles of wine were opened for each training and evaluation session.

2.5. Data analysis

The statistical analyses on volatiles were performed with the peak areas of each compound. To test significant differences among wine volatiles, a one-way analysis of variance (ANOVA) in which wine was the factor was carried out using the software JMP (version 7.0, SAS Institute Inc.). For the sensory data, ANOVA was run with the step function of the lmerTest package (2012) using the R software (version 2.14.2). Principal component analysis (PCA) and partial least squares regression (PLS) were employed in the multivariate analysis. PCA was used to relate the different volatile compounds and/or sensory attributes of wines, and also to identify the specific factors leading to the greatest variability. PLS was applied to correlate instrumental compositions including volatiles, sugars and organic acids (*X*-matrix) to the sensory attributes with significant differences among wine samples (*Y*-matrix). Both PCA and PLS were run using the Unscrambler version 9.7 (CAMO ASA, Oslo, Norway). The data were centred and standardised (1/Sdev), and the models were validated by full cross-validation.

3. Results and discussion

3.1. Oenochemical analysis of wines

Table 3 presents the oenochemical compositions of the twelve Solaris wines. All wines had a suitable alcoholic grade for this type of white wine, ranging from 10.0% to 13.0%, although 10.0% is a low level for the cultivar. The levels of total acid and volatile acid ranged from 5.5 to 9.6 g/L and 0.2 to 0.4 g/L, respectively. Glycerol, ranging between 4.3 and 9.4 g/L, contributed to the viscosity and softness of the wine with a positive effect on its taste at these concentrations. Most wines had a low level of glucose and fructose with the exception of DYR_12 and to a lesser degree SKA_12, ORN_11, and DEG_10. The primary grape-derived acid, tartaric acid, as well as succinic acid, were found at a moderate level between 1.5 and 3.7 g/L, 0.3 and 0.8 g/L, respectively. The analysis of malic and lactic acid contents indicated degree of lactic acid bacteria in the wines. The wines from the organic wineries Vexebo & Annisse and Meonia had gone through a malolactic fermentation for their two vintage productions. All the other wineries allowed an alcoholic fermentation only. The general levels of acids were moderate to low considering the cool climate, which documented the good adaptation of the cultivar to the cool Scandinavian climate (Becker & Toldam-Andersen, 2012). The levels of free and total SO_2 were, in most cases, normal for white wines and sufficient for protecting the wine; however, the wines from Meonia had no detectable SO_2 except low level of total SO_2 in the year 2011. The lack of sulphite addition indicates that the detected malolactic fermentation may have been of a spontaneous character. Degnemosegaard also produced wines with very low levels of SO_2. Insufficient sulphite levels most likely were not able to protect the wines well or provide microbiological stability, thus risking off-flavour development and/or resulting in limited storability.

3.2. Volatile composition of wines

GC–MS analysis allowed the identification of 79 volatile compounds in the wines (Table 4). The volatiles belonged to nine different classes, namely: esters (34), higher alcohols (22),

Table 2
List of sensory attributes and reference standards used in the study.

Attributes	Reference compositions*
Odour	
Floral/elderflower	12 mL elderflower juice
Citrus	2 mL each of fresh grapefruit and lemon juice and some peels
Grapefruit	10 mL fresh grapefruit juice and 2 g grated peels in 10 ml white wine
Lemon	5 mL fresh lemon juice and some peels
Peach/apricot	1 drop each of samples 20 (peach) and 19 (apricot) of "Le Nez du Vin" Jean Lenoir
Green apple	5 mL fresh green apple juice and grated fruit
Muscat	1 drop sample 8 (Muscat) of "Le Nez du Vin" Jean Lenoir
Melon	2 cm^2 piece of fresh ripe honey melon
Banana	1 cm^2 piece of fresh chopped banana
Strawberry	5 mL fresh strawberry juice and 20 g chopped fruit
Prune	6 g chopped prune
Rooibos/smoke	0.25 g rooibos tea
Wood	2 g wood shavings and chip board
Vanilla	1 drop sample 40 (vanilla) of "Le Nez du Vin" Jean Lenoir
Cut grass	1 cm shredded blade of green grass
Mushroom	8 g fresh chopped mushroom in 10 mL wine
Black pepper	pinch of crushed black pepper
Cheese	½ cm^2 fresh chopped mild cheese
Chemical	magic marker pen (Sprittusch, containing xylene and toluene as solvents)
Taste	
Acidic	15 mL white wine vinegar in 60 ml water
Sweet	30 g sucrose in 1 L water
Bitter	0.15 g quinine sulphate in 1 L water
Astringent	1 g citric acid in 1 L water (astringent aspect of the solution)
Alcoholic	3 mL of 95% ethanol

* In 25 mL Pinot Blanc, 2010, 750 mL bottle wine (11.5% v/v) per glass, unless otherwise specified.

Table 3
Oenochemical compositions in the Solaris wines.

