

## Aromatic Characterization of Pot Distilled Kiwi Spirits

Cristina López-Vázquez,<sup>†</sup> Laura García-Llobodanin,<sup>‡</sup> José Ricardo Pérez-Correa,<sup>§</sup> Francisco López,<sup>\*,‡</sup> Pilar Blanco,<sup>†</sup> and Ignacio Orriols<sup>\*,†</sup>

<sup>†</sup>Estación de Viticultura y Enología de Galicia (EVEGA)-Ingacal, Ponte San Clodio, 32427 Leiro, Spain

<sup>‡</sup>Departament d'Enginyeria Química, Facultat d'Enologia, Universitat Rovira i Virgili, Av. Països Catalans 26, Campus Sescelades, 43007 Tarragona, Spain

<sup>§</sup>Departamento de Ingeniería Química y Bioprocesos, Pontificia Universidad Católica de Chile, Casilla 306, Santiago 22, Chile

**ABSTRACT:** This study contributes fundamental knowledge that will help to develop a distillate of kiwi wine, made from kiwis of the Hayward variety grown in the southwest of Galicia (Spain). Two yeast strains, L1 (*Saccharomyces cerevisiae* ALB-6 from the EVEGA yeast collection) and L2 (*S. cerevisiae* Uvaferm BDX from Lallemand) were assessed to obtain a highly aromatic distillate. The kiwi spirits obtained were compared with other fruit spirits, in terms of higher alcohols, minor alcohols, monoterpenols, and other minor compounds, which are relevant in determining the quality and taste of the kiwi spirits. It was found that the kiwi juice fermented with yeast L1 produced a more aromatic distillate. In addition, kiwi distillates produced with both yeasts had the same ratio of *trans*-3-hexen-1-ol and *cis*-3-hexen-1-ol, which is lower than that found in other fruit distillates.

**KEYWORDS:** kiwi spirit, alcoholic beverages, fermentation, aromatic composition

### INTRODUCTION

Kiwi or kiwi fruit, botanically known as *Actinidia chinensis*, originates from the Yangtze River valley in China,<sup>1</sup> where it has been grown for 2000 years. Spanish kiwi production, over 10,000 tons, is mainly located in Galicia, which has more than 60% of the 900 ha cultivated in Spain. The Spanish kiwi is highly valued in the European market, since it is harvested and immediately consumed, an aspect that differentiates it from kiwis imported from other parts of the world.<sup>2</sup> Recently, the development of cultivation techniques and production management in Spain and other countries have significantly increased kiwi production, which, in turn, has led to an excess of supply. Consequently, although kiwis are still mainly consumed fresh, there is an increasing trend to develop new kiwi-based products, such as nectar, jams, and preserves.<sup>1,3</sup> Moreover, some studies have been done on kiwi wine, particularly in Asia. This opens up the possibility of developing kiwi distillates<sup>3–6</sup> that preserve or enhance the aromatic characteristics of the ripened fresh fruit. The kiwi aroma is a combination of different volatile compounds such as ethyl butanoate, unsaturated aldehydes, and alcohols of six carbon atoms.<sup>7</sup> The aromatic profile varies with fruit maturity, which increases the fraction of esters.<sup>8</sup> In general, the Hayward variety is aromatically characterized by C6 aldehydes and alcohols, with some esters produced upon ripening.<sup>9</sup>

Academic research on kiwi wines and distillates is scarce. We found only two papers dealing with kiwi wine in Europe<sup>3,6</sup> and one paper dealing with a distillate of a kiwi enriched grape wine.<sup>3</sup> Therefore, this research aims to provide fundamental knowledge that will help to develop kiwi distillates characterized by an aromatic profile that meets market standards.

### MATERIALS AND METHODS

**Kiwi Samples.** We used kiwi fruits with 7% soluble solids (SS) of the Hayward variety grown in the southwest of Galicia (Spain) and harvested in November 2009. The kiwi fruit used in this study does

not comply with the minimum commercialization weight of 62 g, as indicated in the Official Journal of the European Communities, 2004.<sup>10</sup> Before processing, the fruit was kept in storage at 2 °C for less than 3 months. At 24 h prior to sampling, fruits were taken out of storage and allowed to warm to ambient temperature (around 20 °C). The fruits were processed at the eating-ripe stage, having an average flesh firmness of 0.5 kgf. The flesh firmness was evaluated using a firmness texture analyzer FTA/GS-14 (Infoagro Systems, S.L., Madrid, Spain). A hand-held refractometer with ATC 0-32 Brix (Auxilab S.L., Berain-Navarra, Spain) was used to measure soluble solids content.

**Kiwi Processing.** Kiwi fruits were sorted by size and washed with plenty of running water in order to remove foreign material from the skin (pesticides, hairs, and particles). Next, the kiwi fruits were crushed with an ENO-2 crusher (Magusa, Vilafranca del Penedès, Spain). Previously the mash was treated with a pectolytic enzyme (Uvazym Arom MP, Sepsa Enartis, Vilafranca del Penedés, Spain) to favor juice extraction. The mash obtained was divided into 6 batches of 55 kg and put into 6 fermentation tanks of 50 L. After pulping, 35 mg/L of SO<sub>2</sub> was added.

