Optimizing extraction method of aroma compounds from grape pomace

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ABSTRACT

Grape pomace is a major wine industry by-product. Extraction of volatile compounds from grape pomace is rarely explored. A cost-effective method was developed in this study for aroma compounds extraction from grape pomace with the potential for industrial application. Based on the solvent extraction procedure, experimental factors including pre-treatment, enzymatic hydrolysis time, solvent concentration and distillation time were investigated to optimize the extraction process. Volatile compounds of the pomace extract were analyzed using headspace solid-phase microextraction gas-chromatography mass spectrometry (HS-SPME-GC-MS) method. Results revealed that enzymatic hydrolysis was the optimal pre-treatment method. A maximum extraction efficiency was achieved under 48 hours of enzymatic hydrolysis, 70% of ethanol concentration and 20 minutes of distillation. A total of 65 volatile compounds were identified in the extract, including 16 alcohols, 1 alkane, 1 aldehyde, 9 esters, 3 ketones, 4 phenols, 6 terpenes and 1 furan, of which 15 volatiles were determined as odor-active compounds. This study developed a feasible extraction technique to recycle the under-utilized by-products from wine industry to produce aroma/flavor food additives.

Keywords: grape pomace, volatile compounds, ethanol, distillation, enzymatic hydrolysis

Practical Application: This study develops a cost-effective method for aroma compounds extraction from grape pomace with the potential for industrial application as food additives

1. Introduction

The organoleptic characteristics of grapes and wines are associated with free volatiles and non-volatile glycosides (Liu, Zhu, Ullah, & Tao, 2017). In the grape, the main aroma precursors are glycosidically bound compounds that are present in higher levels than their free volatile counterparts and are predominantly derived from the skin rather than pulp and juice (Liang et al., 2020). Many of these compounds can be hydrolyzed by enzymes or acids to release the aglycones as free aromatic compounds during wine fermentation (Liu et al., 2017). In most white winemaking, grapes are pressed to obtain the juice before fermentation, leaving pomace by-products abundant in non-volatile glycosidic aroma precursors (Darias-Martín, Díaz-González, & Díaz-Romero, 2004). These aroma glycosides can be classified according to their aglycones including terpenes, C₁₃-norisoprenoids, phenols, aliphatic alcohols, aliphatic acids, C₆ compounds, benzenic and phenolic acid derivatives (Liu et al., 2017), which are potential flavoring additives or drugs/medicines for various foods, beverage, pharmaceuticals and cosmetics (Caputi & Aprea, 2011). Therefore, both free volatiles and aroma glycosides need to be considered when extracting aromatic compounds from grape pomace.

Various techniques have been employed for bioactive compounds extraction, such as Soxhlet extraction, microwave-assisted extraction, ultrasound-assisted extraction and supercritical fluid extraction, of which the main limitations are thermal degradation, environmental unfriendliness, high-cost equipment, etc. (Azwanida, 2015). For instance, the thermolabile compounds, anthocyanins and tannins, might undergo degradation at high temperature during microwave-assisted extraction, and the application of supercritical fluid extraction is limited by the expensive equipment (Liazid, Palma, Brigui, & Barroso, 2007; Azwanida, 2015). A few studies have reported the aroma compounds extraction from grape pomace (Vasserot,

Arnaud, & Galzy, 1993; Muñoz-González, Rodríguez-Bencomo, Martín-Álvarez, Moreno-Arribas, & Pozo-Bayón, 2014), where multiple factors determine the process optimization. Pre-treatment is a critical factor assisting in extraction, where enzymatic hydrolysis and ultrasound are the two recommended techniques (Cocito, Gaetano, & Delfini, 1995; Zhang et al., 2019). The role of enzymes in hydrolysis is to disrupt or degrade cell walls and membranes to increase permeability, enabling better release of aroma glycosides (Pinelo, Arnous, & Meyer, 2006), which can then be hydrolyzed into volatile aglycones and glycones via the breakdown of the glycosidic bonds (Pogorzelski & Wilkowska, 2007). Enzyme pre-treatment can lead to less time and solvent consumption, as well as mild processing environments such as lower extraction temperatures, while promoting the yield and quality of the product, thereby conforming to the extraction principles and green chemistry. However, the treatment is limited by the high cost of enzymes. In addition, the currently available enzyme treatments might not attain a complete breakdown of plant cell walls, which would negatively impact aroma glycosides release and thus extraction yields. Further, the enzyme activity may vary with certain environmental conditions like nutrient availability, temperature and the percentage of dissolved oxygen (Puri, Sharma, & Barrow, 2012). On the other hand, ultrasound energy can induce cavitation in a liquid during extraction, thus the high-speed jets of liquid could be produced as the asymmetric cavity collapses, resulting in a high analyte transport from the solid particles to the liquid phase (Peña-Farfal et al., 2004). Ultrasound treatment is recommended for lower energy and time consumption, higher extraction yield and enhanced extraction of thermolabile compounds. The treatment has also been reported with multiple drawbacks mainly including the wave attenuation in the dispersed phase and the decreased sound wave amplitude with distance (Liang, Zhang, & Fang, 2020). In recent years, ultrasound treatment has been applied in the extraction process of virgin olive oil with enhanced aroma profiles at both semi-industrial and industrial scales (Almeida, Valli,

Bendini, & Gallina Toschi, 2017; Taticchi et al., 2019), indicating its great potential in aroma extraction from plant materials at large industrial scale. Incubation time is a decisive factor when pre-treated with enzymatic hydrolysis. Sufficient hydrolysis time of hours to days is required for a complete substrate hydrolysis, however the products can be easily volatilized and lost after a prolonged hydrolysis (Zhang et al., 2019). Regarding the extractant, a wide range of solvents can be used in the extraction, including water, methanol, ethanol and other solvents (Chemat et al. 2011). Food grade ethanol is an economical and widely used solvent considered as Generally Recognized as Safe (GRAS) substance (Food and Drug Administration, 2016), of which the concentration might be a significant factor. Vacuum distillation is generally applied to reduce the system pressure and to achieve lower boiling point, therefore the volatile compounds can be more easily volatilized, condensed and collected (Wu et al., 2019). Distillation time presents as an important factor since different collection time could result in different volatile compositions depending on the differences in compounds volatility.

In the present work, we explored an economic and feasible extraction technique to recycle aromatic compounds from grape pomace, which has the potential to be implemented at industrial scale. Multiple factors potentially affecting the efficiency of aroma extraction were investigated, including pre-treatment methods, enzymatic hydrolysis time, solvent concentration and distillation time, and the aroma profile of the resultant extract was evaluated using HS-SPME-GC-MS. To our knowledge, this is the first study to report the optimization of the enzyme-assisted extraction of aroma compounds from grape pomace, which opens a window for further research on the utilization of wine industry by-products to produce food flavor additives.

2. Materials and Methods

2.1. Chemicals

Chemicals including sodium carbonate anhydrous, sodium chloride, ethanol, SPME fiber assembly polydimethylsiloxane/divinylbenzene (PDMS/DVB) (df 65 μm , Fused Silica/SS), n-alkanes (C7–C30), 4-octanol were purchased from Sigma Aldrich (Castle Hill, NSW, Australia). Rapidase commercial enzyme (pectinase, α - and β -glycosidase) were purchased from DSM Nutritional Products (Wagga Wagga, NSW, Australia). Headspace screw top clear vials (20 mL) and magnetic PTFE/sil hdsp cap were purchased from Agilent Technologies (Santa Clara, California, USA). Purified water was obtained from a Millipore Milli-Q water purification system (Millipore Australia, North Ryde, NSW, Australia).

2.2. Sample preparation

Freshly pressed and unfermented *Vitis vinifera* cv. Chardonnay grape pomace was collected from a commercial winery in the Geelong wine region, Victoria, Australia. Fresh grape pomace was stored in zip-lock bags, transported on dry ice to our laboratory and stored at -20 °C until processing.