	SKA_12	GAL_11	DYR_12	ORN_11	ORN_12	DKU_11	DEG_08	DEG_10	VAN_10	VAN_11	MEO_11	MEO_12	Significance
Ethanol (% v/v)	11.64^{g}	11.17^{i}	11.38^{h}	13.15^{a}	12.12^{f}	12.89^{b}	12.76^{c}	11.46^{h}	12.31^{d}	12.79^{c}	10.01^{j}	12.20^{e}	***
pH	3.38^{b}	3.12^{d}	3.40^{b}	3.31^{c}	3.26^{c}	3.50^{a}	3.13^{d}	2.83^{g}	3.25^{c}	3.39^{b}	2.98^{e}	3.04^{f}	***
Total acid (g/L)	7.16^{f}	9.20^{b}	6.38^{h}	6.98^{g}	8.05^{c}	6.31^{i}	7.30^{e}	9.57^{a}	5.57^{j}	5.51^{k}	7.04^{g}	7.47^{d}	***
Volatile acid (g/L)	0.41^{a}	0.44^{a}	0.18^{d}	0.34^{c}	0.30^{c}	0.27^{c}	0.13^{e}	0.18^{d}	0.19^{d}	0.28^{c}	0.18^{d}	0.35^{b}	***
Glycerol (g/L)	7.95^{b}	9.42^{a}	5.41^{g}	6.59^{e}	5.31^{gh}	6.98^{d}	7.26^{c}	5.21^{h}	4.82^{i}	6.23^{f}	4.26^{j}	4.92^{i}	***
Free SO_2 (mg/L)	17^{g}	19^{f}	63^{a}	34^{d}	39^{b}	37^{c}	3^{h}	1^{i}	23^{e}	39^{b}	0^{j}	0^{j}	***
Total SO_2 (mg/L)	138^{d}	154^{c}	221^{a}	177^{b}	137^{d}	143^{d}	51^{g}	18^{h}	66^{f}	124^{e}	56^{g}	0^{i}	***
Glucose (g/L)	1.37^{c}	0.34^{fg}	7.28^{a}	0.76^{d}	0.22^{fg}	0.35^{f}	0.58^{e}	2.70^{b}	0.19^{gh}	0.16^{gh}	0.04^{i}	0.12^{hi}	***
Fructose (g/L)	4.76^{b}	1.17^{e}	7.84^{a}	4.77^{b}	2.15^{d}	0.42^{g}	0.09^{h}	2.40^{c}	0.04^{h}	0.10^{h}	0.01^{h}	0.70^{f}	***
Tartaric acid (g/L)	1.85^{e}	3.60^{a}	1.47^{f}	1.91^{e}	3.05^{b}	1.69^{ef}	2.67^{cd}	3.51^{a}	2.81^{bc}	2.42^{d}	3.70^{a}	3.55^{a}	***
Succinic acid (g/L)	0.7	0.8	0.4	0.3	0.4	0.7	0.6	0.3	0.4	0.5	0.5	0.6	ns
Malic acid (g/L)	3.51^{b}	2.55^{d}	3.80^{a}	2.95^{c}	3.23^{b}	3.37^{b}	1.92^{e}	3.40^{b}	0.07^{f}	0.20^{f}	0.00^{f}	0.00^{f}	***
Lactic acid (g/L)	0.25^{fg}	0.29^{fg}	0.17^{g}	0.19^{g}	0.91^{e}	0.37^{f}	0.18^{g}	0.00^{h}	2.04^{d}	2.75^{b}	2.21^{c}	3.20^{a}	***

Values with different superscript letters (a–k) within each row are significantly different, according to the Student's *t*-test ($p < 0.05$).
*** Indicates significance at $p < 0.001$, ns means no significant difference between the wines.

aldehydes (8), ketones (3), fatty acids (4), terpenes (4), sulphur compounds (2), acetals (1) and C13-norisoprenoid (1). The majority of volatile compounds were found in all of the wines but at varying levels.

3.2.1. Esters

Esters were the most prevalent class in terms of the number of volatiles in the Solaris wines. Most of them were ethyl esters of fatty acids typically produced enzymatically during yeast fermentation and from ethanolysis of acetyl-CoA that is formed during fatty acids synthesis or degradation (Perestrelo, Fernandes, Albuquerque, Marques, & Câmara, 2006). As can be seen in Table 4, high levels of ethyl lactate and diethyl succinate were found in wines from Vexebo & Annisse and Meonia, which had undergone malolactic fermentation. The esterification of lactic acid and succinic acid with ethanol is typically occurring during malolactic fermentation as also shown in other studies (Gomez Garcia-Carpintero, Gomez Gallego, Sanchez-Palomo, & Gonzalez Vinas, 2012; Izquierdo Cañas, García Romero, Gómez Alonso, & Palop Herreros, 2008). Furthermore, diethyl succinate presented a high level in the two wines without malolactic fermentation (DEG_08 & DEG_10), which might be due to the fact that the lack of sulphite stabilisation had caused an accelerated ageing of the wines. Acetates are products of the reaction of acetyl-CoA with higher alcohols that are formed from degradation of amino acids or carbohydrates (Perestrelo et al., 2006). 3-Methylbutyl acetate and hexyl acetate showed the largest peak areas among the acetates. In addition, methyl hexanoate, butyl butanoate, 3-methylbutyl butanoate, methyl octanoate, 3-methylbutyl octanoate and methyl salicylate were also observed in the Solaris wines apart from the ethyl esters and acetates. Butyl butanoate was the only ester solely found in DYR_12. All esters showed significantly different levels among the wine samples ($p < 0.001$).

