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Preliminary communication

CHARACTERIZATION OF THE EFFECT OF SHORT-TERM HIGH TEMPERATURE AND VIBRATION ON WINE BY QUANTITATIVE DESCRIPTIVE ANALYSIS AND SOLID PHASE MICROEXTRACTION-GAS CHROMATOGRAPHY-MASS SPECTROMETRY

P. Zhao, H. Wang and H. Li*

College of Enology, Northwest A&F University, Taicheng Road 3, 712100 Yangling. China

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The effect of short-term higher ambient temperature (HT) and continuous vibration (CV) treatment was comparatively characterized by sensory evaluation and chemical analysis. Results of quantitative descriptive analysis of modified frequency (MF) showed that HT causes both in red wine and white wine a decrease of fruity and floral characters, an unbalance of taste, and a shortness of aftertaste length. CV wine showed very close sensory characters to control in most terms evaluated. Seventy-four volatile compounds were quantitative analysed by solid phase microextraction-gas chromatography-mass spectrometry, and the principal component analysis (PCA) was conducted on the 23 volatiles of highest odour activity value (OAV). The concentrations of potential fruity and floral aroma attributors like isoamyl acetate, ethyl butanoate, ethyl 2-methylbutanoate, ethyl hexanoate, ethyl octanoate, β-damascenone, and linalool were lower in HT wine than that in original wine and CV wine.

Keywords: wine, higher temperature, vibration, storage, sensory evaluation, chemical analysis

Beside winemaking techniques, wine conveyance and storage also contribute to wine quality. With the fast rise of global trade and online shopping, more wines are shipped. However, because of limited availability of refrigerated containers and the relatively high shipping charges of bottled wine, most wines are stored and shipped under bad conditions.

Higher ambient temperature or continuous vibration during shipping and storage of bottled wine were cited as the possible culprits to cause defective sensory characteristics of wine. Wine quality depends upon its composition, especially aroma-active compounds with higher concentration and lower threshold in wine. Francis and co-workers (1994) reported diminished floral characters and increased oak, honey, and smoky attributes of Chardonnay and Semillon wines caused by heated storage (45±5 °C for 20 days). In the study of Somers and Pocock (1990), a ten-day anaerobic heat treatment (42–45 °C) of Syrah wines caused colour and phenolic composition changes, and the astringency of wine was also reduced. De and Noble (1997) heated two vintages of Chardonnay wine at 40 °C. They noticed that even 5 days could trigger aroma changes in young unoaked Chardonnay, which is 7–9 days for oaked wine. By descriptive analysis, significant decrease in the fruity and floral-like aromas and increase in the oak, butter/vanilla, honey, tea/tobacco, and rubber aromas were found after fifteen days of heating treatment. Robinson and co-workers (2010) explored the effects

* To whom correspondence should be addressed.
Phone: +86 29 87082805; fax: +86 29 87082805; e-mail: lihuawine@nwsuaf.edu.cn

of shipping conditions including three different storage temperature treatments and samples travelling in the trunk of a car on sensory attributes and volatile composition of commercial white and red wines. Both sensory and analytical data indicated that higher temperatures would cause significant changes in wines. Butzke and co-workers (2012) made a similar conclusion that the elevated temperature wine is exposed to has a profound impact on aging reactions and sensory quality, resulting in a more aged wine compared with conventional cellar storage.

Chung and co-workers (2008) studied the effect of vibration on some physico-chemical characteristics of a commercial red wine. Vibrations during wine aging could result in significant changes of the physico-chemical properties in bottled wine. With constant vibration, the evolution of the wine was substantially accelerated.

Oxygen (Wirth et al., 2012; Sáenz-Navajas et al., 2014) and sulphur dioxide (Raposo et al., 2016) were able to influence the chemical and sensory properties of wine during bottle storage. There is no literature indicating that oxygen and sulphur dioxide could change the characteristics of wine during a short time of bottle storage.

The objective of this study was to confirm the influence of excessive ambient temperature and vibration on the sensory characters and chemical attributes, and especially on volatile compounds of wine. Besides, the main correlations between change of chemical composition and changes of sensory properties were to be verified.