**Fermentation.** Two types of kiwi wines (KW) were produced: KWL1, fermented with L1 yeast (*Saccharomyces cerevisiae* ALB-6 from the yeast collection of the Estación de Viticultura e Enología de Galicia, EVEGA, Leiro, Spain), and KWL2, fermented with L2 yeast (*S. cerevisiae* Uvaferm BDX, an active dry yeast from Lallemand, Zug, Switzerland). L1 yeast was previously grown in YEPD medium [1% (w/v) yeast extract, 2% (w/v) peptone and 2% glucose (w/v)] at 28 °C for 24 h, and the cells were recovered by centrifugation, washed with sterile water, and added to kiwi juice at a concentration of 10<sup>6</sup> cells/mL. Following manufacturers' indications, L2 yeast was added to the kiwi juice in the fermentation tanks at a concentration of 25 g of yeast/hL, after rehydration for 20 min in 250 mL of sugared water at 37 °C and finally acclimatizing in 1 L of kiwi juice. Three tanks were inoculated with each strain. All fermentations were carried

Received: July 9, 2011

Revised: January 21, 2012

Accepted: February 10, 2012

Published: February 10, 2012

out at a room temperature of  $12 \pm 1$  °C, and the evolution of the fermentations was monitored by daily measurements of temperature and density in the tanks. When the density reached a plateau, the fermentations were stopped by adding 50 mg/L of SO<sub>2</sub>. The kiwi mashes obtained were stored at 4 °C for less than two weeks, until distillation and chemical characterization of the distillates.

**Distillations with a Charentais Alembic.** In total, 50 kg of fermented kiwi mash was distilled in a 50 L copper Charantais alembic. The base of the boiler was heated by an open flame, and tap water was used to cool the total condenser. The heating power was set to obtain an average distillation rate of 8 mL/min. The first 300 mL of distillate was collected as head, the heart was collected when the ethanol concentration reached 40% v/v, and the tail was obtained and discharged when the ethanol concentration reached 28% v/v. Three distillations were carried out for each wine type, KWL1 and KWL2.

**Chemical Analysis. Classical Parameters.** In the initial mash and in the kiwi wines, the usual parameters were determined in accordance with the Office International de la Vigne et du Vin, OIV,<sup>11</sup> and the Official Journal of the European Communities, 1990:<sup>12</sup> ethanol (steam-distillation of kiwi wine made alkaline by a suspension of calcium hydroxide, and measurement of the alcoholic strength of the distillate by electronic densimetry), reduced sugars (cupric-alkaline method), density (DMA 5000, Anton Paar, GmbH, Graz, Austria), pH (Crison micropH 2000, Barcelona, Spain), total acidity (acid-alkali titration, Crison TitroMatic 1S, Barcelona, Spain), volatile acidity (titration of the volatile acids separated by steam-distillation with sodium hydroxide), citric and malic acid (enzymatic-spectrophotometry, LISA 200 autoanalyzer, TDI, Barcelona, Spain). Anton Paar densimeter (DSA 5000 M, Anton Paar, GmbH, Graz, Austria) was used to determine both the probable alcohol strength of the kiwi juice and the alcoholic strength of the kiwi wines and kiwi distillates as a function of density using conversion charts.<sup>11,12</sup>

**GC Analysis.** The heart fractions obtained were analyzed using gas chromatography coupled with flame ionization detection (GC-FID) with direct injection of the distillate. Analyses were carried out using two different columns. Macroconstituents (methanol, higher alcohols, acetaldehyde, 1,1-diethoxyethane, ethyl acetate, ethyl lactate, 1-hexanol, isobutyraldehyde, ethyl formate, methyl acetate, 2-propenal, 2-butanol, allylic alcohol) were analyzed using a CP Wax-57 CB capillary column (50 m × 0.32 mm i.d. × 0.2 μm film thickness, Varian Medical Systems, Barcelona, Spain) on a GC Agilent 6890 (Agilent Technologies, Waldbronn, Germany) equipped with split/splitless injector with an electronic flow control (EFC) and a FID; conditions were reported in a previous study.<sup>13</sup> The other compounds were separated using a Supelcowax 10 capillary column (30 m, 0.32 mm, 0.25 μm film thickness; Supelco Inc., Bellefonte, PA, USA) in a GC Varian CP3900 (Varian Medical Systems Barcelona, Spain), and the method used was the one described by López-Vázquez et al.<sup>14</sup> Samples were analyzed in triplicate.

**Statistical Analysis.** One-way analysis of variance (ANOVA) was applied to the data obtained from the GC analysis. The aim was to ascertain whether there are significant differences (at 5% level) between the kiwi spirits produced with the two different yeast strains. All the statistical analyses were performed with the SPSS statistical package (version 17.0).

## RESULTS AND DISCUSSION

**Fermentation Process.** The kiwi mash had an initial pH of 3.54, a density of 1.0415 g/mL, and a total sugar concentration of 95 g/L. The total acidity expressed as tartaric acid was 11.4 g/L, and the initial concentration of citric acid was 7.6 g/L. Fermentation runs took on average six days to complete, although fermentation conditions were maintained for four additional days. The characteristics of the final kiwi ferments are summarized in Table 1.

**Table 1. Physical and Chemical Characteristics of Kiwi Wines<sup>a</sup>**

	initial mash	KWL1		KWL2	
		mean	SD	mean	SD
citric acid (g/L)	7.60	6.40	0.53	7.27	0.76
density (g/mL)	1.0415	1.012	<0.001	1.014	0.004
alcoholic strength (% v/v)	5.4 <sup>b</sup>	4.63	0.06	4.80	0.10
reduced sugars (g/L)*	59.6	2.70	0.10	2.40	<0.01
total acidity (mequiv/L)*	152.0	128.4	2.0	138.2	2.0
volatile acidity (g/L acetic acid)*	nd	1.39	0.06	1.26	0.03
pH*	3.54	3.88	0.03	3.76	0.03
malic acid (g/L)*	nd	3.53	0.32	4.60	0.26

<sup>a</sup>\* indicates significant differences ( $P < 0.05$ ) between yeast used. SD: standard deviation ( $p < 0.05$ ). <sup>b</sup>Probable alcohol strength.