2.3. Experimental design

The optimization of aroma compounds extraction was performed through four consecutive steps investigating pre-treatment, enzymatic hydrolysis time, ethanol concentration and distillation time (Fig. 1). All treatments followed a similar procedure: frozen grape pomace was ground with liquid nitrogen, and then sieved (500 μm sieve) to obtain powder. Fifty grams of pomace powder was mixed with 30 mL ethanol/Milli-Q water solution, and the pH of the mixture was adjusted to 5.0 using sodium carbonate to maximize enzyme activity followed by the pre-treatments of ultrasound and/or enzymatic hydrolysis. In ultrasound

treatment ($T_{\text{ultrasound}}$), a tip sonicator SFX150 (Branson Ultrasonics, Danbury, Connecticut, USA) was applied to the mixture at a frequency of 50 kHz for 15 min; In enzyme treatment (T_{enzyme}), Rapidase[®] revelation aroma commercial enzyme (Pectinase ≥ 25000 AVJP/g, β -glucosidase ≥ 4000 BDG/g, DSM Food, USA) was added at 0.9 g/10 mL into the sample vial to initiate enzymatic hydrolysis; In combined ultrasound and enzyme treatment ($T_{\text{ul+en}}$), a tip sonicator SFX150 was used to treat the mixture with rapidase commercial enzyme (0.9 g/10 mL) at a frequency of 50 kHz for 15 min. The treated samples were then shaken at 120 rpm at 35 °C for 48 h. Once the optimal enzymatic hydrolysis pre-treatment was determined, the time of hydrolysis was evaluated by incubating samples at 120 rpm and 35 °C for 24 hr ($T_{\text{en-24h}}$), 48 hr ($T_{\text{en-48h}}$) and 72 hr ($T_{\text{en-72h}}$), respectively. Furthermore, 0% ($T_{\text{et-0\%}}$), 30% ($T_{\text{et-30\%}}$), 50% ($T_{\text{et-50\%}}$) and 70% ($T_{\text{et-70\%}}$) of ethanol in Milli-Q water were added to the sample as the extraction solvents to evaluate their efficiencies in the extraction. The samples were fully mixed and centrifuged at 3,000 rpm for 15 min, and the supernatants were collected and subjected to distillation using Hei-VAP rotary evaporator (Heidolph Instruments, Schwabach, Bavaria, Germany) at 40 °C and the condensates were collected in receiving flask after 20 min ($T_{\text{t-20}}$) and 40 min ($T_{\text{t-40}}$) for each sample.

2.4. Analysis of volatile compounds

2.4.1. Sample preparation and headspace solid-phase microextraction (HS-SPME)

The extraction of volatile compounds was performed by HS-SPME method, using a 65 μm PDMS/DVB fiber installed on a PAL 3 multi-purpose automated sampler (Agilent Technologies). Prior to analysis, the fiber was pre-conditioned at 250 °C for 30 min in the GC injection port according to the manufacturer's specifications. The pomace volatile compounds were extracted according to the modified method of Zhang et al. (2017). Pomace extract (1 mL) was transferred into a 20 mL headspace vial with addition of 9 mL of Milli-Q

water, 2 g of sodium chloride and 20 μ L of internal standard 4-octanol (100 mg/L in ethanol). The vial was shaken at 35 $^{\circ}$ C for 20 min to reach equilibrium, then the SPME fibre was exposed to the headspace at 35 $^{\circ}$ C for 30 min for adsorption.

2.4.2. Gas chromatography-mass spectrometry (GC-MS) analysis

The GC-MS analysis was performed following the method described by Zhang et al. (2017) and Zhang et al. (2016) with slight modifications. In the analysis, an Agilent 6850 GC system (Agilent Technologies) was connected to a 5973 mass spectrometer using a J&W DB-Wax Ultra Inert GC column (30 m $\times$ 250 μ m $\times$ 0.25 μ m). The SPME fiber was desorbed in GC inlet at 200 $^{\circ}$ C for 10 min in splitless mode. Helium was used as the carrier gas at a flow rate of 0.8 mL/min. The oven temperature program was set as follows: an initial temperature at 40 $^{\circ}$ C and held for 2 min, then increased to 160 $^{\circ}$ C at a rate of 3 $^{\circ}$ C /min, and then further increased to 230 $^{\circ}$ C at 7 $^{\circ}$ C /min and held for 8 min. The mass spectrometer was operated in scan mode at 70 eV, with a mass acquisition range of 35-350 m/z; the transfer line, ion source and quadrupole temperature were set at 240 $^{\circ}$ C, 230 $^{\circ}$ C and 150 $^{\circ}$ C, respectively.

2.4.3. Compound identification and quantification

The GC-MS data were analyzed by Agilent G1701EA MSD ChemStation software (Version 1.4.20.0). The linear retention index (RI) of each compound was calculated using a series of n-alkanes (C7-C30). Each compound was identified by comparing mass spectra (MS) with NIST reference database (NIST 11.0), MS and retention indices (RI) with NIST Chemistry Webbook (NIST, 2019), and MS and retention times with available external standards. External standards were analyzed following the same conditions of samples. Peak areas of volatile compounds were integrated via target ions model. Each identified compound was quantified using the calibration curve of the corresponding standard compound as follows:

$$Conc. \left(\frac{\mu g}{mL} \right) = \left(\frac{ion\ peak\ area\ of\ each\ compound}{ion\ peak\ area\ of\ internal\ standard} \times Slope \right) + Intercept$$

Some identified compounds were semi-quantified using the standard curve of similar compound when the standard was not available as described in our previous publications (Luo et al., 2019; Zhang et al., 2016).

2.5. Statistical analysis

The experiment was done in triplicates and the results were reported as means $\pm$ standard deviations. The significant difference of means between each sample was determined by one-way ANOVA with Turkey test at 95% confidence level using Minitab (Minitab 19, Sydney, Australia). Odor active value (OAV) for each compound was calculated by dividing the compound concentration with aroma detection threshold summarized from published literatures (Ahmed, Dennison, Dougherty, & Shaw, 1978; Guth, 1997; Young & Suffet, 1999; Zea, Moyano, Moreno, Cortes, & Medina, 2001; Dragone, Mussatto, Oliveira, & Teixeira, 2009; Giri, Osako, & Ohshima, 2010; Jiang & Zhang, 2010; Javed et al., 2018).

3. Results and Discussions

3.1. Volatile compound profile of the grape pomace extract

A total of 65 compounds were detected from the Chardonnay grape pomace extract, where 33 compounds were identified and quantified based on external standards (ES), mass spectra (MS) and retention index (RI) from the NIST library 11.0 and NIST webbook (NIST, 2019), while 8 were only tentatively identified by MS and RI, and 24 were unidentified (Fig. 2). The identified volatile compounds can be subdivided into eight chemical groups, including 16 alcohols, 1 alkane, 1 aldehyde, 9 esters, 3 ketones, 4 phenols, 6 terpenes and 1 furan (Table 1 & 2). Several compounds were unidentified mainly due to their weak signals (Fig. 2), but the

MS information suggested that these compounds were most likely to be 10-23 carbon alkanes or their derivatives.

Alcohols were determined as the largest group in terms of the diversity and the concentrations in the pomace extract. Among 16 detected alcohols, 1-hexanol (19) was the most abundant one in accordance with a previous study (Garcia, Chacon, Martinez, & Izquierdo, 2003), while (*E*)-2-octen-1-ol (40) was also found at high concentration. These alcohols account for alcoholic, citrus, floral, sweet, green, fruity and vegetable aromas (TGSC, 2019). Phenols were the second most abundant group of volatiles. Four benzenoids including benzaldehyde (31), phenylacetaldehyde (41), benzyl alcohol (57) and 2-phenylethanol (59) were detected, with considerable high levels of benzyl alcohol and 2-phenylethanol, which were in accordance with the study of Garcia et al. (2003). These compounds are responsible for floral, rose, almond, honey and bready odors (TGSC, 2019). Terpenes were also detected with geraniol (56) as the dominant one, which contributes to fruity, rose, sweet and floral aromas (TGSC, 2019). The presence of nerol (55) and geraniol (56) as minor or trace constituents was in good agreement with the report from Garcia et al. (2003). Linalool (36) was detected at low concentration, while its derivatives such as terpendiol (Wilson, Strauss, & Williams, 1984) was not detected in the current study. Other compounds including alkane, aldehyde, esters, ketones, and furan were also identified and quantified in small quantities (Table 1 & 2). Hexanal (2) is a characteristic aldehyde with green, fatty and aldehydic odors (TGSC, 2019), which was detected at lower concentration compared to the study of Garcia et al. (2003). Esters, including 3-methylbutyl acetate (5) and ethyl benzoate (42), were also determined, where 3-methylbutyl acetate (5) is associated with sweet, banana and fruity aromas, while ethyl benzoate (42) has camomile, flower, celery and fruity odors (TGSC, 2019).

Though the volatile aglycones from multiple aroma glycosides were detected in pomace extract, not all of them contribute significantly to the overall aroma characters of grape pomace. Odor active values (OAVs) have been widely used to determine the sensorial impact of individual volatiles, and only those with OAVs greater than 1 can be considered as odor-active compounds (Guth, 1997). The impact of odor-active compounds on the overall aroma can be evaluated based on the OAVs and associated odor descriptor (Table 3 & 4). A total of 15 detected volatiles were determined as odor-active compounds, where fruity aromas were the predominant odor group in terms of diversity, while green aromas with high OAVs, represented a vital contribution to the overall aroma characteristics. Floral, citrus, herbal and earthy aromas also contributed to the aroma properties but with weaker sensorial impact. Overall, hexanal, (*E*)-2-octen-1-ol, phenylacetaldehyde and 1-octen-3-ol exerted stronger impacts on pomace aroma than other volatiles because of their higher OAVs. Future researches may be conducted to further separate the fruity aroma compounds from the green aroma compounds to produce flavor additives for different purposes.