1. Materials and methods

1.1. Wines

Commercial dry white wine (Chardonnay, vintage 2012, corked) and dry red wine (Cabernet Sauvignon, vintage 2012, corked) were purchased from wine cellars. All wine samples involved in this study were from the same lot and kept unopened and stored at room temperature before treatments. Ethanol content, extract, reducing sugars, titration acidity, pH, volatile acidity, total SO_2, total monomeric anthocyanin, and phenols of the sample wines were analysed (data shown in supplementary table) with the methods of GB/T 15038-2006 (GB (Guo Biao, 2006)).

1.2. Treatments

According to publications (Chung et al., 2008; Robinson et al., 2010), high temperature of 40 °C (HT) and shaker provided continuous vibration (CV) were two factors to induce changes in wine. Sample wines were sealed and put into an incubator at 40 °C for fifteen days, labelled as TW for white wine, TR for red wine. Other samples were sealed and put in a shaker at 150 r.p.m. for fifteen days, labelled as VW for white wine, VR for red wine. Wines put in a room with a temperature of 20±3 °C, with no vibration were regarded as control sample, labelled as CW for white wine, CR for red wine. Storage conditions in this study were dark with 60–70% relative humidity. All treatments were done in triplicates and samples were put at room temperature before sensory evaluation and chemical analysis.

1.3. Reagents and chemical standards

Chemical standards used in this study were purchased from Sigma-Aldrich (St. Louis, MO, USA), TCI America (Portland, OR, USA), EKC Inc. (Rosemont, IL, USA), and EMD Chemical Inc. (Gibbstown, NJ, USA), and their purities were >90% in all cases. Details of the compounds are shown in a supplementary table. Milli-Q quality water was obtained from a Milli-Q purification system (Millipore, Boston, MA, USA). Methanol (HPLC grade), dichloromethane (HPLC grade), and acetonitrile (HPLC grade) were from EM Science (Gibbstown, NJ, USA). Standard stock solutions were prepared in methanol, except for fatty acids that were prepared in acetonitrile. All solutions were stored in dark bottles at –20 °C.

1.4. Sensory evaluation

The panel (25 experienced panellists) was recruited from students of faculties in the College. Panellists were trained over 50 days using a "Le Nez du Vin" aroma kit. Panellists were required to identify the different sample from the presented 3 samples. The descriptive analysis was performed as described by Li and co-workers (2008). The panellists were required to score wine from the aspects of appearance, aroma, mouthfeel, and overall quality, respectively using a hundred-mark system for each term. The Quantitative Descriptive Analysis (QDA) was performed as described by Tao and co-workers (2009) to determine the most important terms describing the aroma characteristics of wine samples in this study. Red and white wines in a balanced and completed block design were presented (in triplicates) to panellists. They were required to use 5 to 6 most significant terms to describe the wine aroma, mouthfeel, and taste. Panellists were also asked to score the intensity of each term using a 5-point scale. The data processed were a mixture of intensity and frequency of detection ("modified frequency", MF), which was calculated with the formula proposed by Tao and co-workers (2009).

1.5. Qualitative and quantitative analysis

Main volatiles in the sample were quantitated by headspace SPME-GC-MS as described by Fang and Qian (2006) and Zhao and co-workers (2017). A DVB/CAR/PDMS (Divinylbenzene/Carboxen/Polydimethylsiloxane, Supelco Inc., Bellefonte, PA) fibre was used. Compound separation was achieved with a ZB-wax column (30 m×0.25 mm i.d., 0.5 µm film thickness, Phenomenex). Agilent 6890 gas chromatograph equipped with Agilent 5973 mass selective detector (Agilent, Santa Clara, CA) was used. Quantification was achieved using selected ion monitoring. Each sample was analysed in triplicates. Standard calibration curve was prepared by spiking known amounts of standards into a 20-ml vial with 2 ml of synthetic wine (12% ethanol (v/v), 3.5 g l tartaric acid, pH 3.5) and 8 ml of citrate buffer (0.5 g l^{-1}, pH 3.5, saturated with NaCl). Twenty microlitres of internal standard (50 mg l^{-1}, 4-octanol in methanol) was added (Zhao et al., 2017). Results were calculated with Chemstation software (v10.0, Agilent Technologies, Santa Clara, CA)

1.6. Statistical analysis

Tables and spider charts were crafted by Microsoft excel 2013. Principal component analysis (PCA) was performed on quantified chemical compositions by Canoco 5.0 (Microcomputer Power Inc., Ithaca, NY, USA). ANOVA Duncan's t test was carried out using SPSS 20.0 (SPSS Inc., Chicago, IL, USA).