3.2.2. Higher alcohols

Alcohols were the second largest group of identified volatiles in our study. They are typically formed by yeast via the anabolic pathway from glucose or catabolic pathway from their corresponding

Table 4
Volatile compounds identified in the Solaris wines.

Code	Compounds	Calculated LRI	Reported LRI[a]	ID[d]	Odour description[e]	Mean peak area/10^5	Standard deviation/10^5	Sig.[m]
Esters								
	Ethyl esters							
e1	Ethyl acetate	898	907	MS + LRI	Pineapple	4300	1500	***
e2	Ethyl propanoate	958	951	MS + LRI	Fruit	320	110	***
e3	Ethyl isobutyrate	967	955	MS + LRI	Sweet, rubber	300	200	***
e4	Ethyl butyrate	1046	1028	MS + LRI	Apple	980	340	***
e5	Ethyl 2-methylbutanoate	1064	1050	MS + LRI	Apple	48	35	***
e6	Ethyl 3-methylbutanoate	1080	1060	MS + LRI	Fruit	75	56	***
e7	Ethyl pentanoate	1148	1133	MS + LRI	Yeast, fruit	2.2	0.6	***
e8	Ethyl (*E*)-2-butenoate	1173	1152	MS + LRI	–	24	6.6	***
e9	Ethyl hexanoate	1257	1220	MS + LRI	Apple peel, fruit	2100	790	***
e10	Ethyl pyruvate	1287	1242	MS + LRI	Herbaceous, oil painting, forage[f]	21	22	***
e11	Ethyl (*E*)-3-hexenoate	1324	1301	MS + LRI	–	1.5	0.8	***
e12	Ethyl lactate	1363	1358	MS + LRI	Fruit	1800	1700	***
e13	Ethyl octanoate	1449	1436	MS + LRI	Fruit, fat	1800	730	***
e14	Diethyl malonate	1591	1576	MS + LRI	Apple	0.8	0.5	***
e15	Ethyl 2-furoate	1639	1618[b]	MS	–	8.1	6.0	***
e16	Ethyl decanoate	1651	1636	MS + LRI	Grape	420	330	***
e17	Diethyl succinate	1692	1689	MS + LRI	Wine, fruit	210	200	***
e18	Ethyl 9-decenoate	1705	1694	MS + LRI	Fruit[g]	3.2	3.5	***
e19	Ethyl dodecanoate	1797	1842	MS + LRI	Leaf	15	22	***
	Acetate esters							
ac1	Propyl acetate	978	969	MS + LRI	–	180	180	***
ac2	2-Methylpropyl acetate	1020	1015	MS + LRI	Fruit, apple, banana	370	230	***
ac3	Butyl acetate	1084	1075	MS + LRI	Pear	19	19	***
ac4	3-Methylbutyl acetate	1136	1117	MS + LRI	Banana	7100	5400	***
ac5	Pentyl acetate	1185	1180[c]	MS	–	5.2	6.8	***
ac6	Hexyl acetate	1292	1270	MS + LRI	Fruit, herb	870	1200	***
ac7	(*Z*)-3-Hexenyl acetate	1327	1327	MS + LRI	Green, banana	14	16	***
ac8	Heptyl acetate	1387	1366	MS + LRI	Almond, pear[h]	0.7	1.4	***
ac9	2-Phenylethyl acetate	1798	1829	MS + LRI	Rose, honey, tobacco	130	120	***
	Other esters							
oe1	Methyl hexanoate	1195	1188	MS + LRI	Fruit, fresh, sweet	5.7	4.1	***
oe2	Butyl butanoate	1241	1223[c]	MS	–	1.3	4.2	***
oe3	3-Methylbutyl butanoate	1284	1267[c]	MS	–	2.2	0.8	***
oe4	Methyl octanoate	1401	1389	MS + LRI	Orange	3.7	2.5	***
oe5	3-Methylbutyl octanoate	1671	1658	MS + LRI	–	4.0	2.0	***
oe6	Methyl salicylate	1800	1745	MS + LRI	Peppermint	1.0	1.0	***
Alcohols								
alc1	1-Propanol	1059	1037	MS + LRI	Alcohol, pungent	22	13	ns
alc2	2-Methyl-1-propanol	1110	1099	MS + LRI	Wine, solvent, bitter	730	330	***
alc3	2-Pentanol	1141	1118	MS + LRI	Green	3.0	3.4	***
alc4	1-Butanol	1164	1145	MS + LRI	Medicine, fruit	53	30	***
alc5	3-Methyl-1-butanol	1236	1205	MS + LRI	Whiskey, malt, burnt	15000	2600	***
alc6	3-Methyl-3-buten-1-ol	1271	1263[c]	MS	–	2.2	0.3	ns
alc7	2-Heptanol	1342	1273	MS + LRI	Mushroom	2.7	1.5	***
alc8	3-Methyl-1-pentanol	1348	1325[c]	MS	Vinous, herbaceous, cocoa[i]	22	9.3	***
alc9	1-Hexanol	1373	1360	MS + LRI	Resin, flower, green	1600	570	***
alc10	(*E*)-3-Hexen-1-ol	1381	1386	MS + LRI	Moss, fresh	21	12	***
alc11	3-Ethoxy-1-propanol	1392	1409	MS + LRI	Chemical[j]	2.4	3.1	***
alc12	(*Z*)-3-Hexen-1-ol	1398	1391	MS + LRI	Grass	6.3	3.6	***
alc13	(*E*)-2-Hexen-1-ol	1421	1400	MS + LRI	Green, leaf, walnut	3.8	6.3	***
alc14	(*Z*)-2-Hexen-1-ol	1430	1410	MS + LRI	Leaf, green, wine, fruit	2.7	1.8	***
alc15	1-Heptanol	1470	1467	MS + LRI	Chemical, green	6.3	3.1	***
alc16	2-Ethyl-1-hexanol	1503	1487	MS + LRI	Rose, green	3.8	2.9	***
alc17	2,3-Butanediol	1556	1523	MS + LRI	Fruit, onion	19	10	ns
alc18	1-Octanol	1572	1553	MS + LRI	Chemical, metal, burnt	6.1	1.8	***
alc19	1-Nonanol	1675	1668[c]	MS	Fat, green	1.0	0.4	**
alc20	1-Decanol	1777	1765	MS + LRI	Fat	1.4	0.7	***
alc21	Benzyl alcohol	1795	1865	MS + LRI	Sweet, flower	0.9	0.4	ns
alc22	2-Phenylethanol	1793	–	MS	Honey, spice, rose, lilac	290	160	***
Aldehydes								
ald1	3-Methylbutanal	917	910	MS + LRI	Malt	12	3.0	ns
ald2	Hexanal	1088	1084	MS + LRI	Grass, tallow, fat	4.1	3.0	ns
ald3	Heptanal	1194	1174	MS + LRI	Fat, citrus, rancid	0.6	0.9	ns
ald4	Octanal	1306	1280	MS + LRI	Fat, soap, lemon, green	1.2	1.1	ns
ald5	Nonanal	1405	1385	MS + LRI	Fat, citrus, green	2.6	2.2	ns
ald6	2-Furfural	1478	1455	MS + LRI	Bread, almond, sweet	1.9	1.8	**
ald7	Decanal	1512	1484	MS + LRI	Soap, orange peel, tallow	1.2	0.8	ns
ald8	Benzaldehyde	1541	1495	MS + LRI	Almond, burnt sugar	2.7	4.1	***
Ketones								
k1	2-Heptanone	1190	1170	MS + LRI	Soap	6.4	4.5	***

