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Characterization of the Most Odor-Active Volatiles of Orange Wine Made from a Turkish cv. Kozan (*Citrus sinensis* L. Osbeck)

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The aroma-active compounds of cv. Turkish Kozan orange wine were analyzed by sensory and instrumental analyses. Liquid–liquid extraction with dichloromethane was used for extraction of volatile components. According to sensory analysis, the aromatic extract obtained by liquid–liquid extraction was representative of orange wine odor. A total of 63 compounds were identified and quantified in orange wine. The results of the gas chromatography−olfactometry analysis showed that 35 odorous compounds were detected by the panelists. Of these, 28 aroma-active compounds were identified. Alcohols followed by terpenes and esters were the most abundant aroma-active compounds of the orange wine. Among these compounds, ethyl butanoate (fruity sweet), 3-methyl-1-pentanol (roasty), linalool (floral citrusy), γ-butyrolactone (cheesy burnt sugar), 3-(methylthio)-propanol (boiled potato, rubber), geraniol (floral citrusy), and 2-phenylethanol (floral rose) were the most important contributors to the aroma of the orange wine because they were perceived by all eight panelists.

KEYWORDS: Representativeness; olfactometry; aroma-active compounds; orange wine; cv. Kozan orange

INTRODUCTION

Citrus fruits have wide acceptance because of their attractive flavor and nutritional value. The most widely grown citrus fruit in Turkey is orange, with an annual production of 1 250 000 tons in 2005 (*1*). Oranges is grown throughout the world in tropical and subtropical areas, where suitable soils and climates are found. Among oranges, Kozan is a native orange variety of *Citrus sinensis* and is produced on a large scale in the Kozan area of the Adana province in southern Turkey. Kozan oranges harvested in February and March have been said to be of the best commercial quality for processing, having a good balance of sweet taste and a refreshing aroma. The fruit is medium size with a bright orange flesh (*2, 3*).

Wine is defined as an alcoholic beverage, which is produced by fermentation of fresh grapes or must. Grapes and apples are the crops most widely grown for production of juices for winemaking. Citrus fruit, such as orange, mandarin, and grapefruit, are also used for wine (*2–7*), wine cooler (*8*), and spirit production (*9*). Flavor characteristics of foodstuff were major factors in determining consumer acceptance and preference. Orange flavor is probably the most widely recognized and accepted flavor in the worldwide food and beverage industry; it is widely used to flavor or aromatize foods and beverages because of its distinctive flavor and aroma. Its fresh and uniquely flavor is due to complex combinations of several odor components that have interdependent quantitative relationships. Important contributors to orange juice flavor include esters, aldehydes, ketones, terpenes, and alcohols (*10, 11*).

The volatile content of orange juice can be changed by the ethyl alcohol fermentation, because of the production of yeast volatiles and the metabolism of original fruit volatiles (*12*). The chemical compounds responsible for wine aroma are mainly alcohols, esters, acids, aldehydes and ketones, of which esters are particularly important (*13*). Over 1300 volatile compounds have been identified in alcoholic beverages (*14*). However, it is well-accepted that only a small fraction of the large number of volatile compounds occurring in food actually contributes to the overall aroma. Therefore, a major task in flavor studies is to separate the strongly odor-active compounds from the less odorous or odorless component present in food (*15*). Gas chromatography−olfactometry (GC−O) has become the most widely used technique for identification of key flavor compounds in aroma extracts. This technique uses the human nose as a detector to distinguish the single-volatile compounds (*16*). Up to now, numerous investigations have been performed aimed at identifying the aroma-active compounds in the orange juice and wine samples. Marin et al. (*17*) investigated the effects of plastic polymers on orange juice aroma using the GC−O technique. Hinterholzer and Schieberle (*18*) identified the most

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odor-active volatiles in fresh, hand-extracted juice of Valencia late oranges by odor dilution techniques. Buettner and Schieberle (*19*) evaluated the aroma differences between hand-squeezed juices from Valencia late and Navel oranges by quantitation of key odorants and flavor reconstitution experiments. Guth (*20*) elucidated the most odor-active compounds in Gewürztraminer and Scheurebe white wines by the two GC−O techniques. Gürbüz et al. (*21*) compared the aroma composition in commercial Merlot and Cabernet Sauvignon wines from two disparate wine-growing regions from two different years using GC−O and gas chromatography−mass spectrometry (GC−MS). One of the most important problems in olfactometry studies is that of the extract representativeness. The success of characterization of aroma-active compounds from foodstuffs by GC−O largely depends upon the extraction technique employed. The extraction must be selected with the aim of producing extracts with odor as close as possible to that of the original product. Once the representativeness of the extract has been assessed by sensory evaluation, GC−O may be applied (*22, 23*). Several techniques have been applied to wine aroma extraction. In our study, the aroma extraction method selected was liquid–liquid extraction. This technique has already shown its reliability for the extraction of volatile compounds of different must (*23*) and wine samples (*25*).