3.2. Influences of pre-treatment on the volatile profile of pomace extract

Enzymatic hydrolysis and ultrasound were applied to grape pomace as pre-treatment techniques to investigate their effects on the aroma compounds extraction (Table 1). Results showed that the total volatile contents of $T_{\text{ultrasound}}$ and $T_{\text{ul+en}}$ extracts were significantly ($p \leq 0.05$) lower compared to that of T_{enzyme} extract, while there were no significant ($p > 0.05$) differences in the total alcohol and ester contents among three treatments and in the total content of terpenes between T_{enzyme} and $T_{\text{ul+en}}$. 3-Methylbutan-1-ol, (*E*)-2-hexen-1-ol, phenylacetaldehyde and 2-phenylethanol were detected in considerably ($p \leq 0.05$) higher amounts in T_{enzyme} extract. The extract from $T_{\text{ultrasound}}$ did not contain several volatile

compounds present in the extracts of T_{enzyme} and $T_{\text{ul+en}}$, including 2-methyl-1-propanol, hexanal, methyl hexanoate, beta-cyclocitral, alpha-terpineol and carvone. This implied that these compounds might exist only in glycosidically bound forms, but nil or low in free forms. OAVs also reflect different effects of enzymatic hydrolysis and ultrasound on the extraction outcome (Table 3). Ten and nine odor-active compounds were detected in the T_{enzyme} and $T_{\text{ul+en}}$ extracts, respectively, whereas 6 of them exerted the analogous odor impacts between two treatments. 8 odor-active compounds were found in the extract from $T_{\text{ultrasound}}$, and 6 of them showed no significant differences in OAVs compared to those of $T_{\text{ul+en}}$ extract. Higher OAVs of (*E*)-2-hexen-1-ol, hexanal and phenylacetaldehyde were observed in the T_{enzyme} extract compared to the $T_{\text{ultrasound}}$ and $T_{\text{ul+en}}$ extracts, potentially enhancing fruity and green notes (TGSC 2019). Compared to T_{enzyme} extract, 2-undecanone from $T_{\text{ul+en}}$ and $T_{\text{ultrasound}}$ contributed more significantly to the overall aroma of the extracts, which gave undesired tallow and musty odors (TGSC, 2019). Based on the above results, enzymatic hydrolysis was the optimal pre-treatment amongst the tested methods for higher concentrations of aroma compounds extracted from Chardonnay grape pomace, and the compounds mixture will give fruity and green notes simultaneously.

In the present study, the ultrasound and its combination with enzymatic hydrolysis accounted for the lower extraction yields compared to the enzymatic hydrolysis alone, which was inconsistent to the hypothesis that ultrasound should reduce the long enzymatic hydrolysis time via fast cell membrane disruption and improve extraction efficiency (Peña-Farfal et al., 2004). The outcome might be due to the high ultrasound frequency used in the study. According to Islam, Zhang, and Adhikari (2014), power ultrasound (20–100 kHz) can lead to the inactivation of enzymes by denaturing the protein either via the free radicals from sonolysis of water molecules or via shear forces induced by the formation and collapse of

cavitating bubbles. However, the frequency of the sonicator used in this study was fixed to 50 kHz and not able to apply lower-frequency ultrasound in the extraction process. In addition, the extraction solvent, sample volume and temperature might also affect the extraction efficiency of ultrasound. According to Cocito et al. (1995), dichloromethane was more effective than other solvents in the ultrasound-assisted volatiles extraction from must and wine. Considering the toxicity and health risks of dichloromethane, it is not a suitable extractant for our study. In contrast, the enzymatic hydrolysis displayed superior performance in this study, which supported that hydrolysis can be carried out under mild processing conditions with exquisite specificity and regioselectivity (Gardossi et al., 2010). However, it could take several hours for a complete substrate hydrolysis (Zhang et al., 2019) where the hydrolytic enzymes are expensive and readily deactivated by the hydrolysis products released (e.g. sugars) (Taherzadeh & Karimi, 2007). So far, there is only one comparable study on the proposed technique in aroma compound extraction. Agouillal, Moghrani, Nasrallah, Hanapi, Taher, and El-Enshasy (2018) investigated the efficiency of the coupled pretreatment of ultrasound and enzymatic hydrolysis before hydro-distillation in the essential oil extraction from Algerian *Artemisia herba-alba* Asso where 26 volatile compounds were identified. The whole process totally consumed 120 min for complete extraction, which is significantly faster than the extraction process in the current study. The deficiency is probably due to the different extracting materials. In contrast to several common extraction techniques, the proposed technique is cost-effective in aroma compounds extraction. For instance, the capital cost of the technique is far lower than that of supercritical fluid extraction which requires expensive high-pressure equipment (Meireles, 2008), while the operating cost is lower than the solvent extraction which has been considered as time- and solvent-consuming process (Dawidowicz, Rado, Wianowska, Mardarowicz, & Gawdzik, 2008).

3.3. Influences of enzymatic hydrolysis time on the volatile profile of pomace extract

To identify the optimum time for enzymatic extraction, 24 hr, 48 hr and 72 hr of hydrolysis were carried out (Table 1), and $T_{\text{en-48}}$ and $T_{\text{en-24}}$ extracted the maximum and minimum amounts of total volatiles, respectively. Several aroma compounds were found at significantly ($p \leq 0.05$) higher concentrations under $T_{\text{en-48}}$ than the other treatments, including 1-hexanol, (*E*)-2-hexen-1-ol, (*Z*)-2-hexen-1-ol, phenylacetaldehyde, benzyl alcohol and 2-phenylethanol. It is worth mentioning that nonadecane was only detected in $T_{\text{en-24}}$ extract, while alpha-terpineol and carvone were only observed under $T_{\text{en-48}}$. Compared to $T_{\text{en-48}}$, $T_{\text{en-72}}$ has shown lower total volatile concentration, except for 1-hexanol and (*E*)-2-octen-1-ol at relatively high concentrations. Regarding to the differences in OAVs, 10 odor-active compounds were observed in the extract from $T_{\text{en-48}}$, followed by 8 and 5 compounds in the case of $T_{\text{en-72}}$ and $T_{\text{en-24}}$, respectively (Table 3). Four odor-active compounds were detected in all three treatments, where geraniol, (*E*)-2-octen-1-ol and 1-octen-3-ol showed similar aroma intensity, while phenylacetaldehyde gave a stronger green note in $T_{\text{en-48}}$ extract. Higher OAVs of (*E*)-2-hexen-1-ol and (*Z*)-2-hexen-1-ol were also detected under $T_{\text{en-48}}$ compared to $T_{\text{en-72}}$, conferring fruity and green aromas (TGSC, 2019). Additionally, hexanal, carvone and 1-hexanol were only detected as odor-active compounds in $T_{\text{en-48}}$ extract, providing fruity, minty and herbal odors (TGSC, 2019). Overall, enzymatic hydrolysis of pomace samples for 48 hr resulted in the highest diversity and concentrations of aroma compounds, with higher OAVs in the final extract.

Previous researches have demonstrated that it takes time for enzymes, such as pectinase, α - and β -glycosidase to convert the glycosides into volatile compounds, and this process may depend on various parameters, such as temperature, duration, and pH. It is documented that the enzymatic hydrolysis of grape glycosides could be accomplished after 12 hr of incubation

at 40 °C and pH 5.0 (Gunata, Bayonove, Baumes, & Cordonnier, 1985), and 24 hr of hydrolysis time was also proved to be sufficient for β -glucosidase to break down banana glycosides (Pérez, Cert, Ríos, & Olías, 1997). The lower hydrolysis temperature (35 °C) and the different enzymes used in the current study may be responsible for the discrepancy of the hydrolysis results. The lower total volatile content detected after 72 hr of incubation was probably because that the aroma compounds were readily volatilized and lost with long hydrolysis time (Zhang et al., 2019). In addition, prolonged exposure to hydrolytic enzyme could result in the development of acidic compounds, which will in turn cause a drop in pH of the medium, thereby hampering the enzyme activity (Ruiz-Terán, Perez-Amador, & López-Munguía, 2001).