KWL1 wines showed slightly lower alcohol content, lower total acidity, and lower citric and malic acid concentrations than KWL2 wines ( $p < 0.05$ ). In turn, volatile acidity and pH were slightly higher in KWL1 wines.

**Aroma Analysis of Distillates.** The average ethanol yields of the distillation were  $61.4\% \pm 4.8\%$  and  $63.8\% \pm 7.7\%$  for KWL1 and KWL2, respectively. In addition, the ethanol contents of the hearts distilled from KWL1 and KWL2 were  $42.5 \pm 0.9\%$  v/v and  $42.3 \pm 1.6\%$  v/v, respectively. These values do not show significant differences.

**Major Volatile Compounds in Kiwi Distillates.** Table 2 shows the average concentration of the 18 macroconstituents

**Table 2. Content of Macroconstituents (g/hL p.a.) Present in Distillates Obtained from Kiwi Wines<sup>a</sup>**

	L1 yeast		L2 yeast	
	mean	SD	mean	SD
ethanol (% v/v)	41.5	0.9	42.3	1.6
methanol	1236.6	76.4	1137.3	47.4
ethyl acetate	46.4	8.9	33.0	0.3
acetaldehyde	153.9	38.0	212.2	41.6
1,1-diethoxyethane	51.9	10.6	65.5	16.3
∑acetaldehyde + 1,1-diethoxyethane	205.8	48.6	277.8	57.8
1-propanol	49.57	2.19	50.32	5.17
2-methyl-1-propanol	70.20	1.28	75.30	13.09
1-butanol*	1.12	0.06	0.62	0.05
2-butanol*	1.80	0.84	0.41	0.09
allylic alcohol	<LOD (0.004)		<LOD (0.004)	
2-methyl-1-butanol*	40.07	0.73	52.83	7.45
3-methyl-1-butanol*	154.53	3.27	194.58	14.84
∑total higher alcohols*	317.30	4.37	374.06	28.91
ethyl lactate*	6.48	2.45	1.86	0.67
1-hexanol*	6.27	0.55	4.13	0.22
isobutyraldehyde	0.27	0.04	0.26	0.03
ethyl formate	1.32	0.31	1.17	0.26
methyl acetate	2.62	0.34	2.27	0.22
2-propenal	4.48	0.80	5.27	1.13

<sup>a</sup>\* indicates significant differences ( $P < 0.05$ ) between yeast used. SD: standard deviation. LOD: detection limit (mg/L).

found in our spirits. They are grouped according to the type of kiwi wine.

**Methanol.** In all cases, methanol concentrations surpassed the legal limit (1000 g/hL p.a.) for general fruit spirits. However, the values are similar to those obtained by Sensidoni et al.<sup>3</sup> for kiwi enriched grape wine spirits produced in a distillation column operating at atmospheric pressure (908.67 g/hL p.a.). Our kiwi distillates are within the methanol limits of fruit distillates such as plum, mirabelle, quetsch, apple, pear, raspberry, blackberry, apricot, and peach (1200 g/hL p.a.) and below the limits of Williams pears, redcurrants, blackcurrants, rowanberries, elderberries, quinces, and juniper berries (1350 g/hL p.a., Council Regulation EC No. 110/2008). The high methanol content is probably due to the pectolytic enzymes, which are commonly used in the production of fruit wines and are responsible for the splitting of pectic substances into galacturonic acid and methanol.<sup>6</sup> Nevertheless, this is not a serious limitation, since the high methanol content of distilled spirits can be significantly reduced with different methods. Hou et al.<sup>15</sup> showed that adding phenolic acids before fermentation inhibits the effect of pectolytic enzymes on methanol synthesis in grape wine. Membrane technology has also been applied to separate methanol from ethanol.<sup>16</sup> The traditional method reduces the pH during the fermentation by adding acids that inhibit the activity of microorganisms and enzymes.<sup>17</sup> Da Porto<sup>18</sup> confirmed the effectiveness of this method by adding phosphoric acid to the wine lees to obtain grappa with lower contents of methanol, 2-butanol, and *n*-propanol.

**Acetaldehyde and 1,1-Diethoxyethane.** Acetaldehyde and 1,1-diethoxyethane are highly volatile compounds (boiling points of 20.2 and 102.7 °C, respectively) and distill mainly in the head fraction. Therefore, they can be used as a reference for the head/heart cut. Acetaldehyde provides the beverage with a fruity character if it is present in low concentrations, but adds a pungent smell when present in higher concentrations.<sup>19</sup> Acetaldehyde is mainly produced by yeast during the fermentation process;<sup>19</sup> when the distillates obtained with both yeasts were compared, it was seen that yeast L1 produced lower amounts of acetaldehyde than yeast L2, although the differences were not statistically different. In this case the acetaldehyde concentration found in our distillates ranged between 153 and 212 g/hL p.a., which is higher than in other fruit distillates,<sup>20–22</sup> although the concentration also depends on the distillation system.