The volatile composition of the Kozan orange juice was studied, and 34 components, including 7 esters, 2 aldehydes, 5 alcohols, 5 terpenes, 12 terpenols, and 3 ketones, were identified and quantified in this cultivar (*26*). In addition, there was only one scientific study on the flavor composition of orange wine obtained from this cultivar (*2*). The aim of the following investigation was to elucidate the most odor-active compounds in orange wine by the two GC−O techniques, detection frequency and time intensity. Before the GC−O analysis, the aromatic extract obtained from liquid–liquid extraction was analyzed by sensory evaluation to assess its representativeness.

MATERIALS AND METHODS

Reagents. The water used in the study was purified by a Millipore-Q system (Millipore Corp., Saint-Quentin, France). The following reference aroma compounds were obtained from the following sources: ethyl butanoate, ethyl-3-methylbutanoate, isoamyl acetate, 1-butanol, isoamyl alcohol, D,L-limonene, ethyl hexanoate, 1-pentanol, 3-methyl-3-buten-1-ol, (*Z*)-4-heptenal, 2-methylbutyl acetate, hexyl acetate, *p*-cymene, acetoin, 3-methyl-2-buten-1-ol, linalyl acetate, 3-methyl-1-pentanol, ethyl lactate, 1-hexanol, (*E*)-3-hexen-1-ol, (*Z*)-3-hexen-1-ol, ethyl octanoate, furfural, benzaldehyde, ethyl-3-hydroxybutanoate, 2,3-butanediol, linalool, terpinen-4-ol, ethyl decanoate, γ-butyrolactone, diethyl succinate, α-terpineol, 3-(methylthio)propanol, citronellol, hexanoic acid, geraniol, benzyl alcohol, 2-phenylethanol, diethyl malate, octanoic acid, eugenol, 4-vinyl-2-methoxy-phenol, 4-vinyl guaiacol, monoethyl succinate, 4-vinyl phenol, methoxyeugenol, and vanillin (Aldrich, Steinheim, Germany) and 1-propanol and 1-octanol (Merck, Darmstad, Germany). Dichloromethane, sodium hydroxide, citric acid, ethyl alcohol, and sodium sulfate were obtained from Merck (Darmstad, Germany). Dichloromethane was freshly distilled prior to use.

Oranges. The mature oranges of Kozan variety (1200 kg) were harvested in the middle of March 2005 from three different orchards in the province of Kozan and transported to the Pilot Winery of the Department of Food Engineering, Faculty of Agriculture, University of Cukurova. The orange juice was obtained using the "Indelicato Super Automatic, Type A2 104" extractor.

Wine Making. Orange wine was produced as described previously (*2, 3*). Orange juice obtained from the extractor was passed through the finisher to remove the seeds and pulp, and 50 mg/L SO_2 was added. The orange juices were then transferred into two stainless-steel tanks (250 L) for the fermentation using spontaneous yeasts. Fermentation was performed in duplicate at controlled temperatures (19 ± 2 °C). During the alcoholic fermentation, 145 g/L sugar was also added to obtain a higher ethanol level. After fermentation, the wine was racked by adding 50 mg/L SO_2.

Standard Chemical Analysis. Density, total acidity, pH, extract, ash, total sugar, and ascorbic acid analyses were performed in orange juice and wine. Additionally, the orange wine was analyzed for ethyl alcohol, total phenolics (absorbance at 280 nm), volatile acidity, free and bound SO_2 (*27, 28*).

Extraction of the Volatile Compounds. *Liquid–Liquid Extraction.* Extraction of aroma compounds was performed in dichloromethane, which was the most suitable solvent for isolating volatiles from the grapes and wines (*24, 25, 29*). Before extraction, 40 mg of 2-octanol as an internal standard and 40 mL of dichloromethane were pipetted into a 500 mL flask containing 100 mL of wine. The content was stirred at 4 °C for 30 min under nitrogen gas. The mixture was then centrifuged at 4 °C (9000*g*, 15 min). After the dehydration by anhydrous sodium sulfate, the pooled organic extract was reduced to 5 mL in a Kuderna Danish concentrator fitted with a Snyder column (Supelco, St. Quentin, France) and then to 0.5 mL under a gentle stream of nitrogen. The whole process was repeated 3 times. The extracts were subsequently stored at −20 °C in a glass vial equipped with Teflon-lined cap before the analysis. Each sample was extracted in triplicate and the concentration of volatiles, as 2-octanol equivalents, was obtained as a mean of three repetitions.

Sensory Analysis/Representativeness of the Extract. *Panel.* The panel was composed of 10 assessors (7 females and 3 males between 22 and 50 years old) from LBAI ENITIAA, Laboratoire de Biochimie Industrielle et Alimentaire−Ecole Nationale d'Ingénieurs des Techniques des Industries Agricoles et Alimentaire. The assessors were previously trained in odor recognition and sensory evaluation techniques and had experience in GC−O. As for the training sessions on orange wine descriptors, the first session took place in an ordinary room to generate descriptors for the wine. The panel generated the descriptors of orange wine. A list of nine consensual odor descriptors (fruity, citrus-like, orange, green/grassy, smoky, floral, vanilla, alcohol, and spicy) was established. The other sessions took place in a sensory room (*30*) in isolated booths under natural light color at room temperature.