2. Results and discussion

2.1. Sensory evaluations

Table 1. Statistic table of the results of triangle test

Sample combinations	Right answers	[a] X^2	Level of significant difference
CW & TW	17	12.01	0.1%
CW & VW	7	0.13	none
CR & TR	15	6.85	1%
CR & VR	9	0.25	none

[a]: Chi-square (X^2) distribution, $X^2=([4X_1-2X_2]-3)^2/8N$, X_1 and X_2: correct and false answers; N: the total number of judgements

Results of the triangle test are shown in Table 1. HT treatment made both white and red wine samples taste different at 0.1% and 1% levels. No sensory character changes of white or red wine samples were observed for CV treatment. Based on scores given by 25 panellists (Fig. 1), HT treatment stressed the wine in the terms of aroma, mouthfeel, and overall quality. CV treatment had no effect on wine quality.

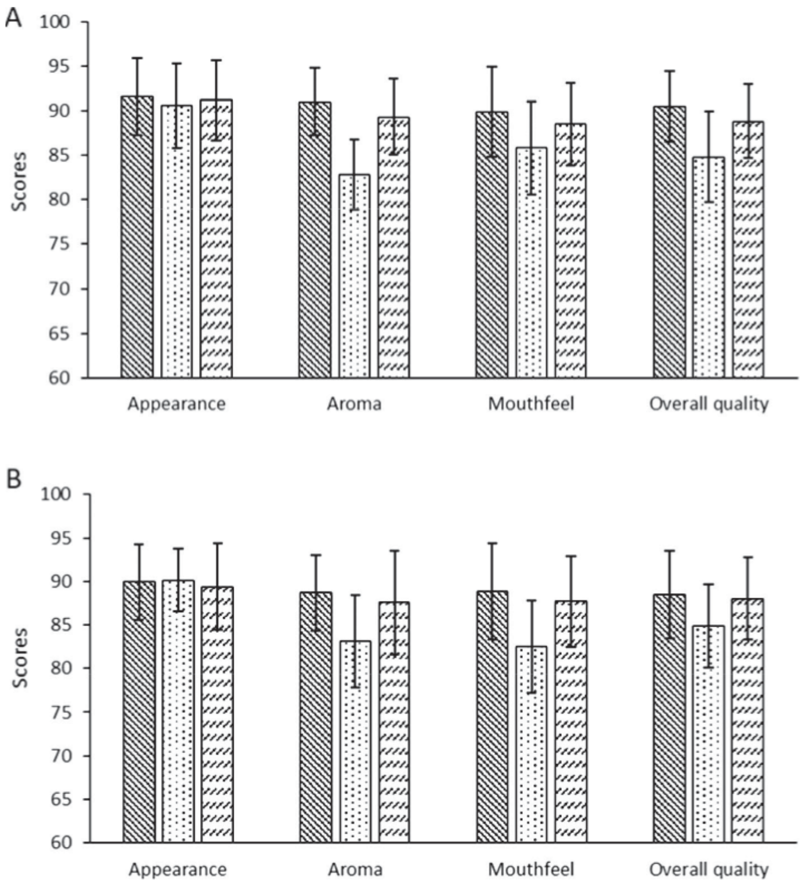

Fig. 1. Bar chart of sensory evaluation scores of white (A) and red (B) wine samples
A. ▨: CW; ⚃: TW; ▩:VW; B. ▨: CR; ⚃: TR; ▩: VR