**Ethyl Acetate and Methyl Acetate.** Ethyl acetate and methyl acetate are also highly volatile compounds, although they have higher boiling points than acetaldehyde (bp ethyl acetate = 77 °C, bp methyl acetate = 56.9 °C). The average concentration of ethyl acetate in the samples studied ranged between 33.0 and 46.4 g/hL p.a., which is much lower than the perception threshold (180 g/hL p.a.).<sup>20</sup> Hence, the quality of the distillates is not expected to be affected.

**Ethyl Lactate.** Ethyl lactate is linked to bacterial spoilage, specifically to lactic acid bacteria.<sup>19</sup> High concentrations of this compound negatively contribute to the organoleptic quality of spirits, while low concentrations stabilize the distillate's flavor and soften its harsh character.<sup>19,23</sup> The low concentrations of this compound found in our distillates indicate no sign of spoilage and contribute positively to the flavor of our kiwi spirits.

**Higher Alcohols.** Higher alcohols are mostly formed during fermentation. They make an important contribution to the aroma profile of distillates, imparting a positive aroma and essential character.<sup>20</sup> However, high amounts of higher alcohols can be detrimental to the distillate flavor, giving a pungent smell and taste.<sup>20</sup> In our distillates, it was observed that yeast L1 produced slightly lower amounts of higher alcohols than yeast

L2. In addition, the values we found in kiwi distillates are in the range of 50–60% and 30–40% lower than those reported by Sensidoni et al.<sup>3</sup> for a related spirit distilled at atmospheric pressure and under vacuum, respectively. It is worthy to note though that Sensidoni et al.<sup>3</sup> fermented a kiwi juice enriched with rectified concentrated grape must, to increase the alcohol content of the obtained wine. Therefore, it is not possible to discern if the aromas in their distillate came from the kiwi or the grapes. Moreover, the concentration of higher alcohols in our distillates are similar to those found in other fruit spirits such as pear,<sup>24</sup> orujo,<sup>14</sup> koumaro (obtained from the strawberry tree),<sup>21</sup> and mouro (obtained from the mulberry tree)<sup>20</sup> and slightly lower than the levels found in such other distillates as apple,<sup>22,25</sup> cherry, and plum.<sup>26</sup> An exception is 1-butanol, which presents lower values than orujo<sup>14</sup> and apple.<sup>22</sup> Nevertheless, these low values are in agreement with the levels reported previously for kiwi wines.<sup>6</sup> The concentration of 2-butanol was also low in our distillates: below 2.0 g/hL p.a. in all cases. This is positive because 2-butanol increases with bacterial spoilage in the ensiled pomace; in fact, pomace with less than 2.0 g/hL p.a. is regarded as free from bacterial spoilage.

Finally, 1-hexanol may have an origin which is partly varietal. It plays a positive role in spirits, but when it exceeds 10–15 g/hL p.a., its strong herbal smell becomes unpleasant.<sup>27</sup> Soufleros et al.<sup>6</sup> found levels of 1-hexanol in kiwi wine similar to those in grape wines. In our distillates, concentrations were similar to those obtained in orujos from Galicia.<sup>14</sup>

**Esters. Higher Alcohol Acetates.** Higher alcohol acetates are of fermentative origin and supply the distillates with apple and banana scents.<sup>22</sup> The amounts found in our distillates (Table 3)

**Table 3. Content of Microconstituents (Esters) (g/hL p.a.) Present in Distillates Obtained from Kiwi<sup>a</sup>**

	L1 yeast		L2 yeast	
	mean	SD	mean	SD
isobutyl acetate	<LOD (0.026)		<LOD (0.026)	
butyl acetate	0.03	0.01	0.01	0.01
isoamyl acetate	0.02	0.00	0.03	0.02
hexyl acetate	<LOD (0.018)		<LOD (0.018)	
2-phenylethyl acetate	<LOD (0.063)		<LOD (0.063)	
∑acetates of higher alcohols	0.05	0.01	0.04	0.02
ethyl butyrate	0.09	0.04	0.08	0.04
ethyl hexanoate	0.03	0.01	0.02	0.02
ethyl octanoate	0.79	0.20	0.69	0.15
ethyl decanoate	1.11	0.66	1.13	0.69
ethyl dodecanoate	0.22	0.23	0.25	0.26
∑ethyl esters C6–C12	2.14	1.06	2.04	1.00
ethyl tetradecanoate	0.02	0.03	0.03	0.06
ethyl hexadecanoate	0.11	0.18	0.14	0.23
ethyl octadecanoate	0.03	0.05	<LOD (0.052)	
ethyl 9-octadecenoate	<LOD (0.113)		0.04	0.07
ethyl 9,12-octadecadienoate	0.15	0.05	0.07	0.13
ethyl 9,12,15-octadecatrienoate	0.12	0.17	0.16	0.26
∑ethyl esters C14–C18	0.44	0.42	0.44	0.75
diethyl succinate	0.16	0.04	0.11	0.03

<sup>a</sup>SD: standard deviation. LOD: detection limit (mg/L).

are very low compared to the usual concentrations found in other distillates such as orujo,<sup>28</sup> grappa,<sup>29</sup> and apple<sup>22</sup> and even

lower than those reported in kiwi wine.<sup>6</sup> In addition, we found 10 times less isoamyl acetate than that found in kiwi enriched distillates.<sup>3</sup>