(*continued on next page*)

Table 4 (*continued*)

Code	Compounds	Calculated LRI	Reported LRI[a]	ID[d]	Odour description[e]	Mean peak area/10^5	Standard deviation/10^5	Sig.[m]
k2	3-Hydroxy-2-butanone	1303	1287	MS + LRI	Butter, cream	6.8	6.7	***
k3	6-Methyl-5-hepten-2-one	1354	1340	MS + LRI	Pungent[k]	2.8	1.7	ns
Acids								
acid1	Acetic acid	1465	1450	MS + LRI	Sour	39	29	ns
acid2	Butyric acid	1655	1619	MS + LRI	Rancid, cheese, sweat	3.2	1.8	***
acid3	Hexanoic acid	1796	1829	MS + LRI	Sweat	21	14	***
acid4	Octanoic acid	1786	–	MS	Sweat, cheese	27	21	**
Terpenes								
t1	Neroloxide	1486	1479	MS + LRI	Oil, flower	11	11	***
t2	Linalool	1560	1537	MS + LRI	Flower, lavender	3.2	3.5	***
t3	Hotrienol	1623	1623	MS + LRI	Hyacinth	6.7	3.9	**
t4	α-Terpineol	1717	1688	MS + LRI	Oil, anise, mint	0.8	0.4	**
Other compounds								
ot1	2,4,5-Trimethyl-1,3-dioxolane	942	956	MS	–	26	43	***
ot2	*S*-methyl thioacetate	1054	-	MS	Rotten, cooked vegetables, sulphurous[l]	7.0	5.8	***
ot3	2-Methyldihydro-3(2*H*)-thiophenone	1547	1506	MS	Cabbage, onion, must	12	13	***
ot4	β-Damascenone	1797	1813	MS + LRI	Apple, rose, honey	1.6	1.0	***

[a] Linear retention indices (LRI) reported in Flavournet and Pherobase for DB-Wax capillary GC column.
[b] Duarte et al. (2010).
[c] Bianchi, Careri, Mangia, and Musci (2007).
[d] Identification method: MS, mass spectrum agrees with mass spectrum in the database and/or reference standard; LRI, linear retention index is in close range of the reference standard run on a similar column or reported in Flavournet/Pherbase.
[e] Odour description based on Flavournet.
[f] Duarte et al. (2010).
[g] Bordiga et al. (2013).
[h] Jiang et al. (2013).
[i] Gomez Garcia-Carpintero et al. (2012).
[j] Tao and Zhang (2010).
[k] Riu-Aumatell et al. (2011).
[l] Moreira, Guedes de Pinho, Santos, and Vasconcelos (2010).
[m] *, ** and ***Indicate significance at $p < 0.05$, $p < 0.01$ and $p < 0.001$, respectively; ns means no significant difference between the wines.