Sample Preparation and Presentation. Different methods can be used to evaluate the representativeness of the odor of aromatic extracts depending upon the type of investigation. Two different tests were used for checking representativeness of the extract obtained from liquid–liquid extraction with dichloromethane in this study. A total of 2 mL of orange wine was placed in a 15 mL brown coded flask as a reference for two tests. At the start, an aliquot of the orange wine extract was adsorbed onto a cardboard smelling strip (reference 7140 BPSI, Granger-Veyron, Lyas, France). After 1 min (the time necessary for solvent evaporation), the extremities of the strips were cut off, then placed in dark coded flasks (15 mL), and presented to the panel after 15 min. Dichloromethane was a very volatile solvent and, hence, was evaporated in the air during 1 min. No panelists detected the odor of the solvent. The model orange wine was prepared containing the matrix compounds (7.5 g/L citric acid, water/ethanol solution/12.3% (v/v) ethanol, and 43.4 g/L sugar). The pH of the model wine was adjusted to 3.8 using 0.1 N sodium hydroxide. A total of 2 mL of model wine was placed in a 15 mL brown coded flask. An aliquot of the orange wine extract was added in the flask and stirred for 5 min. The solvent from aromatic extract was eliminated under a nitrogen flux. The model wine was presented to the panel after 15 min. All of the samples were assessed at room temperature (20 °C).

Similarity Test. A similarity test was performed to evaluate the closeness between the odor of the extract and the orange wine (reference sample). The panelists were instructed to sniff and memorize the aroma of the reference sample and, for the extract, to sniff the smelling strip and model wine odor and determine the similarity of their odors. For each test, a 100 mm unstructured scale was used, anchored with "very different from the reference" on the left and "identical to the reference" on the right. The position of the sample on the unstructured scale was read as the distance in millimeters from the left anchor.

Odor Intensity Evaluation. The panelists were asked to assess the odor intensity of the extract. A 100 mm unstructured scale was used,

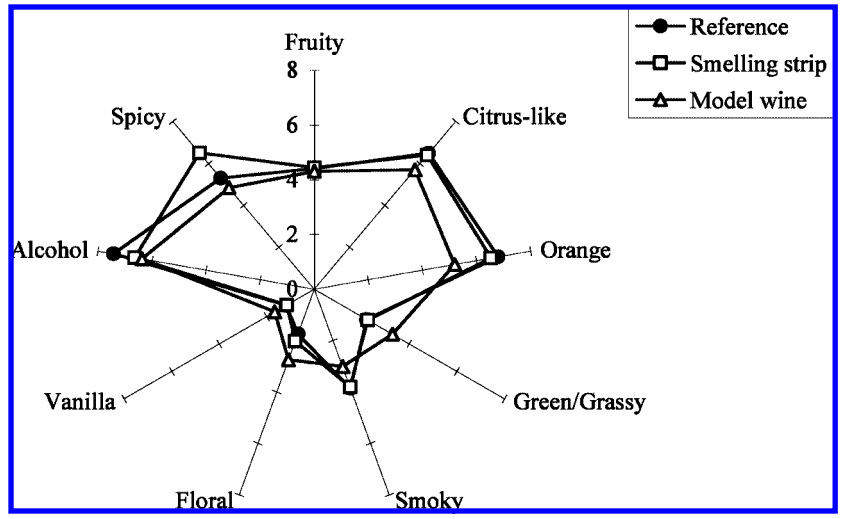

Figure 1. Odor sensory profiles of orange wine and extract (units: centered average marks given by the judges on the 100 mm unstructured scales).

anchored with "no odor" on the left and "very strong odor" on the right. The position of the sample on the unstructured scale was read as the distance in millimeters from the left anchor.

Descriptive Analysis of Orange Wine and Its Extract. A list of nine descriptors (fruity, citrus-like, orange, green/grassy, smoky, floral, vanilla, alcohol, and spicy) that describe the aroma of orange wine were previously determined by the panelists and subsequently used to describe the extract. The extract in the model wine, smelling strip, and reference sample were presented to the panel, and assessors were asked to describe the odorous characteristics of each sample by evaluating the intensity of each given descriptor on an unstructured scale of 100 mm, anchored at the left end with "no odor" and the right end with "very strong odor". The intensity notes were given by averaging the distance in millimeters from the left anchor to the marks of the judges. Sensory analysis results were analyzed with an analysis of variance with Statgraphics Plus software (Manugistic, Inc., Rockville, MD).

GC−flame ionization detector (FID), GC−MS, and GC−O Analysis of Volatile Compound. The GC system consisted of Agilent 6890 chromatograph equipped with FID, Agilent 5973-Network-mass selective detector (MSD) (Wilmington, DE), and Gerstel ODP-2 (Baltimore, MD) sniffing port supplied with humidified air at 40 °C using a deactivated fused silica capillary (30 cm × 0.3 mm). This system allowed us to simultaneously obtain a FID signal for the quantification, an MS signal for the identification, and the odor characteristics of each compound detected by sniffing port. The GC effluent was split 1:1:1 among the FID, MSD, and sniffing mode. Volatile compounds were separated on DB-Wax (30 m length × 0.25 mm i.d. × 0.5 μm thickness, J&W Scientific, Folsom, CA) column. A total of 3 μL of extract was injected in pulsed splitless (40 psi; 0.5 min) mode. This mode was chosen to minimize artifact formation by thermal degradation of analytes in the injection port. Injector and FID detectors were set at 270 and 280 °C, respectively. The flow rate of the carrier gas (helium) was 1.5 mL/min. The oven temperature of the DB-Wax column was increased from 50 to 250 °C at a rate of 4 °C/min with a final hold at 250 °C for 10 min.