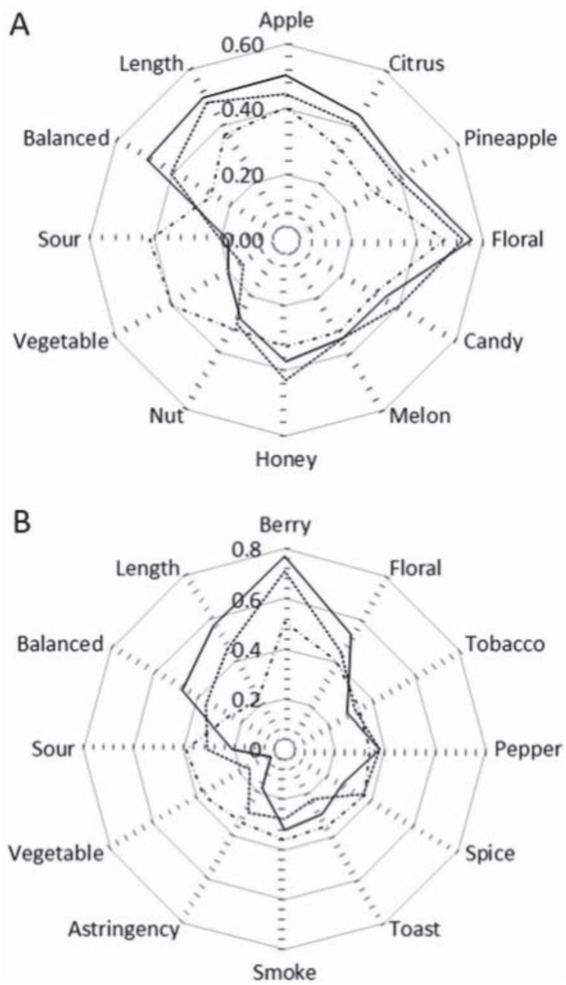

Fig. 2. Spider chart of the top 12 modified frequencies (MF) of descriptors in white (A) and red (B) wine sample
——: CW; ------:SW; ·······: RW; ——: CR; ------:SR; ·······: RR

According to the results of QDA and MF, 12 terms were drafted to describe the sensory attributes of wine samples (Fig. 2). For control white wine, floral, apple, length, balanced terms were detected as the most important attributes. After HT treatment, vegetable and sour terms were given higher values, floral and apple terms lower. VW was found closer to the control by the sensory terms. For red wine samples, blackcurrant, blackberry, blueberry, raspberry, etc. descriptors were grouped into the berry term. As shown in Figure 2B, terms berry, length, floral, balanced, and pepper were rated as top sensory terms in CR. Terms of vegetable, sour, and astringency presented higher MF values in TR; terms berry, floral, balanced, and length terms received lower MF values. VR sample was given lower values at terms floral, smoke, balanced, length, and toast, higher in astringency, sour, and spice.

Table 2. Concentrations of volatile compounds quantitated in wine samples (in μg l^{-1}, mean ± SD) with odour activity values (OAV)>0.5