amino acids (valine, leucine, isoleucine and phenylalanine) (Jiang, Xi, Luo, & Zhang, 2013). With the exception of 2-pentanol and (*E*)-2-hexen-1-ol, all alcohols were detected in each of the twelve wine samples. Significant differences were observed among all alcohols except for 1-propanol, 3-methyl-3-buten-1-ol, 2,3-butanediol and benzyl alcohol. 3-Methyl-1-butanol, a typical catabolic breakdown product of leucine, was the higher alcohol with the largest peak area. This was coincident with other studies on different grape varieties where 3-methyl-1-butanol also exhibited the highest level among alcohols (Liang, Chen, Reeves, & Han, 2013; Noguerol-Pato, Gonzalez-Alvarez, Gonzalez-Barreiro, Cancho-Grande, & Simal-Gandara, 2012). 1-Hexanol, 2-methyl-1-propanol and 2-phenylethanol also showed obviously high prevalence in the Solaris wines. 1-Hexanol is a C6 alcohol which when found in high concentration can have a negative effect on the quality of the wine, due to a vegetable and herbaceous odour. 2-Methyl-1-propanol is produced as the transamination product of valine (Sun et al., 2013), while 2-phenylethanol is the most important phenol-derived higher alcohol. These four alcohols had high peak areas in wines DEG_08, DEG_10, VAN_10, VAN_11, MEO_11 and MEO_12, indicating a risk of concentrations high enough to induce pungent smell and taste.

3.2.3. Aldehydes and ketones

Aldehydes and ketones are considered to be produced from the direct oxidation of their corresponding alcohols, or from the oxidative degradation of amino acids and sugars (Weldegergis et al., 2011). Of the aldehydes, only 2-furfural and benzaldehyde were found to vary significantly between the wine samples. The most mature wine (DEG_08) contained the highest level of 2-furfural. Increasing levels of 2-furfural with ageing was also observed in other studies (Gomez Garcia-Carpintero et al., 2012; Noguerol-Pato et al., 2012). The possible degradation product of leucine, 3-methylbutanal, was identified as the most abundant aldehyde. This again emphasised the significance of the catabolism of leucine in the Solaris wine fermentation.

Of the three ketones identified in the wines, 6-methyl-5-hepten-2-one, contributing with pungent notes, did not significantly differ in quantity among wines. The levels of 3-hydroxy-2-butanone (acetoin) were distinctly high in the wines that had undergone malolactic fermentation (VAN_10, VAN_11, MEO_11 and MEO_12). This corroborated with the fact that acetoin is formed by the activity of lactic acid bacteria and yeasts (Noguerol-Pato et al., 2012).

3.2.4. Fatty acids

Four different fatty acids were identified in the present study. Among them, acetic acid showed the highest peak area and no significant difference was found among wines. High levels of acetic acid could impart a vinegar off-odour so it must thus be kept at low levels. Butyric acid, hexanoic acid and octanoic acid have been reported to result in cheese, sweat, and rancid notes (Gomez Garcia-Carpintero et al., 2012), and contribute to freshness and equilibrate the fruity aromas of wines (Sun et al., 2013). In our study, DYR_12 possessed the highest level of butyric acid and ORN_12 had the most hexanoic acid and octanoic acid.

3.2.5. Terpenes

Among the four terpenes identified, neroloxide was found at the highest level in the most mature wine (DEG_08). The terpene alcohols linalool, hotrienol and α-terpineol are often considered as possible impact odorants in white wines since they have low odour