3.4. Influences of ethanol concentration on the volatile profile of pomace extract

Four different ethanol concentrations were tested to optimize the extraction of aroma compounds (Table 2), and T_{et-0%} was found to extract the maximum amount of total volatiles. When the ethanol concentration was increased from 0% to 50%, the volatile content significantly ($p \leq 0.05$) dropped, while a similar content was detected under T_{et-70%} compared to T_{et-0%}. T_{et-0%} accounted for the highest content of total alcohols, while the maximum amounts of aldehyde, esters, ketones, phenols and terpenes were observed in T_{et-70%} extract. 2-Methyl-1-propanol was only detected under T_{et-0%} and 3-methylbutan-1-ol, 1-butanol, 1-hexanol, 1-pentanol and benzyl alcohol were found at much higher concentrations compared to other treatment groups. In the extract from T_{et-70%}, (Z)-2-hexen-1-ol, 1-octen-3-ol, (E)-2-octen-1-ol, as well as hexanal and geranylacetone that were not present in T_{et-0%} extract, were detected at high concentrations. It is worth mentioning that nerol and nonadecane were only identified in the extracts from T_{et-30%} and T_{et-50%}, respectively, and hexanal was found as a minor volatile constituent in T_{et-30%} extract. Regarding to the OAVs, 13 odor-active

compounds were detected in T_{et-70%} extract (Table 4), and hexanal, (*E*)-2-octen-1-ol, phenylacetaldehyde and 1-octen-3-ol represented the characteristic aroma compounds with higher OAVs, accounting for fruity, green and earthy odors (TGSC, 2019). Nine, ten and five odor-active compounds were observed in the extracts from T_{et-0%}, T_{et-30%} and T_{et-50%}, respectively (Table 4). 3-Methylbutyl acetate was the odor-active compound unique to T_{et-0%} but with minimal sensorial impact. Based on the OAVs, (*E*)-2-hexen-1-ol, ethyl octanoate and 2-undecanone showed no significant differences in their contribution to overall aroma among T_{et-0%}, T_{et-30%} and T_{et-70%}, while 1-hexanol displayed similar aroma intensity between T_{et-0%} and T_{et-70%}. The above results indicated that 70% ethanol might be the optimum solvent concentration for aroma compounds extraction.

In the present study, the lower total volatile contents observed under T_{et-30%} and T_{et-50%} might be attributed to the inhibiting effect of ethanol (Pogorzelski & Wilkowska, 2007). It is well demonstrated that ethanol exhibits high capacity to extract phenolic compounds (Pinelo et al., 2006), and polyphenols (e.g. catechin) might inhibit the release of aromatic compounds due to their intermolecular interactions with volatile compounds (Dufour & Bayonove, 1999). On the other hand, the high extraction yield was observed under both T_{et-0%} and T_{et-70%}, while the ethanol form of T_{et-70%} extract might account for high stability (Khalid, Zhari, Amirin, & Pazilah, 2011), high antioxidative activity and high antimicrobial potential (Basile et al., 2005), which may expand its potential utilization in pharmaceutical and cosmetic industries (Rahman & Kang, 2009). Likewise, Genovese, Gambuti, Lamorte, and Moio (2013) reported that the solvent extraction using 0% ethanol can obtain higher number and amount of free and bound volatiles from Aglianico grape skin than using 6% and 12% ethanol, although 10-15% ethanol have been mostly used in the solvent extraction of aroma compounds from grape

skins with or without enzyme pre-treatment (Rodríguez-Bencomo, Cabrera-Valido, Pérez-Trujillo, & Cacho, 2011; Vázquez, Pérez-Coello, & Cabezudo, 2002).

3.5. Influences of distillation time on the volatile profile of pomace extract

Different distillation collection times were compared for volatile compounds in the final extracts (Table 2). In the 0% and 50% ethanol extracts, there was no significant ($p > 0.05$) difference in the concentrations of total volatile compounds between T_{t-20} and T_{t-40} . In the 0% ethanol extract, a few aroma compounds were detected at higher concentrations in the T_{t-20} sample, including 1-octen-3-ol, 1-nonanol and 2-methyl-1-propanol. In the 50% ethanol extracts, (*Z*)-2-hexen-1-ol, 1-octen-3-ol, nonadecane and ethyl benzoate were detected in larger amounts under T_{t-20} . In the 30% and 70% ethanol extracts, T_{t-20} accounted for higher levels of most aroma compounds compared to T_{t-40} , except for phenols and terpenes. In the 30% ethanol extract, most alcohols, aldehyde and esters had higher concentrations under T_{t-20} than T_{t-40} with a few exceptions, including (*Z*)-2-hexen-1-ol, ethyl octanoate, ethyl nonanoate and ethyl benzoate. In the 70% ethanol extract, there were no significant ($p > 0.05$) differences in the concentrations of (*E*)-2-hexen-1-ol, (*Z*)-2-hexen-1-ol and (*E*)-2-octen-1-ol between T_{t-20} and T_{t-40} , while benzaldehyde was the only phenol detected at higher concentration under T_{t-20} . In terms of the OAVs, more odor-active compounds were found in the extracts from T_{t-20} compared to T_{t-40} (Table 4). In the 0% and 50% ethanol extracts, no significant ($p > 0.05$) differences were detected in most odor-active compounds between the two distillation times, except for the higher OAVs of ethyl octanoate and 1-octen-3-ol detected under T_{t-20} in the 0% ethanol extract, conferring intense fruity and earthy aromas (TGSC, 2019). In the 30% and 70% ethanol samples, significantly ($p \leq 0.05$) higher OAVs of most odor-active compounds were observed under T_{t-20} than T_{t-40} . Other compounds, such as 2-undecanone, geraniol and phenylacetaldehyde had similar OAVs between T_{t-20} and T_{t-40} ,

accounting for fruity, floral and green aromas (TGSC, 2019). In the 70% ethanol extract, there were 13 and 11 odor-active compounds identified from T_{t-20} and T_{t-40} , respectively. Green aromas were the predominant odor group, where (*E*)-2-octen-1-ol accounted for the highest OAVs under both T_{t-20} and T_{t-40} , giving green, citrus and vegetable scents (TGSC, 2019). Therefore, 20 minutes of distillation was relatively more effective in recovery of aroma compounds.

In the current study, higher levels of alcohols, aldehyde and esters in the T_{t-20} samples might be resulted from their high volatilities and low boiling points. For instance, 3-methylbutan-1-ol, hexanal and 3-methylbutyl acetate have the relatively low boiling points of 131 to 133 °C, 130 to 131 °C and 142 to 143 °C respectively (TGSC, 2019), allowing them to be readily volatilized under reduced pressure. On the other hand, the large amounts of phenols and terpenes observed in the T_{t-40} samples may be due to the intermolecular hydrogen bonding of phenols (Norwitz, Nataro, & Keliher, 1986) and relatively high boiling points of terpenes. For instance, the high boiling points of geraniol and linalool, 229 to 230 °C and 194 to 197 °C (TGSC, 2019), might hinder their volatilization. The vacuum distillation used in this study has multiple advantages, including environmental friendliness, low operation costs and high efficiency (Chen, Xu, Yang, Liu, & Yang, 2017). However, the high temperature required in vacuum distillation (Guo et al., 2010) might decompose thermolabile materials that can be separated or purified by an alternative method - steam distillation (Karger, Snyder, & Horvath, 1973). Using this distillation technique, compounds responsible for fruity, floral and green aromas were collected together at both T_{t-20} and T_{t-40} , and future study should consider the possibility of employing other collection techniques to separately collect aroma compounds for these attributes in order to produce products meeting different requirements of the industry.

3.6 Recovery of volatile compounds from pomace extract

As discussed above, the optimal experimental conditions of the proposed extraction method are as follows: 48 hr of enzymatic hydrolysis, 70% ethanol/water and 20 min of distillation. In this background, the recovery of volatile compounds from Chardonnay grape pomace can reach 37.15 $\mu\text{g/mL}$ pomace extract (Table 2), which can be converted to 22.29 $\mu\text{g/g}$ pomace powder. Solvent extraction is the common method to isolate aroma volatiles from grape skins. In the study of Genovese et al. (2013), the solution at pH 3.2 and 0% ethanol can successfully extract 78 μg free volatile compounds and 83 μg bound volatile compounds from 1 kg of Aglianico grape skin, indicating a significantly lower aroma recovery compared to our method. Similarly, Vázquez, Pérez-Coello, and Cabezudo (2002) employed 10% ethanol as extractant for 15-day solvent extraction with the pre-treatment of glycosidase hydrolysis to isolate aroma compounds from Chardonnay grape skin. The concentration of extracted volatiles detected in macerated skin solution is 3779.2 $\mu\text{g/L}$, confirming the lower recovery and larger time consumption of the solvent extraction. In addition, pressurized liquid extraction is also reported with lower recovery (121.07 $\mu\text{g/kg}$) when extracting glycosidic aroma precursors from Verdejo grape pomace (Muñoz-González et al., 2014). However, the pressurized liquid extraction can be completed in less than one hour, which is considerably faster than our method. The freezing step in sample preparation may impacted the aroma profile of the extract product. For instance, in the study of Chen, Capone, Nicholson, and Jeffery (2019), pre-fermentation freezing treatment of grape berry can significantly boost the concentrations of both free and bound thiols through the possible mechanisms such as other thiol biogenesis pathways or derivation from known precursors. It is worth mentioning that the freezing treatment in this study lasted for one month, which is far longer than our treatment time. Therefore, the positive effect of freezing treatment on aroma extraction might not be pronounced in our study.