Compounds	[1]Threshold	White wine			Red wine		
		CW	TW	VW	CR	TR	VR
Ethyl butanoate	20	123±13	106±15	126±12	119±25	108±9	169±14
Ethyl hexanoate	14	124±11a	75±6b	83±10b	141±12a	76±8b	188±10c
Ethyl octanoate	5	395±50	328±51	356±36	286±58a	99±10b	222±30a
Ethyl isobutanoate	15	194±30	177±18	161±14	321±38	274±45	347±33
Ethyl 2-methylbutanoate	18	31±4a	12±1b	26±3a	38±8a	28±3b	59±9b
Ethyl isovalerate	3	26±2a	19±3b	30±2a	86±7a	47±17b	117±12c
Isoamyl acetate	30	289±24a	186±12b	267±18a	203±26a	140±23b	192±16a
Ethyl acetate	12300	44667±473a	47266±947b	50863±732c	49574±1013	51115±894	49900±880
Isobutyl alcohol	40000	101959±9167a	82026±11732b	65519±17773c	53543±5954a	72970±4171b	68619±8142b
Isoamyl alcohol	30000	209357±12697	179339±48119	200339±21066	199426±30819	220472±11549	213256±23249
2-phenethanol	14000	19046±1571a	24384±1102b	25406±2816b	18721±1551ab	17201±1588a	20378±1928b
Butanoic acid	173	157±7a	236±14b	191±7c	267±9a	247±23a	400±53b
Hexanoic acid	420	699±17	734±47	746±36	580±44	580±33	522±84
Octanoic acid	500	383±57	391±90	380±83	513±121a	463±31a	247±41b
3-methylbutanoic acid	33	120±3a	141±8b	144±6b	189±6	191±16	187±26
Eugenol	6	18±3	16±2	15±2	23±4a	14±2b	24±5a
Linalool	15	12.8 ± 0.4a	8.4 ± 0.3b	12.3 ± 0.3a	10.1 ± 0.6a	7.4 ± 0.3b	9.5 ± 0.1a
Rose oxide	0.2	0.19±0.04	0.14±0.02	0.16±0.03	0.19±0.02a	0.19±0.03a	0.13±0.01b
β-damascenone	0.05	1.14±0.2	0.97±0.09	1.17±0.44	1.85±0.25a	1.42±0.11b	1.99±0.54a
Trans-whiskylactone	32	0.66±0.24a	1.13±0.2b	0.89±0.6ab	17±2.5a	17.1±1.4a	28.4±9b
2-aminoacetophenone	1.4	0.45±0.20	0.43±0.15	0.8±1.1	2.5±1.2	2.8±0.8	2.3±0.2
Acetaldehyde	500	4747±221a	6894±63b	6479±361b	9814±420a	11711±1394b	10814±961b
3-isobutyl-2-methoxypyrazine	0.002	0.003±0.002	0.004±0.002	0.003±0.002	0.006±0.001	0.007±0.002	0.007±0.003

[1]: Odour threshold of the volatiles were presented in μg l^{-1}, measured in model wine, water/ethanol (90/10, w/w) unless otherwise indicated, referenced from literature (Guth, 1997; Ferreira et al., 2000; López et al., 2002; Li et al., 2008; Parker et al., 2012).
[2]: Different letters within rows indicate statistical differences by Duncan test (P<0.05)

The observed results of sensory analysis above were supported by other chemists. In the research of De and Noble (1997), they found that younger unoaked Chardonnays only needed five days of elevated temperature (40 °C) storage to present a significant difference in aroma in triangle tests. Similar results were obtained by Owens and co-workers (1998).

2.2. Chemical analysis

The selected 74 volatile compounds in sample wines were quantified, and the odour active values (OAV, the ratio of concentration data and odour threshold) were calculated. As shown in Table 2, a concentration decrease of ethyl esters of linear fatty acid derivatives and branched acid derivatives in HT treatment sample was observed in both white and red wine samples. Especially ethyl butanoate, ethyl hexanoate, ethyl octanoate, ethyl 2-methylbutanoate, and ethyl isovalerate, which have low odour threshold, contributed to the fruity attributors of wine. Among the quantified aroma compounds, 23 volatiles were with OAV≥0.5. They were analysed by PCA according to their concentrations. The bi-plot (Fig. 3) revealed 88.2% of total variance (PC1 and PC2 contributing 72.5% and 15.7%, respectively). White wine samples CW, VW, and TW were located in the second and third quadrant, and red wine sample CR, VR, and TR were located in the first and fourth quadrants.