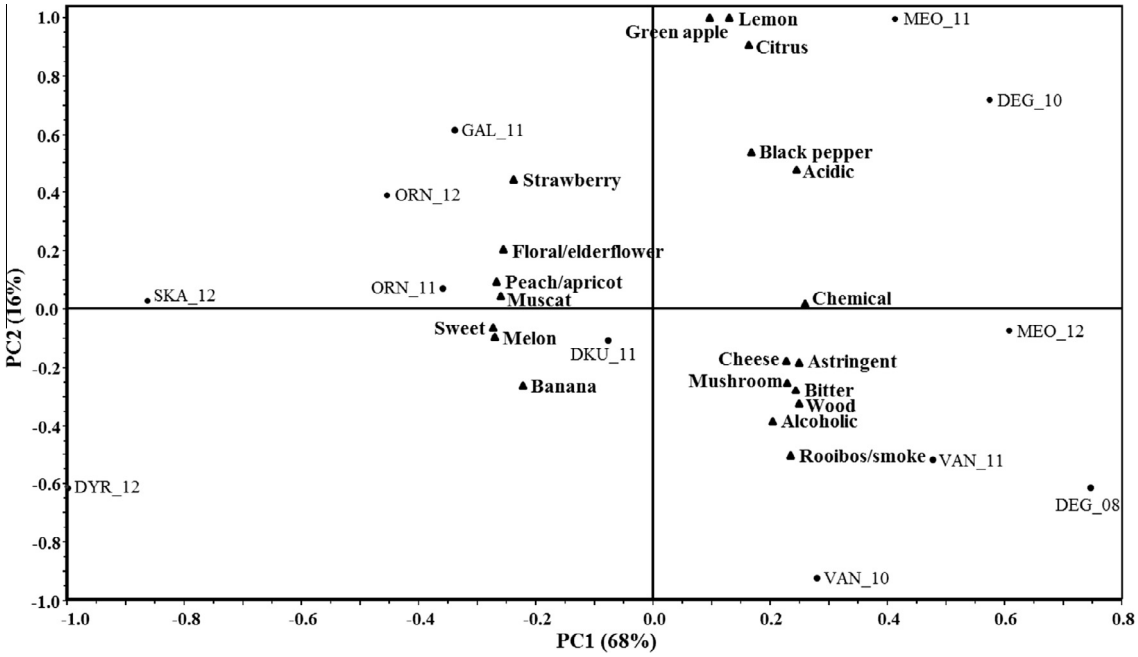

Fig. 1. PCA bi-plot of sensory attributes for the Solaris wines.

thresholds and might contribute to aroma even when present in low amounts (Welke, Manfroi, Zanus, Lazarotto, & Alcaraz Zini, 2012). DEG_08 wine had the largest level of hotrienol and α-terpineol, but showed a quite low level of linalool. Changes in concentration and the formation of new compounds might occur during wine ageing due to acid-catalysed rearrangements (Skouroumounis & Sefton, 2000; Williams, Strauss, Wilson, & Massy Westropp, 1982b; Williams, Strauss, Wilson, & Massy-Westropp, 1982a). Apparently, Solaris grapes did not provide a large number of terpenes to the wine. However, the significance of terpenes in the Solaris wine flavour may need more detailed study.

3.2.6. Other compounds

One acetal, one C13-norisoprenoid and two sulphur compounds were identified and all of them showed significant differences ($p < 0.001$) among the wines. The acetal of acetaldehyde and 2,3-butanediol, 2,4,5-trimethyl-1,3-dioxolane, has previously been reported in other wine varieties (Baumes, Cordonnier, Nitz, & Drawert, 1986; Schreier, Drawert, & Winkler, 1979), where it was shown to be responsible for the oxidised aromatic character of wines. The highest levels of this compound were observed in both MEO_11 and MEO_12, followed by DEG_08 and DEG_10. The only C13-norisoprenoid detected in the Solaris wine, β-damascenone, has been reported to be an influential contributor to the wine aroma, due to its quite low odour threshold value (Ferreira, Ortín, Escudero, López, & Cacho, 2002; Kotseridis & Baumes, 2000; López, Ferreira, Hernández, & Cacho, 1999). The identified sulphur compound *S*-methyl thioacetate was observed in high levels in the six wines (DEG_08, DEG_10, VAN_10, VAN_11, MEO_11 and MEO_12). Similarly, the highest level of 2-methyldihydro-3(2*H*)-thiophenone was found in MEO_11 and MEO_12 with a dominantly large peak area, followed by VAN_10 and VAN_11; DEG_08 and DEG_10 wines did not show a high level for this compound.

3.3. Sensory analysis of wines

The descriptive sensory panel evaluated the wines on 24 odour and taste attributes. From an ANOVA of the sensory data, apart from grapefruit, prune, vanilla and cut grass, all attributes were significant ($p < 0.01$) among the wine samples, indicating that most of the attributes were useful in characterising differences among the wines. The four non-significant attributes were eliminated in further multivariate analyses.

The relationships between samples and sensory attributes were visualised by principal component analysis (PCA). Fig. 1 shows the bi-plot for the first two principal components, which accounted for 84% of the total variance (68% and 16% for PC1 and PC2, respectively). The sensory attributes were mainly explained by the loadings of PC1, contrasting sweet, melon, peach/apricot and Muscat to acidic, chemical, astringent and rooibos/smoke. The loadings of PC2 were positively linked with green apple, citrus and lemon, and negatively associated with alcoholic and rooibos/smoke notes. The six wines on the left side of the plot were primarily characterised by floral and fruity attributes, which are positive characters of young white wine. DYR_12, the wine with the highest sugar content, was positively correlated with sweet as expected as well as banana and melon, and negatively associated with green apple, citrus, lemon, black pepper and acidic characters. SKA_12 had the highest rating in floral/elderflower, Muscat, peach/apricot and strawberry. GAL_11, ORN_11, ORN_12 and DKU_11 were also closely linked with floral and fruity attributes. In contrast, the wines on the right side were characterised by less desirable attributes. MEO_12 was highly associated with respect to alcoholic as well as astringent characteristics; DEG_08 reached the highest value of rooibos/smoke, wood, mushroom, cheese and chemical attributes. Both of them also had the highest scores in bitter taste. In addition, DEG_10 and MEO_12 were positively associated with green apple, citrus, lemon, acidic and black pepper, while VAN_10 and VAN_11 exhibited strong rooibos/smoke, wood and alcoholic notes.