**C6–C12 Ethyl Esters.** C6–C12 ethyl esters come from the fruit and are also produced during fermentation. Later, during the distillation process, the heat causes significant amounts to be released from the yeast cells.<sup>21</sup> They have a floral and fruity character, so their presence in spirits is highly desirable.<sup>30</sup> Soufleros et al.<sup>6</sup> and Winterhalter<sup>31</sup> argue that ethyl butyrate is one of the major volatile components identified in kiwi. In addition, Jordan et al.<sup>32</sup> found ethyl butyrate to be the main ethyl ester in a commercial essence of kiwi. Even though ethyl butyrate cannot be distinguished from hexanal in a chromatogram, it is likely that it is a main ingredient in a fresh puree. The concentration of ethyl butyrate is low in our distillates (Table 3), however, their ethyl ester profile is similar to that of kiwi wines.<sup>5,6</sup> Moreover, the levels of ethyl decanoate in our distillates are similar to those found in kiwi wine by Craig.<sup>33</sup> On the other hand, the concentrations of C6–C12 ethyl esters in our kiwi distillates are about 50% lower than those obtained in kiwi enriched distillates,<sup>3</sup> except for ethyl octanoate, which was slightly higher in our case (Table 3). In addition, our distillates are low in C6–C12 ethyl esters compared to orujo,<sup>14</sup> pear,<sup>24,34</sup> and apple distillates<sup>22</sup> but similar to those of Greek blackberry distillates.<sup>20</sup> Finally, the levels of C6–C12 ethyl esters are similar for both yeasts tested (Table 3).

**C14–C18 Ethyl Esters.** C14–C18 ethyl esters provide a waxy-rancid hint and are mainly derived from the yeast; hence, their concentrations in the distillate depend strongly on the yeast level and type.<sup>22</sup> Some of these compounds, which have been found before in kiwi wines by Peng et al.,<sup>5</sup> are in lower concentrations in our distillates. For example, they present lower concentrations of ethyl tetradecanoate than the kiwi enriched distillate of Sensidoni et al.,<sup>3</sup> the only high molecular ethyl ester identified by these authors. Likewise, our values are lower than those obtained in other distillates such as orujo,<sup>14</sup> and apple spirit.<sup>22,25</sup> Finally, in our distillates, levels of C14–C18 ethyl esters were similar with the two types of yeasts used (Table 3).

**Diethyl Succinate.** Diethyl succinate can be increased by bacterial spoilage in the ensiled pomace if pH is high.<sup>23</sup> It may also be linked to a deficient heart/tail cut during distillation. Hence, it can be argued that in our case bacterial spoilage was minimal and the heart/tail cut was well-defined, since concentrations of diethyl succinate are very low in both kiwi distillates (Table 3), even lower than those obtained in orujo,<sup>14</sup> apple spirit,<sup>25</sup> koumaro,<sup>21</sup> and mouro.<sup>20</sup>

**Minor Alcohols, Monoterpenols, and Other Compounds.** *Minor Alcohols.* Hexenols are usually called “leaf alcohols” because of the flavor they impart to spirits when present at relatively high concentrations.<sup>22</sup> In our kiwi distillates, the concentrations of these alcohols are low (Table 4), although the concentrations of *cis*-3-hexen-1-ol are higher for yeast L1 ( $p < 0.05$ ). According to Versini et al.,<sup>35</sup> the ratio between *trans*-3-hexen-1-ol and *cis*-3-hexen-1-ol could be linked to varietal characteristics. Since this ratio is significantly lower in our spirits than the range of 0.48–1.33, normally found in apple distillates<sup>22</sup> and orujo,<sup>14</sup> it seems that kiwi distillates present a differentiating characteristic.

Linear alcohols from C7 to C10 are rather fruity-floral compounds derived from the decomposition of fatty acids during the fermentative process.<sup>22</sup> Their concentration in the kiwi distillates is quite low for both yeasts (Table 4). However,

**Table 4.** Content of Microconstituents (Minor Alcohols, Monoterpenols, and Other Compounds) (g/hL p.a.) Present in Distillates Obtained from Kiwi<sup>a</sup>

	L1 yeast		L2 yeast	
	mean	SD	mean	SD
<i>trans</i> -3-hexen-1-ol	0.03	0.01	0.02	0.01
<i>cis</i> -3-hexen-1-ol*	0.14	0.02	0.08	0.02
<i>trans</i> -2-hexen-1-ol	0.03	0.01	0.01	0.01
ratio <i>trans/cis</i>	0.24	0.06	0.24	0.16
1-pentanol	0.23	0.02	0.19	0.03
1-heptanol*	0.05	0.00	0.03	0.01
1-octanol*	0.11	0.03	0.04	0.00
1-nonanol	0.04	0.00	0.03	0.01
1-decanol*	0.03	0.01	<LOD (0.127)	
benzyl alcohol	0.05	0.01	0.03	0.01
2-phenylethanol	0.76	0.11	1.09	0.43
∑minor alcohols*	0.46	0.03	0.30	0.40
benzaldehyde*	0.01	0.00	<LOD (0.047)	0.00
furfuraldehyde	1.56	0.28	1.40	0.52
3-ethoxy-1-propanol	0.04	0.03	0.03	0.00
1-octen-3-ol*	0.02	0.00	<LOD (0.068)	
3-hydroxy-2-butanone	1.43	1.12	0.05	0.04
<i>trans</i> -furan linalool oxide	0.26	0.03	0.23	0.06
<i>cis</i> -furan linalool oxide	<LOD (0.043)		<LOD (0.043)	
linalool	0.76	0.19	0.44	0.08
$\alpha$ -terpineol	0.19	0.02	0.16	0.03
citronellol*	0.19	0.01	0.07	0.02
nerol	<LOD (0.029)		<LOD (0.029)	
geraniol	<LOD (0.055)		<LOD (0.055)	
hotrienol <sup>b</sup>	0.21	0.02	0.18	0.04
∑monoterpenols*	1.61	0.20	1.07	0.22

<sup>a</sup>\* indicates significant differences ( $P < 0.05$ ) between yeast used. SD: standard deviation. LOD: detection limit (mg/L). <sup>b</sup>Quantification as linalool (RF = 1.02).