The same oven temperature programs were used for the mass-selective detector. The MS (electronic impact ionization) conditions were as follows: ionization energy of 70 eV, mass range m/z of 30–300 amu, scan rate of 2.0 scan/s, interface temperature of 250 °C, and source temperature of 180 °C.

The volatile compounds were identified by comparing their retention index and their mass spectra on the DB-Wax column to those of a commercial spectra database (Wiley 6, NBS 75k) and the internal library of the instrument created from the previous laboratory studies. Some of the identifications were confirmed by the injection of the chemical standards into the GC−MS system. Retention indices of the compounds were calculated using a *n*-alkane series (*31*).

Frequency of the Detection Method. A panel of eight judges trained in odor detection and recognition and having experience in GC−O was selected from the 10 previous panelists. Sniffing of the chromatogram was divided into two parts of 20 min. Each panelist participated in the sniffing of both parts but during two distinct sessions to remain alert. The panelists assigned the odor properties of each odorant detected. Detection of odor at the sniffing port by fewer than three of the eight assessors was considered to be noise (*24*). The eight individual aromagrams were summed, yielding the final aromagram [detection frequency versus retention index (RI)].

Time Intensity Method. The time intensity method (*24*) was used to measure the odor intensity of the compounds detected. The same panelists used before were trained to evaluate aroma intensity using a nine-point intensity scale (1 = very weak intensity, 3 = weak intensity, 5 = moderate intensity, 7 = strong intensity, and 9 = very strong intensity). Sniffing conditions were the same as for the frequency of detection, except that the panelists were also asked to assess intensity (according to the nine-point scale) for each odorous area. Times and intensities of areas detected by at least three panelists were averaged, and a consensus aromagram (averages versus RI) was created.

RESULTS AND DISCUSSION

Sensory Analysis. *Odor Comparison of the Orange Wine and Its Extract.* The aromatic extract on the smelling strip and model wine were compared to the orange wine sample (reference) by 10 panelists. **Figure 1** shows the mean intensity ratings for the reference sample and its extract plotted on a spider graph using nine descriptors. As shown in the figure, odor descriptors of orange wine and its extract were described as fruity, citrus-like, orange, green/grassy, smoky, floral, vanilla, alcohol, and spicy. Odor descriptors used by our panelists have already been generated by other panels to characterize the odor of orange juices. To investigate the sensorial quality of orange juice extract obtained from the SPME technique, Rega et al. (*32*) used five similar descriptors, including orange, citrus-like, fruity, green, and spicy. Similarly, three of them, citrus-like, fruit, and grassy, were also used by Buettner and Schieberle (*19*) to evaluate the aroma differences between hand-squeezed juices from Valencia late and Navel oranges.

Among the descriptors, vanilla was the descriptor with the lowest score, while orange, alcohol, citrus-like, and spicy reached the highest scores. The scores obtained by sniffing on both smelling strip and model wine after evaporation of the solvent were no different from the scores obtained on the reference sample. This can be visualized on the plot of average sensory scores of the reference sample compared to its extract in **Figure 1**. No statistical differences were found between the

extract and reference of the nine descriptors. These results show a close relationship between the odor properties of orange wine and its extract.

Intensity and Similarity Evaluation. The intensity scores of the aromatic extract on the smelling strip and model wine were found to be 68.9 and 66.1 mm on a 100 mm unstructured scale, respectively. The intensity scores of the extract were quite high and similar. The extract adsorbed on the smelling strip presented a slightly higher intensity score than the model wine. The differences of the intensity score for two tests were found statistically insignificant. With regard to similarity evaluation, the similarity score of the aromatic extract in model wine (71.4 mm on a 100 mm unstructured scale) was found to be better than on the smelling strip (51.9 mm on a 100 mm unstructured scale). Similarity scores of two tests were found to be significantly different. Both similarity scores were acceptable and high; however, model wine was scored relatively more closely to the reference sample. This result suggests that the model wine contains more character impact aroma compounds of orange wine than the smelling strip. Panelists have also noted that the aroma of the model wine as being more orange wine-like and consider it as having the full complexity of orange wine. Smelling strips modified the aroma of extracts, which corroborate the results obtained by Sarrazin et al. (*33*). Similar results were also observed on salmon extracts by Varlet et al. (*34*). They compared a redeposition on a real matrix and smelling strips for salmon extract and found that smelling strips cause the loss of about 9% representativeness. As previously stated, it is of great importance to assess the representativeness of the aromatic extracts in a matrix with characteristics similar to those of the original product (*35, 36*). When we compared our results to previous studies, similarity scores of the different grape must extracts were found between 37 and 69 mm by Serot et al. (*24*), for apple extracts between 49.1 and 57.0 mm by Mehinagic et al. (*37*), for orange juice extracts between 51 and 63 mm by Rega et al. (*32*), and for rainbow trout extract 51.1 mm by Selli et al. (*38*).