4. Conclusion

In this study, an enzyme-assisted extraction method was developed to extract aroma compounds from Chardonnay grape pomace under optimal experimental conditions (48 hr of enzymatic hydrolysis, 70% ethanol/water, 20 min of distillation). A total of 65 aromatic compounds including 15 odor-active compounds were detected in the samples. 40 hours of enzymatic hydrolysis was the optimal pre-treatment amongst the tested methods for higher concentrations of aroma compounds extracted from Chardonnay grape pomace, however the compounds mixture extracted gave fruity and green notes simultaneously. In terms of extraction method, 70% ethanol was the optimum solvent concentration for aroma compounds extraction and 20 minutes of distillation was more effective in recovery of aroma compounds. Considering the large amount of grape pomace produced every year worldwide, the present method offers an essential prerequisite for the future utilization of grape pomace to produce flavor additives for food, cosmetic and perfumery applications. The quantitative results also suggest the feasibility of this method in aroma compounds extraction from other plant materials. In the future study, multiple collection techniques and sensory evaluations could be employed to provide further perspectives for aroma/flavor additives production.

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6. Author Contributions

Zijian Liang and Ahalya Pai performed the experiments, interpreted the results, wrote, edited and proofread the manuscript. Zhongxiang Fang, Jiaqiang Luo, Di Liu and Jihong Wu contributed to the data interpretation and the editing and proofreading of the manuscript, and

provided critical feedback for revision. Pangzhen Zhang made a substantial contribution in the study concept and design, and contributed to the overall reviewing, editing and approval of the final submission.

7. Conflict of Interest

The authors declare no conflict of interest.

8. References

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Table 1 Effects of pre-treatment and enzymatic hydrolysis time on the volatile compounds in Chardonnay grape pomace extracts ($\mu\text{g/mL}$)

Peak No.	Compound	RI*	Pre-treatment					ID**	Odor description***
			Ultrasound	Enzyme			UI + En		
				Hydrolysis time (hr)					
				24	48	72			
4	2-Methyl-1-propanol*** *	1090	ND	ND	0.01±0.00	ND	ND	RI, MS	Ethereal, winey, cortex
7	1-Butanol	1143	0.84±0.08 ^a	0.81±0.00 ^A	0.96±0.10 ^{aA}	0.87±0.01 ^A	0.84±0.10 ^a	RI, MS, ES	Fusel, oil, sweet, balsam, whiskey
10	3-Methylbutan-1-ol	1206	tr	tr	0.15±0.38	tr	tr	RI, MS, ES	Fusel, oily, alcoholic, whiskey, fruity, banana
13	1-Pentanol*** *	1249	tr	tr	tr	tr	tr	RI, MS	Pungent, fermented, bready, yeasty, fusel, winey, solvent
16	2-Heptanol	1320	tr	tr	tr	tr	tr	RI, MS, ES	Fresh, lemongrass, herbal, sweet, floral, fruity, green
19	1-Hexanol	1354	4.21±0.72 ^a	1.58±1.10 ^B	9.25±4.20 ^{aA}	4.33±0.98 ^{AB}	4.70±2.61 ^a	RI, MS, ES	Pungent, ethereal, fusel, oily, fruity, alcoholic, sweet, green
22	(E)-2-Hexen-1-ol	1407	0.72±0.16 ^b	0.38±0.12 ^B	2.01±0.69 ^{aA}	0.99±0.19 ^{AB}	1.01±0.44 ^{ab}	RI, MS, ES	Fatty, green, fruity, vegetable, leafy, herbal
22	(Z)-2-Hexen-1-ol	1407	0.40±0.43 ^a	0.21±0.13 ^B	1.98±0.76 ^{aA}	0.47±0.48 ^{AB}	0.65±0.77 ^a	RI, MS, ES	Vegetable, fatty, fusel, alcoholic, whiskey
26	1-Octen-3-ol	1452	0.02±0.02 ^a	0.02±0.01 ^A	0.05±0.04 ^{aA}	0.03±0.01 ^A	0.03±0.02 ^a	RI, MS, ES	Earthy, green, oily, vegetable, fungal
27	1-Heptanol	14	tr	tr	0.01±0.	tr	tr	RI,	Musty, pungent, leafy, green, vegetable, fruit

		57			02			MS, ES	y, apple, banana
30	2-Ethyl-1-hexanol	14 94	tr	tr	tr	tr	tr	RI, MS, ES	Citrus, fresh, floral, oily, sweet
32	(S)-3-Ethyl-4-methylpentanol****	15 11	0.01±0.02 ^a	tr	0.03±0.03 ^{aa}	0.01±0.01 ^A	0.02±0.02 ^a	RI, MS	--
34	2-Nonanol	15 24	tr	ND	tr	tr	tr	RI, MS, ES	Waxy, green, creamy, citrus, orange, cheesy, fruity
37	1-Octanol	15 64	0.01±0.01 ^a	tr	0.01±0.01 ^{aa}	0.01±0.00 ^A	0.01±0.01 ^a	RI, MS, ES	Waxy, green, orange, aldehydic, rose, mushroom
40	(E)-2-Octen-1-ol	16 21	4.13±0.16 ^a	2.14±3.02 ^A	5.15±0.85 ^{aa}	4.46±0.19 ^A	4.75±0.65 ^a	RI, MS, ES	Green, citrus, vegetable, fatty
43	1-Nonanol	16 65	0.02±0.00 ^a	0.02±0.00 ^A	0.02±0.00 ^{aa}	0.02±0.00 ^A	0.02±0.00 ^a	RI, MS, ES	Fatty, floral, rose, orange, dusty, wet, oily
	Total alcohol		10.37±1.92^a	5.17±4.22^B	19.64±6.70^{aa}	11.19±1.24^{AB}	12.03±4.57^a		
58	Nonadecane****	19 00	ND	0.06±0.02	ND	ND	ND	RI, MS	Alkane
	Total alkane		ND	0.06±0.02	ND	ND	ND		
2	Hexanal	10 69	ND	ND	0.03±0.10	tr	tr	RI, MS, ES	Fresh, green, fatty, aldehydic, grass, leafy, fruity, sweaty
	Total aldehyde		ND	ND	0.03±0.10	tr	tr		
5	3-	11	0.02±0	ND	0.02±0	0.01±0	0.01±0	RI,	Sweet, banana, fruity, ripe estery

	Methylbutyl acetate	12	.01 ^a		00 ^{aA}	00 ^A	00 ^a	MS, ES	
8	Methyl hexanoate	1178	ND	ND	tr	tr	tr	RI, MS, ES	Fruity, pineapple, ether
12	Ethyl hexanoate	1226	tr	tr	tr	tr	tr	RI, MS, ES	Sweet, fruity, pineapple, waxy, green, banana
14	Hexyl acetate	1265	ND	ND	tr	tr	tr	RI, MS, ES	Fruity, green, apple, banana, sweet
25	Ethyl octanoate	1432	ND	ND	0.01±0.00 ^{aA}	0.01±0.00 ^A	0.01±0.00 ^a	RI, MS, ES	Fruity, wine, waxy, sweet, apricot, banana, brandy, pear
35	Ethyl nonanoate	1533	ND	ND	0.01±0.00 ^{aA}	0.01±0.00 ^A	0.01±0.00 ^a	RI, MS, ES	Waxy, cognac, estery, fruity, apple, banana, tropical, winey
42	Ethyl benzoate	1656	0.02±0.00 ^a	0.01±0.00 ^A	0.01±0.00 ^{aA}	0.01±0.00 ^A	0.01±0.00 ^a	RI, MS, ES	Camomile, flower, celery, fruit
44	Diethyl succinate	1676	tr	ND	0.02±0.01 ^A	0.01±0.00 ^A	tr	RI, MS, ES	Mild, fruity, cooked apple, ylang
52	Ethyl benzene acetate	1782	tr	ND	tr	tr	tr	RI, MS, ES	Floral, honey, rose, balsamic, dark chocolate, cocoa, anisic black licorice
	Total ester		0.03±0.00^a	0.01±0.00^B	0.06±0.02^{aA}	0.05±0.01^{AB}	0.04±0.01^a		
38	2-Undecanone ****	1595	0.01±0.00 ^a	0.01±0.00 ^A	tr	0.01±0.00 ^A	0.01±0.00 ^a	RI, MS	Waxy, fruity, ketonic, fatty, pineapple
56	Geranylacetone****	1851	ND	ND	ND	ND	ND	RI, MS	Green, fruity, waxy, rose, woody, magnolia, tropical
17	Sulcatone	1328	0.02±0.00 ^a	0.02±0.00 ^A	0.02±0.01 ^{aA}	0.02±0.00 ^A	0.02±0.00 ^a	RI, MS	Fruity, apple, musty, ketonic, creamy, cheesy, banana