1-pentanol showed significantly higher concentrations than the other linear alcohols. This compound has been found in high concentrations in kiwi fruit<sup>33</sup> and fresh kiwi puree.<sup>32</sup>

Among the minor alcohols present in the kiwi distillates, 2-phenylethanol shows the highest concentration (Table 4). However, the amounts found are low compared to those of a kiwi enriched distillate,<sup>3</sup> grape spirits,<sup>14</sup> koumaro,<sup>21</sup> and mouro,<sup>20</sup> while they are similar to some apple spirits.<sup>22</sup> Low values of 2-phenylethanol were also found by Soufleros et al.<sup>6</sup> in kiwi wine. This component is produced by yeast during the fermentation process and is derived from L-phenylalanine. In fruit puree, it has a pleasant aroma that resembles that of roses.<sup>36</sup> Therefore, it is considered to be a positive compound in spirits when present at low concentrations. 2-Phenylethanol is also a typical tail product; therefore, a high concentration in the heart fraction is indicative of bad heart/tail cut supervision.<sup>19</sup> The moderate levels found in our distillates (around 1 g/hL p.a.) indicate a good separation of the tail fraction. The rest of the minor alcohols and benzyl alcohol presented similar concentration values. Finally, in most minor alcohols, the differences caused by the yeasts used were not statistically significant.

**Monoterpenols.** Terpenoids are widespread in nature. They play an important role in the flavor profile of fruits and fruit distillates, even when present at low concentrations. In kiwi fruit, some monoterpenols such as linalool,  $\alpha$ -terpineol, nerol, and geraniol have been identified.<sup>8</sup> In our distillates, the so-called “skin monoterpenols” (geraniol, nerol, and citronellol)

had lower concentrations than linalool (Table 4). Jordan et al.<sup>32</sup> compared varietal compounds in a kiwi essence and a kiwi puree. Linalool was found in the essence, but not in the puree, possibly indicating that this compound is formed during processing, which occurs at high temperature and low pH. These conditions induce the liberation of bound terpenes into free hotrienol, linalool, and  $\alpha$ -terpineol.<sup>37</sup> In our spirits, linalool is the major monoterpenol, indicating that this compound is released and concentrated during distillation. In a kiwi enriched distillate, lower concentrations of linalool and fewer monoterpenes were identified,<sup>3</sup> although the *trans*-furan linalool oxide was also observed to be dominant over the *cis* isomer. A similar tendency was noticed in apple distillates<sup>22</sup> and orujos.<sup>14</sup> It should be pointed out that the distillates obtained from the kiwi fermented with yeast L1 presented a significantly higher concentration of monoterpenols than distillates obtained with yeast L2. This could be linked to differences in the release, during the fermentation process, of bound forms present in the mash.<sup>22</sup>

**Other Minor Compounds.** Benzaldehyde provides a spicy, almond-like aroma to the spirits,<sup>22</sup> while 3-hydroxy-2-butanone (acetoin) provides a buttery flavor. The concentration of both compounds was very low for both yeasts in all cases (Table 4), less than that found in kiwi enriched distillates<sup>3</sup> and in orujos.<sup>14</sup> Furfuraldehyde is a toxic compound formed during distillation by the dehydration of fermentable sugars caused by heating in acid conditions and/or by the Maillard reaction.<sup>19</sup> Its odor is reminiscent of bitter almond, so its presence in the distillates is undesirable. Furfuraldehyde is very soluble in water and, therefore, distills mainly in the tail fraction,<sup>38</sup> so it can also be considered as an indicator of a defective heart/tail cut. In our distillates, furfuraldehyde shows a very low concentration (Table 4). Therefore, it can be concluded that the separation of the tail fraction was well-defined, which confirms the results obtained for the 2-phenylethanol.

The ANOVA test shows that most of the significant differences ( $p < 0.05$ ) between the distillates obtained with yeasts L1 and L2 were found in the concentration of minor alcohols and monoterpenols, mainly octanol, linalool, and citronellol. These results partly agree with those found by Woo et al.<sup>39</sup> in kiwi wines produced with different yeast strains. They found that the concentration of several alcohols differed among the kiwi wines produced with distinct yeasts, while the other volatile compounds studied showed no significant differences. It is well-known that alcohols give body and character to spirits (at the concentrations found in our distillates) and monoterpenols impart a fruity character. Therefore, kiwi distillates obtained with yeast L1 are expected to have these attributes enhanced. However, a sensory evaluation should be performed in the future to confirm this point.

Chemical analysis has shown that our kiwi wines did not present any indication of bacterial spoilage according to the identified compounds. Furthermore, kiwi distillates present a *trans*-3-hexen-1-ol and *cis*-3-hexen-1-ol ratio different from those found in other fruit distillates.

Therefore, kiwi fruit is a suitable raw material for obtaining distinctive spirits. Nevertheless, additional studies with other yeasts, distillation systems, or enzymatic treatment are necessary to obtain a spirit accepted by consumers (i.e., lower levels of methanol and acetaldehyde and higher levels of higher alcohols, ethyl esters C6–C12, and monoterpenols).