The results of our sensory analysis suggested that the extract from cv. Kozan orange wine was considered to be representative for the olfactometric analysis.

General Composition of Orange Juice and Wine. The chemical and physicochemical properties of orange juice and wine are shown in **Table 1**. The juice yield was 39 L/100 kg (39%). The juice and wine compositions were in agreement with the previous studies carried out on cv. Kozan orange juice and wine (*2, 3, 26*).

Volatile Compositions of Orange Wine. The volatile compounds identified in orange wine and linear retention index values on the DB-Wax column for these compounds are presented in **Table 2**. Mean values (μg/L) of the GC analyses of triplicate extractions and standard deviations are reported. A total of 63 compounds were identified and quantified in orange wine, most of which have already been identified by previous studies (*2, 9*). The wine had 132.41 mg/L volatile compounds, which included esters (18), higher alcohols (16), terpenes (14), volatile phenols (6), aldehydes (3), acids (3), ketones (2), and lactone (1).

Higher alcohols and esters were the most dominant compounds in orange wine, because they accounted for the largest proportion (93.4%) of the total volatiles. These compounds, produced during alcoholic fermentation, play an important role in the flavor of wines, which varies depending upon types of compounds present and their concentration (*13, 14*). Among the higher alcohols, isoamyl alcohol showed the highest concentration (79 043 μg/L) in orange wine. Another alcohol present at a very high concentration was 2-phenylethanol (27 261 μg/L). At concentrations below 300 mg/L, they contribute to the desirable complexity of wine; when their concentrations exceed 400 mg/L, higher alcohols are regarded as a negative quality factor (*13*). The total concentration of higher alcohols in Kozan wine was below 300 mg/L (**Table 2**). After higher alcohols, esters were clearly the dominant constituents in the orange wine sample. These compounds are an important group of volatile compounds produced by yeast cells during alcoholic fermentation. The total amount of esters was 6957.8 μg/L. Their concentration is dependent upon several factors, mainly juice composition, fermentation temperature, yeast strains, and aeration degree (*13, 14*).The major esters identified important quantities of ethyl lactate (1770 μg/L), ethyl-4-hydroxy-butanoate (1508 μg/L), and monoethyl succinate (1002 μg/L), respectively.

Table 1. Chemical and Physicochemical Properties of Orange Juice and Wine

juice composition	
density (20 °C/20 °C)	1.054 ± 0.00
total acidity[a] (g/L)	9.2 ± 0.02
pH	3.4 ± 0.00
ascorbic acid (mg/100 mL)	59 ± 0.63
total sugar (g/L)	105.4 ± 0.82
ash (g/L)	3.4 ± 0.01
extract (g/L)	132 ± 0.28
wine composition	
density (20 °C/20 °C)	1.0028 ± 0.00
total acidity[a] (g/L)	7.8 ± 0.01
pH	3.5 ± 0.00
alcohol (%, v/v)	12.3 ± 0.01
ascorbic acid (mg/100 mL)	30 ± 0.21
extract (g/L)	50 ± 0.20
total phenolics (Abs_{280}, nm)	26.9 ± 0.12
volatile acidity[b] (g/L)	0.18 ± 0.02
total sugar (g/L)	43.4 ± 0.07
ash (g/L)	3.1 ± 0.02
free SO_2 (mg/L)	8.1 ± 0.11
bound SO_2 (mg/L)	73 ± 0.08

[a] Citric acid. [b] Acetic acid.

GC−O Results. The results of olfactometric analysis are summarized in **Table 3**. A total of 35 aroma-active compounds were detected in orange wine by GC−O, including alcohols (10), terpenes (7), esters (6), volatile phenols (2), lactone (1), aldehyde (1), and acid (1). In addition, seven odorants, unknown, were detected by GC−O but could not be identified by GC−MS. Only the compounds that were detected at least 3 times by one of the panelists were considered as contributors to the aroma of orange wine. The aroma-active compounds of orange wine were predominantly alcohols, followed by terpenes and esters. Among these compounds, ethyl butanoate, 3-methyl-1-pentanol, linalool, γ-butyrolactone, 3-(methylthio)-propanol, geraniol, and 2-phenylethanol were the most important contributors to the aroma of the orange wine because they were perceived by all eight panelists.