								ES	
	Total ketone		0.03±0.00^a	0.03±0.00^A	0.02±0.01 ^{aA}	0.03±0.00^A	0.03±0.00^a		
31	Benzaldehyde	1507	0.07±0.03 ^a	0.01±0.01 ^A	0.19±0.16 ^{aA}	0.05±0.02 ^A	0.05±0.02 ^a	RI, MS, ES	Strong, sharp, sweet, bitter, almond, cherry
41	Phenylacetaldehyde	1630	0.23±0.06 ^b	0.08±0.04 ^B	0.87±0.40 ^{aA}	0.37±0.10 ^{AB}	0.28±0.07 ^{ab}	RI, MS	Honey, floral, rose, sweet, powdery, fermented, chocolate, earthy
57	Benzyl alcohol	1873	0.38±0.13 ^a	0.17±0.04 ^B	0.92±0.36 ^{aA}	0.53±0.07 ^{AB}	0.61±0.14 ^a	RI, MS, ES	Floral, rose, phenolic, balsamic
59	2-Phenylethanol	1909	0.29±0.08 ^b	0.16±0.04 ^B	0.76±0.26 ^{aA}	0.40±0.04 ^{AB}	0.43±0.08 ^{ab}	RI, MS, ES	Sweet, floral, fresh, bread, rose, honey
	Total phenol		0.98±0.24^b	0.42±0.05^B	2.74±1.12^{aA}	1.35±0.17^{AB}	1.37±0.14^{ab}		
36	Linalool	1550	0.01±0.00 ^a	ND	0.01±0.00 ^{aA}	0.01±0.00 ^A	0.01±0.00 ^a	RI, MS, ES	Citrus, floral, sweet, rose, woody, green, blueberry
39	β-Cyclocitral	1607	ND	ND	tr	tr	tr	RI, MS, ES	Tropical, saffron, herbal, clean, rose, sweet, tobacco, green, fruity
46	α-Terpineol	1695	ND	ND	0.13±0.00 ^a	ND	0.13±0.00 ^a	RI, MS, ES	Pine, terpenic, lilac, citrus, woody, floral
48	Carvone***	1721	ND	ND	0.14±0.02 ^a	ND	0.13±0.01 ^a	RI, MS	Minty, licorice
55	Nerol	1804	0.03±0.00 ^a	ND	0.03±0.01 ^{aA}	0.03±0.00 ^A	0.03±0.00 ^a	RI, MS, ES	Sweet, natural, neroli, citrus, magnolia
56	Geraniol	1851	0.21±0.00	0.21±0.00 ^A	0.22±0.01 ^{aA}	0.21±0.00 ^A	0.22±0.01 ^a	RI, MS, ES	Floral, sweet, rose, fruity, citronella, citrus
	Total		0.25±0.02	0.21±0.00	0.54±0.01	0.25±0.00	0.51±0.01		

	terpene		.01 ^b	0.00 ^B	10 ^{aA}	00 ^B	09 ^a		
11	2-Pentylfuran	12-24	tr	tr	tr	tr	tr	RI, MS, ES	Fruity, green, earthy, beany, vegetable
	Total furan		tr	tr	tr	tr	tr		
	Total		11.65± 1.76 ^b	5.90± 4.24 ^B	23.03± 7.60 ^{aA}	12.86±1 .26 ^{AB}	13.98± 4.58 ^{ab}		

* RI = retention index

** ID = Identification by comparison of mass spectra (MS) with in-house NIST library 11.0, MS and retention index (RI) with NIST Webbook (NIST, 2019), and MS and retention time with external standards (ES).

*** = Odor description were obtained from TGSC (2019)

**** = Semi-quantification: 2-Methyl-1-propanol, 1-Pentanol, (S)-3-Ethyl-4-methylpentanol, 2-Undecanone, Carvone, Geranylacetone and Nonadecane were semi-quantified using the calibration curves of 3-Methylbutyl acetate, 3-Methylbutan-1-ol, 2-Ethyl-1-hexanol, beta-Cyclocitral, alpha-Terpineol, Geraniol and 2-Phenylethanol, respectively.

ND = Not detected or value below the detection threshold.

tr = trace

-- = Not available

a-b = Values with different superscripts in the same row are significantly different ($P \leq 0.05$) (Pre-treatment)

A-B = Values with different superscripts in the same row are significantly different ($P \leq 0.05$) (Hydrolysis time)

Table 2 Effects of ethanol concentration and distillation time on the volatile compounds in Chardonnay grape pomace extracts (µg/mL)

Peak No.	Compound	RI	Ethanol concentration in the extraction solution (%)								ID	Odor description
			0		30		50		70			
			Distillation time (min)									
			20	40	20	40	20	40	20	40		
4	2-Methyl-1-propanol	1090	0.02±0.01 ^{a'}	0.01±0.00 ^{b'}	ND	ND	ND	ND	ND	ND	RI, MS	Ethereal, winey, cortex
7	1-Butanol	1143	1.72±0.32 ^{aa'}	1.25±0.45 ^{Aa}	1.16±0.13 ^{aba''}	0.86±0.03 ^{Ab''}	0.81±0.00 ^{baA'}	0.70±0.02 ^{AA}	1.29±0.05 ^{abA''}	0.90±0.12 ^{AB''}	RI, MS, ES	Fusel, oil, sweet, balsam
10	3-Methylbutan-1-ol	1206	5.32±1.84 ^{aa'}	1.86±2.18 ^{a'}	0.58±0.22 ^b	tr	tr	tr	0.86±0.18 ^b	tr	RI, MS, ES	Fusel, oily, alcoholic, whiskey
13	1-Pentanol	1249	0.30±0.32	tr	tr	tr	tr	tr	tr	tr	RI, MS	Pungent, fermented, bready
16	2-Heptanol	1320	0.04±0.01 ^a	tr	0.01±0.00 ^b	tr	tr	tr	0.04±0.01 ^{aA''}	0.01±0.01 ^{B''}	RI, MS, ES	Fresh, lemongrass, herbal
19	1-Hexanol	1354	23.26±5.93 ^{aaa'}	9.70±8.14 ^{Aa}	12.13±2.48 ^{ba''}	3.75±1.07 ^{Ab''}	1.58±1.10 ^{caA'}	0.62±0.46 ^{AA}	17.91±2.10 ^{abA''}	8.39±4.00 ^{AB''}	RI, MS, ES	Pungent, ethereal, fusel, oily
22	(E)-2-Hexen-1-ol	1407	2.03±0.45 ^{aa'}	1.08±0.60 ^{Aa}	1.54±0.40 ^{aaa''}	0.80±0.23 ^{Ab''}	0.38±0.12 ^{baA'}	0.30±0.08 ^{AA}	2.01±0.30 ^{aA''}	1.20±0.60 ^{AA''}	RI, MS, ES	Fatty, green, fruity, vegetable
22	(Z)-2-Hexen-1-ol	1407	0.10±0.08 ^{ba'}	0.02±0.13 ^{Ca}	0.32±0.54 ^{ba''}	0.24±0.57 ^{Ba''}	0.21±0.13 ^b	tr	1.99±0.31 ^{aA''}	0.80±0.98 ^{AA''}	RI, MS, ES	Vegetable, fatty, fusel
26	1-Octen-3-ol	1452	0.05±0.01 ^{bca'}	0.01±0.01 ^{Bb}	0.11±0.02 ^{ba''}	0.02±0.01 ^{Bb''}	0.02±0.01 ^c	tr	0.38±0.06 ^{aA''}	0.15±0.08 ^{AB''}	RI, MS, ES	Earthy, green, oily, vegetable
27	1-Heptanol	1457	0.05±0.01 ^a	tr	0.02±0.00 ^b	tr	tr	tr	0.04±0.01 ^{aA''}	0.01±0.01 ^{B''}	RI, MS,	Musty, pungent, leafy,