## AUTHOR INFORMATION

### Corresponding Author

\*Tel: +34 988488033. Fax: +34 988-488191. E-mail: evegadir@cesga.es, francisco.lopez@urv.cat.

### Funding

J.R.P.-C. appreciates the support of AGAUR from the Generalitat de Catalunya through Grant 2007PIV-00017 and the Pontificia Universidad Católica de Chile for financial support for a sabbatical visit to the Department d'Enginyeria Química of the Universitat Rovira i Virgili. C.L.-V. acknowledges the Ph.D. fellowship from INIA. The reported research has been funded by FEDER and INIA (Instituto Nacional de Investigación y Tecnología Agraria y Alimentaria) (RTA2009-00123-C02-01).

### Notes

The authors declare no competing financial interest.

## ACKNOWLEDGMENTS

Editorial assistance from Lisa Gingles is highly appreciated. We are also very grateful to the anonymous referees for their constructive feedback that helped us to improve the quality of the paper significantly.

## REFERENCES

- (1) Luh, B. S.; Wang, Z. Kiwifruit. *Adv. Food Res.* **1984**, *29*, 279–307.
- (2) Anonim. El kiwi español, listo para comer. *Mercados* October **2010**, No. 91, [http://www.revistamercados.com/imprimir\\_articulo.asp?Articulo\\_ID=548](http://www.revistamercados.com/imprimir_articulo.asp?Articulo_ID=548) (21-11-10, 18.28).
- (3) Sensidoni, A.; Da Porto, C.; Dalla Rosa, M.; Testolin, R. Utilisation of reject kiwifruit fruit for alcoholic and non-alcoholic beverages. *Acta Hort.* **1997**, *444*, 663–670.
- (4) Jang, S. Y.; Woo, S. M.; Kim, O. M.; Choi, I. W.; Jeong, Y. J. Optimum alcohol fermenting conditions for kiwi (*Actinidia chinensis*) wine. *Food Sci. Biotechnol.* **2007**, *16*, 526–530.
- (5) Peng, B.; Yue, T.; Yuan, Y. Quality evaluation of kiwi wine. *Int. J. Food Eng.* **2006**, *2* (4), Article 2.
- (6) Soufleros, E. H.; Pissa, I.; Petridis, D.; Lygerakis, M.; Mermelas, K.; Boukouvalas, G.; Tsimitakis, E. Instrumental analysis of volatile and other compounds of Greek kiwi wine; sensory evaluation and optimization of its composition. *Food Chem.* **2001**, *75*, 487–500.
- (7) Young, H.; Paterson, V. J. Characterisation of bound flavour components in kiwifruit. *J. Sci. Food Agric.* **1995**, *68*, 257–260.
- (8) Young, H.; Paterson, V. J. The effect of harvest maturity, ripeness and storage on kiwi fruit aroma. *J. Sci. Food Agric.* **1985**, *36*, 352–358.
- (9) Friel, E. N.; Wang, M.; Taylor, A. J.; Macrae, E. A. In vitro and in vivo release of aroma compounds from yellow-fleshed kiwifruit. *J. Agric. Food Chem.* **2007**, *55*, 6664–6673.
- (10) ECC Commission Regulation VO. 1673/2004; Laying down the marketing standard applicable to kiwifruit. *Off. J. Eur. Communities: Legis.* September 25, **2004**, No. L 305, pp 5–10.
- (11) International Organisation of Vine and Wine (OIV). (2009) Compendium of international methods of wine and must analysis, Vol. 1 and 2.
- (12) ECC Commission Regulation VO. 2676/90; Concerning the establishment of common analytical methods in the sector of wine. *Off. J. Eur. Communities: Legis.* October 3, **1990**, No. L 272, pp 1-192.
- (13) López-Vázquez, C.; Bollaín, M. H.; Bertsch, K.; Orriols, I. Fast determination of principal volatile compounds in distilled spirits. *Food Control* **2010**, *21*, 1436–1441.
- (14) López-Vázquez, C.; Bollaín, M. H.; Moser, S.; Orriols, I. Characterization and differentiation of monovarietal grape pomace distillate from native varieties of Galicia. *J. Agric. Food Chem.* **2010**, *58*, 9657–9665.
- (15) Hou, C. Y.; Lin, Y. S.; Wang, Y. T.; Jiang, C. M.; Lin, K. T.; Wu, M. C. Addition of phenolic acids on the reduction of methanol content in wine. *J. Food Sci.* **2008**, *73*, C432–C437.