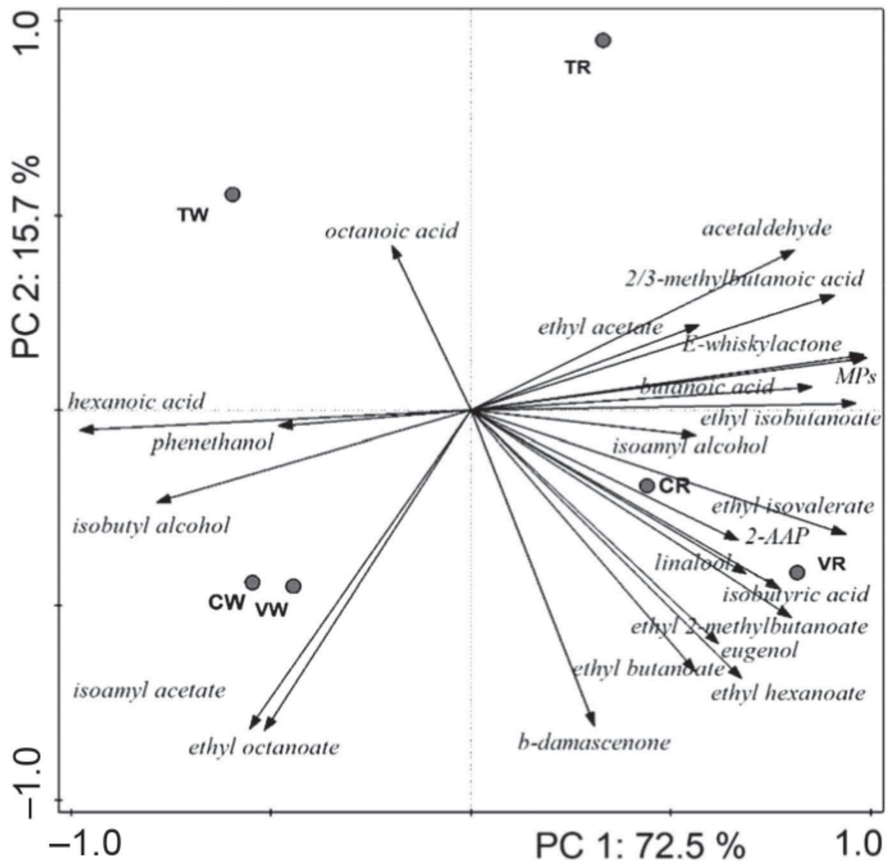

Fig. 3. PCA biplot of chemical composition with OAV≥0.5 in wine sample

Compared to CW and VW, TW showed lower correlation of isoamyl acetate, ethyl butanoate, ethyl 2-methylbutanoate, ethyl hexanoate, ethyl octanoate, etc. esters (fruity-like odour), and β-damascenone (maple syrup and tobacco like odour), linalool (citrus and floral like odour), eugenol (violet and spice like odour), etc., which were potential high aroma contributors in wine. A similar trend was obtained in red wine samples. CR and VR samples were determined to have higher OAV of isoamyl alcohol (brandy like odour), 2-aminoacetophenone (grape and sweet odour), and *E*-whiskylactone (toast nut and creamy odour). 2-Aminoacetophenon was reported as an atypical aging off-flavour compound, which was released from the oxidative degradation of phytohormone indole-3-acetic acid (Schmarr et al., 2016). The results of HT treatments observed were consistent with the work carried out by Pérez and co-workers (2003), Wicks (2009), and Robinson and co-workers (2010). Vibration possibly led to a rapid browning of wine, an increase of the formation of ethyl carbamate (EC) (Stevens, 1995), and other destructive oxidative reactions, which can help release glucose-bound flavour precursors like fruity-floral terpenes prematurely (Francis et al., 1994), due to a decline in protective free sulphur dioxide (Ough, 1985).

3. Conclusions

The sensory evaluation in this paper revealed that heat exposure changed the organoleptic properties of wine: a weaker fruity and floral character, a muted and unbalanced overall perception, a shorter aftertaste length were experienced.

Chemical analysis of the major contributing volatiles demonstrated that wine samples exposed to high temperature showed significantly lower concentrations of potential fruity and floral aroma contributors like esters, β-damascenone, and linalool. No direct effect of short time vibration on wine could be detected. Further study of the mechanism of the changes of chemical and sensory properties affected by bad storage conditions would be of great significance in the future.

During the shipping and storage of wine, no heating and vibration should be allowed, as these, for even as short as 15 days, are harmful for wine quality. In the future, it would be inspiring to study whether a deliberate use of regulated storage conditions could accelerate planned wine aging.

Conflict of interest

The authors declare that they have no conflict of interest.

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