											ES	green
30	2-Ethyl-1-hexanol	1494	tr	tr	tr	tr	tr	tr	tr	tr	RI, MS, ES	Citrus, fresh, floral, oily
32	(S)-3-Ethyl-4-methylpentanol	1511	0.02±0.00 ^{ba}	tr	0.06±0.01 ^{ba}	0.01±0.01 ^{Ab}	tr	tr	0.12±0.02 ^{aA}	0.04±0.03 ^{AB}	RI, MS	--
34	2-Nonanol	1524	ND	ND	tr	tr	ND	ND	tr	tr	RI, MS, ES	Waxy, green, creamy, citrus
37	1-Octanol	1564	0.01±0.00 ^b	tr	0.02±0.00 ^b	tr	tr	tr	0.08±0.01 ^{aA}	0.03±0.02 ^B	RI, MS, ES	Waxy, green, orange
40	(E)-2-Octen-1-ol	1621	4.39±0.15 ^{ba}	4.13±0.26 ^{Aa}	5.59±0.39 ^{ba}	4.50±0.33 ^{Ab}	2.14±3.02 ^{ba}	3.99±0.10 ^{AA}	8.00±1.04 ^{aA}	5.91±1.29 ^{AA}	RI, MS, ES	Green, citrus, vegetable
43	1-Nonanol	1665	0.02±0.00 ^b	ND	0.02±0.00 ^{ba}	0.02±0.00 ^{Ab}	0.02±0.00 ^{ba}	0.02±0.00 ^{AA}	0.04±0.00 ^{aA}	0.02±0.00 ^{AB}	RI, MS, ES	Fatty, floral, rose, orange, oily
	Total alcohol		37.34±8.38 ^{aa}	18.07±11.68 ^{Aa}	21.58±2.95 ^{ba}	10.19±2.19 ^{Ab}	5.17±4.22 ^{ca}	5.62±1.02 ^{AA}	32.75±3.60 ^{abA}	17.46±7.03 ^{AB}		
58	Nonadecane	1900	ND	ND	ND	ND	0.06±0.02	ND	ND	ND	RI, MS	Alkane
	Total alkane		ND	ND	ND	ND	0.06±0.02	ND	ND	ND		
2	Hexanal	1069	ND	ND	0.33±0.19 ^b	tr	ND	ND	1.78±0.55 ^{aA}	0.24±0.15 ^B	RI, MS, ES	Fresh, green, fatty, aldehydic
	Total aldehyde		ND	ND	0.33±0.19 ^b	tr	ND	ND	1.78±0.55 ^{aA}	0.24±0.15 ^B		
5	3-Methylbuty	1112	0.05±0.01 ^{aa}	0.05±0.02 ^{Aa}	0.03±0.01 ^{aa}	0.01±0.00 ^{Bb}	ND	ND	0.02±0.00 ^{aA}	0.02±0.00 ^{BA}	RI, MS	Sweet, banana, fruity

	l acetate										ES	
8	Methyl hexanoate	1178	ND	ND	tr	tr	ND	ND	0.01±0.00	tr	RI, MS, ES	Fruity, pineapple, ether
12	Ethyl hexanoate	1226	tr	ND	0.01±0.01 ^b	tr	tr	ND	0.09±0.03 ^{aA}	0.01±0.01 ^B	RI, MS, ES	Sweet, fruity, pineapple
14	Hexyl acetate	1265	ND	ND	tr	tr	ND	ND	tr	tr	RI, MS, ES	Fruity, green, apple, banana
25	Ethyl octanoate	1432	0.01±0.00 ^a	ND	0.01±0.00 ^{aa}	0.01±0.00 ^{Aa}	ND	ND	0.01±0.00 ^{aA}	0.01±0.00 ^{AA}	RI, MS, ES	Fruity, wine, waxy, sweet
35	Ethyl nonanoate	1533	ND	ND	0.01±0.00 ^{aa}	0.01±0.00 ^{Aa}	ND	ND	0.01±0.00 ^{aA}	0.01±0.00 ^{AA}	RI, MS, ES	Waxy, cognac, estery, fruity
42	Ethyl benzoate	1656	0.01±0.00 ^b	ND	0.02±0.00 ^{ba}	0.01±0.00 ^{Aa}	0.01±0.00 ^b	ND	0.03±0.00 ^{aA}	0.02±0.00 ^{AB}	RI, MS, ES	Camomile, flower, celery
44	Diethyl succinate	1676	ND	ND	ND	tr	ND	tr	ND	tr	RI, MS, ES	Mild, fruity, cooked apple
52	Ethyl benzene acetate	1782	ND	ND	tr	tr	ND	ND	tr	tr	RI, MS, ES	Floral, honey, rose, balsamic
	Total ester		0.07±0.01 ^{ba}	0.05±0.02 ^{Aa}	0.07±0.01 ^{ba}	0.04±0.00 ^{Bb}	0.01±0.00 ^b	tr	0.17±0.04 ^{aA}	0.06±0.01 ^{ABB}		
38	2-Undecanone	1595	0.01±0.00 ^{aa}	0.01±0.00 ^{Aa}	0.01±0.00 ^{aa}	0.01±0.00 ^{Aa}	0.01±0.00 ^{aA}	0.01±0.00 ^{AA}	0.01±0.00 ^{aA}	0.01±0.00 ^{AA}	RI, MS	Waxy, fruity, ke tonic, fatty
56	Geranylacetone	1851	ND	ND	ND	ND	ND	ND	0.26±0.01	ND	RI, MS	Green, fruity, waxy, rose
17	Sulcatone	1328	0.03±0.00 ^{bca}	0.02±0.00 ^A	0.03±0.00 ^{ba}	0.02±0.00 ^{Bb}	0.02±0.00 ^{cA}	0.02±0.00 ^{BA}	0.07±0.01 ^{aA}	0.04±0.01 ^{AB}	RI, MS	Fruity, apple, musty, ketonic

				Ba'							ES	
	Total ketone		0.04±0.00^{ba'}	0.03±0.00^{Aa}	0.04±0.00^{ba''}	0.03±0.00^{Aa''}	0.03±0.00^{ba}	0.03±0.00^{AA}	0.34±0.02^{aA''}	0.05±0.01^{AB''}		
31	Benzaldehyde	1507	0.01±0.01 ^c	tr	0.14±0.02 ^{ba''}	0.06±0.03 ^{Ab''}	0.01±0.01 ^c	tr	0.23±0.04 ^{aA''}	0.11±0.05 ^{AB''}	RI, MS, ES	Strong, sharp, sweet, bitter
41	Phenylacetaldehyde	1630	0.07±0.05 ^{ba'}	0.05±0.05 ^{Ba}	0.43±0.29 ^{aba''}	0.33±0.01 ^{ABa''}	0.08±0.04 ^{ba'}	0.13±0.01 ^{BA'}	0.54±0.15 ^{aA''}	0.84±0.41 ^{AA''}	RI, MS	Honey, floral, rose, sweet
57	Benzyl alcohol	1873	0.69±0.16 ^{aa'}	0.87±0.25 ^{Aa}	0.44±0.28 ^{aba''}	0.40±0.10 ^{BCa''}	0.17±0.04 ^{ba'}	0.18±0.02 ^{CA'}	0.56±0.14 ^{abA''}	0.69±0.08 ^{ABA''}	RI, MS, ES	Floral, rose, phenolic
59	2-Phenylethanol	1909	0.53±0.09 ^{aa'}	0.68±0.18 ^{Aa}	0.34±0.21 ^{aa''}	0.29±0.07 ^{Ba''}	0.16±0.04 ^{aA'}	0.15±0.01 ^{BA'}	0.52±0.14 ^{aA''}	0.62±0.07 ^{AA''}	RI, MS, ES	Sweet, floral, fresh, bread
	Total phenol		1.29±0.23^{aba'}	1.60±0.47^{Ba}	1.34±0.77^{aba''}	1.09±0.18^{ABa''}	0.42±0.05^{ba}	0.46±0.02^{BA}	1.84±0.40^{aA''}	2.25±0.53^{AA''}		
36	Linalool	1550	0.01±0.00 ^a	ND	0.01±0.00 ^{aa''}	0.01±0.00 ^{Aa''}	ND	ND	0.01±0.00 ^{aA''}	0.01±0.00 ^{AA''}	RI, MS, ES	Citrus, floral, sweet, rose
39	β-Cyclocitral	1607	ND	ND	ND	ND	ND	ND	0.01±0.00 ^{A''}	0.01±0.00 ^{A''}	RI, MS, ES	Tropical, saffron, herbal
46	α-Terpineol	1695	ND	ND	ND	ND	ND	ND	ND	ND	RI, MS, ES	Pine, terpenic, lilac, citrus
48	Carvone	1721	ND	ND	ND	ND	ND	ND	ND	ND	RI, MS	Minty, licorice
55	Nerol	1804	ND	ND	0.03±0.00 ^{a''}	0.03±0.00 ^{Aa''}	ND	ND	ND	0.03±0.00 ^A	RI, MS, ES	Sweet, natural, neroli, citrus
56	Geraniol	18	0.21±0	0.21±	0.22±0.0	0.21±0.0	0.21±	0.21±	0.24±0.0	0.23±0.0	RI	Floral, sweet, rose