- (16) Sandström, L.; Lindmark, J.; Hedlund, J. Separation of methanol and ethanol from synthesis gas using MFI membranes. *J. Membr. Sci.* **2010**, *360*, 265–275.
- (17) Versini, G. Elaborazione di grappe di qualità: Criteri da seguire. *Bollettino ISMA* **1993**, *1*, 15–25.
- (18) Da Porto, C. Volatile composition of 'grappa low wines' using different methods and conditions of storage on an industrial scale. *Int. J. Food Sci. Technol.* **2002**, *37*, 395–402.
- (19) Apostolopoulou, A.; Flouros, A.; Demertzis, P.; Akrida-Demertzi, K. Differences in concentration of principal volatile constituents in traditional Greek distillates. *Food Control* **2005**, *16*, 157–164.
- (20) Soufleros, E. H.; Mygdalia, A. S.; Natskoulis, P. Characterization and safety evaluation of the traditional Greek fruit distillate "Mouro" by flavor compounds and mineral analysis. *Food Chem.* **2004**, *86*, 625–636.
- (21) Soufleros, E. H.; Mygdalia, A. S.; Natskoulis, P. Production process and characterization of the traditional Greek fruit distillate "Koumaro" by aromatic and mineral composition. *J. Food Compos. Anal.* **2005**, *18*, 699–716.
- (22) Versini, G.; Franco, M. A.; Moser, S.; Barchetti, P.; Manca, G. Characterisation of apple distillates from native varieties of Sardinia island and comparison with other Italian products. *Food Chem.* **2009**, *113*, 1176–1183.
- (23) Silva, M. L.; Malcata, X. Relationships between storage conditions of grape pomace and volatile composition of spirits obtain therefrom. *Am. J. Enol. Vitic.* **1998**, *49*, 56–64.
- (24) García-Llobodanin, L.; Senn, T.; Ferrando, M.; Güell, C.; López, F. Influence of the fermentation pH on the final quality of *Blanquilla* pear spirits. *Int. J. Food Sci. Technol.* **2010**, *45*, 839–848.
- (25) Rodríguez-Madrera, R.; Blanco-Gomis, D.; Mangas-Alonso, J. J. Influence of distillation system, oak wood type, and aging time on volatile compounds of cider brandy. *J. Agric. Food Chem.* **2003**, *51*, 5709–5714.
- (26) Schehl, B.; Lachenmeier, D.; Senn, T.; Heinisch, J. Effect of the stone content on the quality of plum and cherry spirits produced from mash fermentations with commercial and laboratory yeast strains. *J. Agric. Food Chem.* **2005**, *53*, 8230–8238.
- (27) Cantagrel, R.; Lurton, L.; Vidal, J. P.; Galy, B. From vine to cognac. In *Fermented Beverage Production*, 1st ed.; Lea, A. G. H., Piggott, J. R., Eds.; Blackie Academic and Professional: London, U.K., 1997; pp 208–228.
- (28) Orriols, I.; Cortés, S.; Fornos, D. Caractéristiques des distillats de marc du commerce Orujo de Galicia de l'Espagne. In *Les eaux-de-vie traditionnelles d'origine viticole*; Bertrand, A., Ed.; Lavoisier-Tec&Doc: Paris, 2008; pp 173–177.
- (29) Versini, G.; Orriols, I.; Dalla Serra, A.; Camin, F.; Barchetti, P. Characterisation of Italian (Trentino) and Spanish (Galicia) marc distillates on the basis of volatile and stable isotope parameters. *Proceedings of the V Brazilian meeting on Chemistry of food and beverages: abstract book* **2004**, 11.
- (30) Cortés, S. M.; Gil, M. L.; Fernández, E. The influence of redistillation in the distribution of volatile components of marc spirit (Aguardiente) and its repercussion on the aromatic quality. *Sci. Aliments* **2002**, *22*, 265–275.
- (31) Winterhalter, P. Fruit IV. In *Volatile Compounds in Foods and Beverages*; Maarse, H., Ed.; TNO-CIVO Food Analysis Institute: Zeist, The Netherlands, 1991; pp 389–410.
- (32) Jordán, M. J.; Margaria, C. A.; Shaw, P. E.; Goodner, K. L. Aroma active components in aqueous kiwi fruit essence and kiwi fruit puree by CG-MS and multidimensional GC/CC-O. *J. Agric. Food Chem.* **2002**, *50*, 5386–5390.
- (33) Craigt, J. T. A comparison of the headspace volatiles of kiwifruit wine with those of wine of *Vitis-vinifera* variety Muller-Thurgau. *Am. J. Enol. Vitic.* **1988**, *39*, 321–324.
- (34) García-Llobodanin, L.; Ferrando, M.; Güell, C.; López, F. Pear distillates: influence of the raw material used on final quality. *Eur. Food Res. Technol.* **2008**, *228*, 75–82.
- (35) Versini, G.; Inama, S.; Pilzer, B. Aroma characteristics of Gewurztraminer grape distillate and grappa in relation to the varietal aroma distribution in berry parts and in comparison with other monovarietal distillates. *Proceedings of the 1st Symposium Scientifique International de Cognac*; Cognac, France; 1992; pp 69–76.
- (36) Gerogiannaki, M. Development for a procedure for the determination of 2-phenylethanol in Hellenic wine distillates (*Vitis vinifera* L.) and their changes during distillation. *J. Int. Sci. Vigne Vin* **2009**, *43*, 171–178.
- (37) Marais, J. Terpenes in the aroma of grapes and wines: a review. *S. Afr. J. Enol. Vitic.* **1983**, *4*, 49–58.
- (38) Bollain-Rodriguez, M. H.; Pérez-Fernández, J. E.; Orriols-Fernández, I. L'eau-de-vie de marc ("augardente") d'Albariño: Influence de trois systemes de distillation sur les teneurs en composés volatils. *Les eaux-de-vie traditionnelles d'origine viticole*; Bertrand, A., Ed.; Lavoisier-Tec & Doc: Paris, 1991; pp 43–49.
- (39) Woo, S. M.; Lee, M. H.; Seo, J. H.; Kim, Y. S.; Choi, H. D.; Choi, I. W.; Jeong, Y. J. Quality characteristics of kiwi wine on alcohol fermentation strains. *J. Korean Soc. Food Sci. Nutr.* **2007**, *36*, 800–806.