Alcohols. A total of 10 aroma-active alcohols were perceived in the wine aromatic extract by panelists. Three of them including 3-methyl-1-pentanol (roasty), 3-(methylthio)-propanol (boiled potato rubber), and 2-phenylethanol (floral rose) can be considered as the most potent odorants of orange wine because they were detected by eight panelists. The other two alcohols identified in the sample, (*E*)-3-hexen-1-ol and (*Z*)-3-hexen-1-ol, were found to carry moderate green-floral and green odors, respectively, which was in agreement with previous observations in French and Romanian hybrid musts (*24*), orange juice (*32*),

Table 2. Volatile Flavor Compounds of Orange Wine

number	LRI[a]	compounds	concentration[b] (mean ± SD)	identification[c]
1	1018	isobutyl acetate	29.6 ± 2.24	LRI, MS, tent.
2	1033	1-propanol	1158 ± 12.62	LRI, MS, std
3	1041	ethyl butanoate	307 ± 4.04	LRI, MS, std
4	1070	ethyl-3-methylbutanoate	5.1 ± 0.32	LRI, MS, std
5	1086	isobutyl alcohol	6197 ± 19.10	LRI, MS, tent.
6	1133	isoamyl acetate	496 ± 3.84	LRI, MS, std
7	1141	1-butanol	19.9 ± 1.63	LRI, MS, std
8	1217	isoamyl alcohol	79 043 ± 290	LRI, MS, std
9	1234	D,L-limonene	430 ± 4.75	LRI, MS, std
10	1241	ethyl hexanoate	173 ± 3.09	LRI, MS, std
11	1247	1-pentanol	35.1 ± 0.36	LRI, MS, std
12	1249	3-methyl-3-buten-1-ol	28.4 ± 0.88	LRI, MS, std
13	1251	(*Z*)-4-heptenal	6.8 ± 0.40	LRI, MS, std
14	1254	(*E*)-β-ocimene	11.8 ± 0.95	LRI, MS, tent.
15	1258	γ-terpinene	10.7 ± 1.20	LRI, MS, tent.
16	1262	2-methylbutyl acetate	16.5 ± 1.24	LRI, MS, std
17	1272	hexyl acetate	37.4 ± 2.04	LRI, MS, std
18	1280	*p*-cymene	5.2 ± 0.16	LRI, MS, std
19	1287	acetoin (3-OH-2-butanone)	76.5 ± 3.10	LRI, MS, std
20	1293	α-terpinolene	12.8 ± 1.56	LRI, MS, tent.
21	1311	3-methyl-2-buten-1-ol	25.0 ± 1.34	LRI, MS, std
22	1319	linalyl acetate	49.3 ± 2.07	LRI, MS, std
23	1324	3-methyl-1-pentanol	22.5 ± 2.22	LRI, MS, std
24	1335	ethyl lactate	1770 ± 13.40	LRI, MS, std
25	1343	1-hexanol	254 ± 3.52	LRI, MS, std
26	1354	(*E*)-3-hexen-1-ol	217 ± 2.08	LRI, MS, std
27	1374	(*Z*)-3-hexen-1-ol	214 ± 6.72	LRI, MS, std
28	1428	ethyl octanoate	279 ± 2.31	LRI, MS, std
29	1452	furfural	129 ± 3.48	LRI, MS, std
30	1469	(*Z*)-furan linalool oxide	33.7 ± 0.20	LRI, MS, tent.
31	1502	benzaldehyde	9.5 ± 0.38	LRI, MS, std
32	1509	ethyl-3-hydroxybutanoate	305 ± 2.28	LRI, MS, std
33	1524	2,3-butanediol	1800 ± 11.07	LRI, MS, std
34	1548	linalool	1640 ± 9.02	LRI, MS, std
35	1568	1-octanol	169 ± 1.25	LRI, MS, std
36	1606	terpinen-4-ol	919 ± 6.08	LRI, MS, std
37	1628	ethyl decanoate	292 ± 2.33	LRI, MS, std
38	1647	γ-butyrolactone	491 ± 1.97	LRI, MS, std
39	1665	diethyl succinate	291 ± 2.74	LRI, MS, std
40	1690	α-terpineol	836 ± 4.38	LRI, MS, std
41	1714	3-(methylthio) propanol	166 ± 3.30	LRI, MS, std
42	1720	ethyl acetylacetate	19.9 ± 1.05	LRI, MS, tent.
43	1729	valencene	13.5 ± 1.11	LRI, MS, tent.
44	1748	citronellol	137 ± 2.31	LRI, MS, std
45	1796	ethyl-4-hydroxybutanoate	1508 ± 6.68	LRI, MS, tent.
46	1818	hexanoic acid	558 ± 3.12	LRI, MS, std
47	1831	geraniol	271 ± 2.25	LRI, MS, std
48	1844	(*Z*)-carveol	40.0 ± 0.36	LRI, MS, tent.
49	1864	benzyl alcohol	55.3 ± 1.96	LRI, MS, std
50	1908	2-phenylethanol	27 261 ± 65.04	LRI, MS, std
51	1953	3,7-dimethyl-oct-1-en-3,7-diol	294 ± 1.95	LRI, MS, tent.
52	2029	diethyl malate	183 ± 2.82	LRI, MS, std
53	2061	octanoic acid	1098 ± 8.96	LRI, MS, std
54	2120	eugenol	33.2 ± 0.24	LRI, MS, std
55	2191	4-vinyl-2-methoxy-phenol	552 ± 3.53	LRI,MS, std
56	2218	4-vinyl guaiacol	336 ± 4.82	LRI, MS, std
57	2285	ethyl-2-hydroxy-3-phenyl propanoate	194 ± 2.18	LRI, MS, tent.
58	2360	monoethyl succinate	1002 ± 11.13	LRI, MS, std
59	2390	4-vinyl phenol	112 ± 4.11	LRI,MS, std
60	2507	dodecanoic acid	38.4 ± 1.02	LRI, MS, tent.
61	2551	methoxyeugenol	190 ± 1.44	LRI, MS, std
62	2586	nootkatone	459 ± 2.78	LRI, MS, tent.
63	2597	vanillin	40.0 ± 0.96	LRI, MS, std
		total flavor compounds	132 407.1	