The large sensory difference among commercial wines has been reported in previous studies and reasons can be due to differences in both oenological processing operations and viticultural factors. Parr et al. (2013) studied thirteen commercial Marlborough Sauvignon Blanc wines and found that type of grape processing at harvest provided means for influencing sensory properties of wines, in which machine-harvested-fruit wines were perceived overall as fruitier, less acidic, and as having better concentration, balance

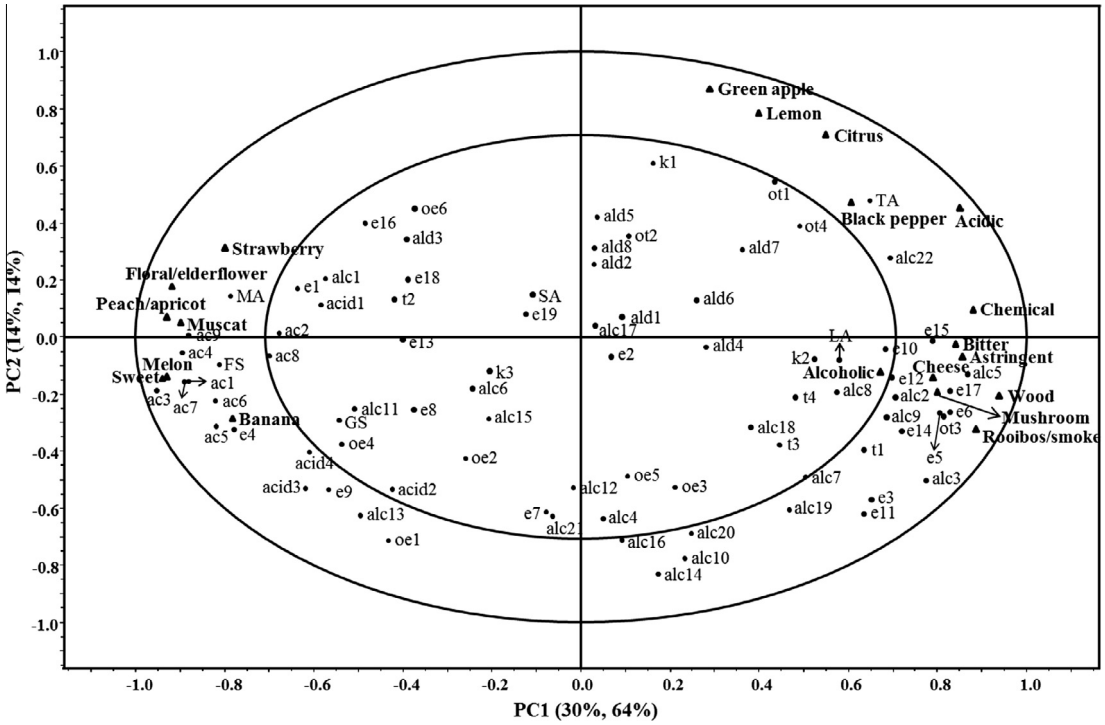

Fig. 2a. PLS loadings plot for the Solaris wines based on the instrumental and sensory analyses. *X*-variable (●): volatile compounds (codes referring to the order listed in Table 4), sugars and organic acids (GS: glucose; FS: fructose; TA: tartaric acid; SA: succinic acid; MA: malic acid; LA: lactic acid); *Y*-variable (▲): sensory attributes.

and persistence in mouth than the wines made from hand-harvested fruit. Aruani et al. (2012) found that Argentinean commercial Malbec wines from Luján were high in plum and floral aroma and flavour, while Valle de Uco (which includes Tupungato and San Carlos) were more associated with red fruit aroma and astringency. In the present study a major part of the clustering appeared to be due to different winemaking skills of the producers. Half of the wines were characterised by less pleasant flavours, which could be because of insufficient chemical and microbiological stability, resulting in an accelerated ageing or spontaneous malolactic fermentation. Moreover, in the wines analysed differences caused by producers were more distinct than vintages. The two samples from the same winery were relatively close to each other although they were from different vintages (Fig. 1 and Fig. 2b). However, it should be mentioned that these observations are limited to the number of wine samples as only one cultivar is examined here.

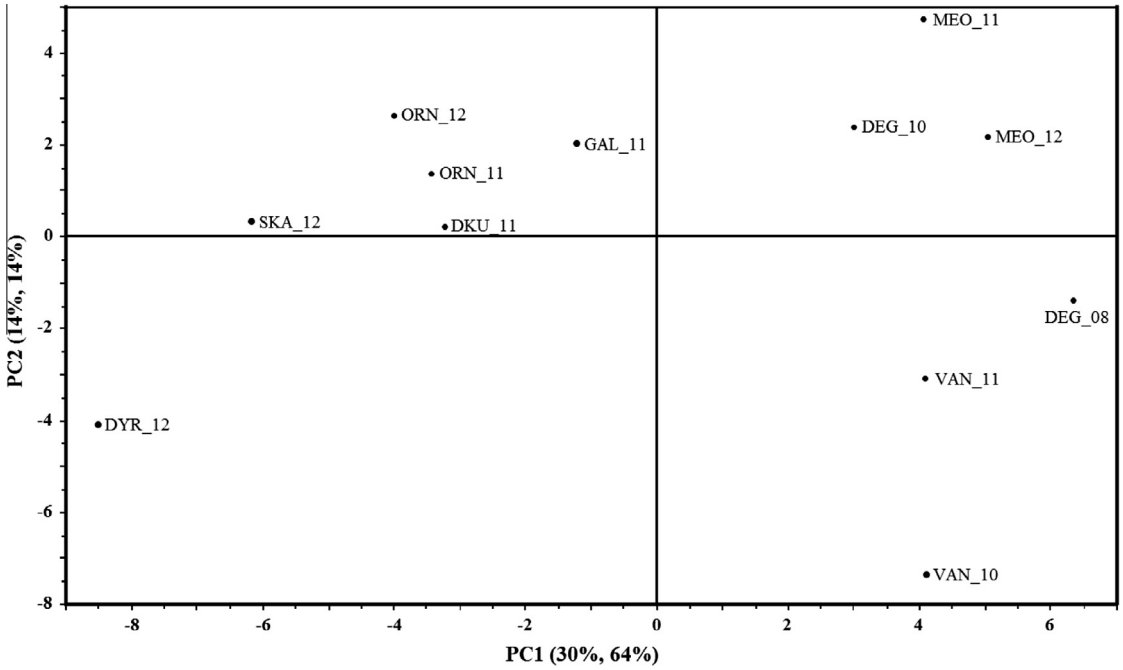

Fig. 2b. PLS scores plot for the Solaris wines based on the instrumental and sensory analyses.

3.4. Correlation between chemical compounds and sensory attributes of wines

Although numerous studies (Jiang et al., 2013; Vilanova et al., 2013) employed Odour Activity Value (*OAV*) to rank volatiles according to their impact to wine aroma, there are some limitations using this concept. *OAV* does not take into account the interactions among volatiles especially the partial suppression occurring when assessed in complex mixtures (Laing, 1994). Some compounds with $OAV < 1$ may also contribute to wine aroma because of the additive effect of similar compounds while compounds with $OAV > 1$ may be not perceived in the wine at all (Francisi & Newton, 2005; González Álvarez et al., 2011). Therefore, *OAV* was not applied in our study. Instead, we employed all of the volatiles identified in the wine for multivariate analysis to explore latent relationships to the sensory data.

The partial least squares (PLS) regression correlation loadings plot for the chemical compounds and sensory attributes is presented in Fig. 2a. The first two PLS components explained 44% of the *X*-variance and 78% of the *Y*-variance. Based on proximity on the left side of the plot, it can be observed that the odour attributes floral/elderflower, peach/apricot, Muscat, melon, banana and strawberry were co-varied positively to the acetates with a high correlation coefficient (r mainly between 0.60 and 0.80) and also several ethyl esters like ethyl butyrate and ethyl acetate. In contrast, mushroom, rooibos/smoke, wood, chemical, cheese and black pepper, located on the right side of the plot, were mainly correlated with some ethyl esters of branched-chain fatty acids, such as ethyl 2-methylbutanoate, ethyl 3-methylbutanoate, ethyl pyruvate, ethyl lactate, ethyl 2-furoate, diethyl malonate and diethyl succinate. The distribution of esters along PC1 and PC2 confirmed observations of Díaz-Maroto, Schneider, and Baumes (2005), who demonstrated that acetates and ethyl esters of straight-chain fatty acids were considered important contributors to young wine aroma and exhibited floral and fruity odours, while ethyl esters of branched-chain fatty acids were negligible contributors to wine aroma. Positive correlations were also found between floral/elderflower and linalool ($r = 0.60$, $p < 0.05$), banana and butyl butanoate ($r = 0.93$, $p < 0.001$), rooibos/smoke and *S*-methyl thioacetate ($r = 0.90$, $p < 0.001$). Negative correlations were found between floral/elderflower and 2-pentanol ($r = -0.79$, $p < 0.01$), citrus and hexanoic acid ($r = -0.62$, $p < 0.05$), chemical and (Z)-3-hexenyl acetate ($r = -0.82$, $p < 0.01$).

With respect to the sugars and organic acids measured, fructose and glucose were positively linked with the taste term sweet ($r = 0.86$, $p < 0.001$ and $r = 0.60$, $p < 0.05$, respectively), and tartaric acid was more related to acidic as was expected ($r = 0.77$, $p < 0.01$). Situated close to the central axis, the variability of succinic acid could not be explained by the model. Malic acid and lactic acid, the precursor and product of malolactic fermentation, were linked with fruity flavours and chemical flavours, respectively.

4. Conclusions

This is the first study to survey the flavour properties of Danish white wines from the grape variety Solaris. The analysis of the volatiles in 12 wine samples showed 3-methyl-1-butanol, 3-methylbutyl acetate, ethyl acetate and ethyl hexanoate to be important in quantity among the 79 compounds identified. Combined with the analysis of the major non-volatile components, clear evidence was shown for inadequate sulphite management, causing accelerated ageing or spontaneous malolactic fermentation. This was also reflected in the sensory differences among the wine samples, half of which were characterised by floral and fruity flavours (peach/apricot, Muscat, melon, banana and strawberry) while the remainder were mainly described by less desirable flavours for white wine, such as chemical, wood and rooibos/smoke. Acetates and ethyl esters of straight-chain fatty acids were correlated with floral and fruity odours, while ethyl esters of branched-chain fatty acids were less linked to young wine flavour character. This work clearly supports the need for further studies on the special cultivars grown in Denmark to further explore the quality potential of this young winemaking country.

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