[a] LRI, linear retention index calculated on a DB-Wax capillary column. [b] Results are the means of three repetitions in μg/L. [c] Methods of identification: LRI (linear retention index), MS tent. (tentatively identified by MS), and std (chemical standard). When only MS or LRI is available for the identification of a compounds, it must be considered as an attempt of identification.

and freshly distilled cognac (*39*). These two C-6 alcohols are mainly formed during juice processing prior to fermentation. Oliviera et al. (*40*) also reported that (*E*)-3-hexen-1-ol and (*Z*)-3-hexen-1-ol have been referred as the most important because their ratio can act as an indicator of the variety of wine origin. Among the alcohols, 2-phenylethanol had the highest aroma intensity detected by GC−O; therefore, it might be of great importance for the orange wine aroma. This aromatic alcohol

Table 3. Aroma-Active Compounds of Orange Wine

number	LRI[a]	compound	odor description[b]	intensity[c]	number of judges[d]
1	1033	1-propanol	plastic	5	7
2	1041	ethyl butanoate	fruity, sweet	5	8
3	1118	unknown	plastic	4	8
4	1166	unknown	green, floral	4	6
5	1217	isoamyl alcohol	chemical, harsh	5	7
6	1254	(*E*)-β-ocimene	fruity, minthy	5	5
7	1272	hexyl acetate	floral	4	5
8	1324	3-methyl-1-pentanol	roasty	6	8
9	1354	(*E*)-3-hexen-1-ol	green, floral	4	6
10	1374	(*Z*)-3-hexen-1-ol	green	5	6
11	1398	unknown	floral	4	5
12	1438	unknown	bread, roasty	4	7
13	1452	furfural	pungent	3	6
14	1469	(*Z*)-furan linalool oxide	green, sweet	5	5
15	1524	2,3-butanediol	creamy	5	5
16	1548	linalool	floral, citrusy	7	8
17	1568	1-octanol	floral	6	6
18	1606	terpinen-4-ol	rancid	4	6
19	1647	γ-butyrolactone	cheesy, burnt sugar	7	8
20	1690	α-terpineol	green, violet	5	7
21	1714	3-(methylthio) propanol	boiled potato, rubber	5	8
22	1720	ethyl acetylacetate	fruity	4	4
23	1748	citronellol	floral	6	6
24	1781	ethyl-4-hydroxy-butanoate	fruity, floral	5	5
25	1831	geraniol	floral, citrusy	7	8
26	1864	benzyl alcohol	citrusy, sweet	4	7
27	1908	2-phenylethanol	floral, rose	7	8
28	1974	unknown	plastic, roasty	5	7
29	2029	diethyl malate	caramel	7	6
30	2061	octanoic acid	sweaty	3	4
31	2120	eugenol	clove	6	5
32	2164	unknown	cotton candy	5	7
33	2193	4-vinyl-2-methoxy-phenol	nutty, spicy	4	6
34	2285	ethyl-2-hydroxy-3-phenyl propanoate	caramel, roasty	5	7
35	2318	unknown	rubber	6	5

[a] LRI, Linear retention index calculated on a DB-Wax capillary column. [b] Odor description as perceived by panelists during olfactometry. [c] Average intensity. [d] Number of judges who detected an odor.

was the second most abundant alcohol, being present at levels higher than its perception threshold [10 000 μg/L in water/ethanol (90 + 10, w/w) according to Guth (*20*)] in orange wine. The intensity value of 2-phenylethanol was 7. At the present study, this compound has been identified as providing a floral and rose odor by panelists, in agreement with the literature for Merlot and Cabernet Sauvignon wines (*21*), freshly distilled cognac (*39*), and Bavarian pilsner-type beer (*41*). The levels of 2-phenylethanol in wines depends upon must composition, yeast species, and fermentation temperatures (*14*).

Terpenes. (*E*)-β-Ocimene, (*Z*)-furan linalool oxide, linalool, terpinen-4-ol, α-terpineol, citronellol, and geraniol have been detected in orange wine extracts. Some of these compounds, namely, linalool, terpinene-4-ol, and α-terpineol, were found in significant amounts, and the concentration of linalool (1640 μg/L) was the highest. On the basis of the detection frequencies and intensity values, the most powerful terpene odorants of orange wine were linalool and geraniol, both described as having a floral and citrusy note. They were detected by all eight panelists. Both compounds were present at concentrations higher than their corresponding odor threshold values [15 μg/L for linalool and 30 μg/L for geraniol in water/ethanol (90 + 10, w/w) according to Guth (*20*)]. Linalool makes a positive contribution to orange flavor in combination with several other orange volatiles (*11*). Schieberle et al. (*42*) and Buettner et al. (*43*) indicated that linalool was characterized by floral odor for fresh and processed mandarin oranges and peel oil of clementines. The odorant α-terpineol was encountered with green-violet odors by seven panelists. This compound is a degradation product of limonene, the major orange oil constituent, and is known as the prime contributor to the off flavor in orange juice at levels of 2 ppm or higher. The α-terpineol has been found to increase linearly with storage time and has also been suggested for use as an indicated compound for heated and stored juice (*44*). The concentration of α-terpineol was 836 μg/L and found to be below 2 ppm in orange wine. Within terpenes, terpinen-4-ol was identified as the compound that provides undesirable contribution to the rancid odor in orange wine extract. It does not have a strong impact on the overall odor because it is perceived by a low-intensity value (4). As previously stated, this compound was detected with a pine and musty odor in key lime (*Citrus aurantifolia* Swingle) essential oils (*45*) and a dusty odor in aqueous banana essence (*46*). Our results were also similar to those obtained by Pérez-López and Carbonell-Barrachina (*47*), who concluded that the increases in the sensory intensity of off flavors in mandarin juices detected by the trained panel could be related to the increases in terpinen-4-ol and α-terpineol.

Esters. Among ester compounds, ethyl butanoate (fruity sweet), hexyl acetate (floral), ethyl acetylacetate (fruity), ethyl-4-hydroxy-butanoate (fruity floral), diethyl malate (caramel), and ethyl-2-hydroxy-3-phenyl propanoate (caramel roasty) were detected as aroma-active compounds in orange wine extracts (**Table 3**). Esters are known to contribute to the "top note" of orange aroma (*48*) and are formed mostly through esterification of alcohols with fatty acids during the fermentation and aging period. Ethyl butanoate was the only ester compound that was detected by all eight panelists. This compound is generally the major volatile ester in orange juice and an important contributor to desirable flavor in orange products. Shaw (*11*) reported that ethyl butanoate was present in varying amounts (80–1400 mg/L) in orange juices. Diethyl malate could also be an important aroma contributor in this fraction based on its very strong GC−O intensity. The intensity value of this ester was 7. The two aroma-active hydroxy fatty acid esters detected in wines were ethyl-4-hydroxy-butanoate and ethyl-2-hydroxy-3-phenyl propanoate. These esters are characterized by fruity-floral and caramel-roasty notes, respectively. As previously stated, ethyl-2-hydroxy-3-phenyl propanoate was detected with a smoky odor in Chinese liquors. Hydroxy esters are formed from the esterification of corresponding hydroxy fatty acids, which could be produced from the reduction of keto acids (*49*).

Volatile Phenols. Eugenol and 4-vinyl-2-methoxyphenol were identified as aroma-active volatile phenols of orange wine. Volatile phenols detected in wine samples can originate from *p*-coumaric and ferulic acids by enzymatic or thermal decarboxylation (*13*). At the present study, eugenol was detected by five panelists and described as having a clove odor (**Table 3**). Similarly, eugenol has been identified as a potent aroma-active compound in fresh apricots, providing a clove-like odor with a low threshold value of 6 μg/L in water (*50*) and a clove-balsamic odor in aged Spanish red wines (*51*). Another aroma-active volatile phenol, 4-vinyl-2-methoxyphenol, was detected with a nutty and spicy odor by six panelists. This odorant has been

reported earlier as a smoky-clove-like note in a Bavarian pilsner-type beer by Fritsch and Schieberle (*41*) and a phenolic-smoky odor in aged Spanish red wines by Culleré et al. (*51*).

Lactone. γ-Butyrolactone was only lactone compound that was detected by GC−O analysis. This compound was detected by all eight panelists. It contributes to the characteristics cheesy and burnt sugar odor of the orange wine based on its strong aroma intensity value (7). The odor threshold value of γ-butyrolactone was 35 μg/L in a 10% water/ethanol mixture according to Culleré et al. (*51*).

The other two aroma-active compounds identified in orange wine were furfural (pungent) and octanoic acid (sweaty). These odorant were detected by six and four panelists, respectively, with a low intensity value (3).

Seven unknown compounds may contribute to the global aroma of orange wine. Unknown 3 (LRI = 1118), with a plastic odor, was perceived by all of the panelists. In addition, unknown 12 (LRI = 1438), 28 (LRI = 1974), and 32 (LRI = 2164) with bread-roasty, plastic-roasty, and cotton candy odors, respectively, were detected by seven panelists. The unknown 35 (LRI = 2318) was determined in orange wine as providing a rubber odor with the highest intensity value (6).

In summary, this study revealed potent odorants that are responsible for the overall flavor of the cv. Kozan orange wine using two GC−olfactometric methods, including detection frequency and time intensity. On the basis of the results, aroma-active compounds of orange wine are the results of the interaction of 35 odorants. The main aroma-active compounds were predominantly alcohols, followed by terpenes and esters. Within these, ethyl butanoate, 3-methyl-1-pentanol, linalool, γ-butyrolactone, 3-(methylthio)-propanol, geraniol, and 2-phenylethanol were the most powerful contributors to the aroma of the orange wine because they were perceived by all eight panelists.

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