		51	.00 ^{ba'}	0.00 ^{Aa}	0 ^{ba''}	00 ^{Aa''}	0.00 ^{ba'}	0.00 ^{AA}	01 ^{aA'}	01 ^{AA'}	MS, ES	se, fruity
	Total terpene		0.22±0.00 ^{ba'}	0.21±0.00 ^{Aa}	0.25±0.01 ^{aa''}	0.25±0.02 ^{Aa''}	0.21±0.00 ^{ba}	0.21±0.00 ^{AA}	0.26±0.01 ^{aA''}	0.27±0.03 ^{AA''}		
11	2- Pentylfuran	12 24	tr	ND	tr	tr	tr	tr	tr	tr	RI, MS, ES	Fruity, green, ea rthy, beany
	Total furan		tr	ND	tr	tr	tr	tr	tr	tr		
	Total		38.95±8.61 ^{aa'}	19.96±13.20 ^{Aa'}	23.61±3.81 ^{ba''}	11.59±2.25 ^{Ab''}	5.90±4.24 ^{ca'}	6.31±1.01 ^{AA}	37.15±3.94 ^{abA''}	20.32±7.75 ^{AB''}		

a-c = Values with different superscripts in the same row are significantly different ($P \leq 0.05$)

(20 min distillation time)

A-B = Values with different superscripts in the same row are significantly different ($P \leq 0.05$)

(40 min distillation time)

a'-b'= Values with different superscripts in the same row are significantly different ($P \leq 0.05$)

(0% ethanol concentration)

a''-b''= Values with different superscripts in the same row are significantly different ($P \leq 0.05$) (30% ethanol concentration)

A'-B'= Values with different superscripts in the same row are significantly different ($P \leq 0.05$) (50% ethanol concentration)

A''-B''= Values with different superscripts in the same row are significantly different ($P \leq 0.05$) (70% ethanol concentration)

Table 3 Effects of pre-treatment and enzymatic hydrolysis time on odor active values of the volatile compounds in Chardonnay grape pomace extracts

Peak No.	Compound	Odor descriptor*	Odor threshold (µg/L)	Reference	Pre-treatment				
					Ultrasound	Enzyme			Ul + En
						Hydrolysis time (hr)			
						24	48	72	
	Fruity								
22	(E)-2-Hexen-1-ol**	Green grass, herb	400	(Jiang & Zhang, 2010)	1.806±0.401 ^b	0.951±0.297 ^B	5.032±1.717 ^{aA}	2.474±0.479 ^{AB}	2.526±1.095 ^{ab}
2	Hexanal**	Green, cut grass	4.5	(Guth, 1997)	--	--	6.473±22.894	<0.001	<0.001
5	3-Methylbutyl acetate	Sweet, banana, estery	30	(Guth, 1997)	0.544±0.187 ^a	--	0.505±0.052 ^{aA}	0.472±0.032 ^A	0.423±0.061 ^a
8	Methyl hexanoate	Fresh (Pome)	70	(Li et al., 2008)	--	--	<0.001	<0.001	<0.001
12	Ethyl hexanoate	Apple peel, fruit (Pome)	5	(Li et al., 2008)	<0.001	<0.001	<0.001	<0.001	<0.001
14	Hexyl acetate	Pleasant, fruity, Pear	1500	(Li et al., 2008)	--	--	<0.001	<0.001	<0.001
25	Ethyl octanoate**	Apricot, banana, pear	2	(Guth, 1997)	2.620±0.029 ^a	--	2.600±0.000 ^{aA}	1.747±1.513 ^A	2.600±0.000 ^a
44	Diethyl succinate	Light fruity	200000	(Guth, 1997)	<0.001	--	<0.001	<0.001	<0.001
38	2-Undecanone**	Tallow, musty	5.5	(Giri et al., 2010)	1.195±0.129 ^a	1.781±0.199 ^A	0.563±0.899 ^{bB}	1.297±0.022 ^{AB}	1.115±0.055 ^a
31	Benzaldehyde	Almond, cherry	2000	(Dragone et al., 2009)	0.037±0.013 ^a	0.005±0.006 ^A	0.095±0.079 ^{aA}	0.027±0.012 ^A	0.023±0.012 ^a
11	2-Pentylfuran	Green bean like	5.8	(Giri et al., 2010)	<0.001	<0.001	<0.001	<0.001	<0.001
	Floral								
43	1-Nonanol	Rose, orange, d	50	(Javed et al.,	0.359±0.	0.338±0.	0.350±0.0	0.349±0.	0.358±0.

		usty		2018)	031 ^a	006 ^A	25 ^{AA}	011 ^A	027 ^a
52	Ethyl benzene acetate	Honey, rose, balsamic	650	(Dragone et al., 2009)	0.001±0.001 ^a	--	0.002±0.001 ^{AA}	0.001±0.000 ^A	0.001±0.000 ^a
56	Geranylacetone	Rose, woody, magnolia	60	(Javed et al., 2018)	--	--	--	--	--
57	Benzyl alcohol	Rose, phenolic, balsamic	200000	(Jiang & Zhang, 2010)	0.002±0.001 ^a	0.001±0.000 ^B	0.005±0.002 ^{AA}	0.003±0.000 ^{AB}	0.003±0.001 ^a
59	2-Phenylethanol	Bready, rose, honey	1104	(Guth, 1997)	0.265±0.071 ^b	0.146±0.038 ^B	0.686±0.240 ^{AA}	0.360±0.040 ^{AB}	0.389±0.076 ^{ab}
56	Geraniol**	Sweet, rose, citronella	40	(Javed et al., 2018)	5.356±0.172 ^a	5.174±0.010 ^A	5.567±0.244 ^{AA}	5.346±0.036 ^A	5.554±0.091 ^a
	Fermented								
7	1-Butanol	Fusel, balsam, whiskey	150000	(Jiang & Zhang, 2010)	0.006±0.001 ^a	0.005±0.000 ^A	0.006±0.001 ^{AA}	0.006±0.000 ^A	0.006±0.001 ^a
10	3-Methylbutanol-1-ol	Fusel, whiskey, banana	30000	(Guth, 1997)	<0.001	<0.001	0.005±0.013	<0.001	<0.001
13	1-Pentanol	Bready, yeasty, fusel	64000	(Zea et al., 2001)	<0.001	<0.001	<0.001	<0.001	<0.001
	Waxy								
4	2-Methyl-1-propanol	Ethereal, winey, cortex	65000	(Dragone et al., 2009)	--	--	<0.001	--	--
34	2-Nonanol	Creamy, orange, cheesy	58	(Javed et al., 2018)	0.032±0.002 ^a	--	0.028±0.000 ^{AA}	0.029±0.003 ^A	0.034±0.000 ^a
37	1-Octanol	Aldehydic, rose, mushroom	900	(Li et al., 2008)	0.008±0.007 ^a	0.004±0.002 ^B	0.009±0.010 ^{AA}	0.008±0.003 ^A	0.013±0.010 ^a
35	Ethyl nonanoate	Cognac, estery, fruity	1300	(Li et al., 2008)	--	--	0.006±0.000 ^{AA}	0.006±0.000 ^A	0.006±0.000 ^a
	Citrus								
16	2-Heptanol	Lemongrass, herbal, sweet	70	(Jiang & Zhang, 2010)	<0.001	<0.001	0.036±0.194	<0.001	<0.001
30	2-Ethyl-1-hexanol	Citrus, fresh, floral	8000	(Jiang & Zhang, 2010)	<0.001	<0.001	<0.001	<0.001	<0.001
17	Sulcatone	Apple, musty, k	50	(Javed et al.,	0.421±0.	0.387±0.	0.428±0.1	0.417±0.	0.406±0.

		etonic		2018)	046 ^a	022 ^A	11 ^{aA}	020 ^A	023 ^a
36	Linalool	Citrus, floral, blueberry	25	(Guth, 1997)	0.237±0. 000 ^a	--	0.254±0.0 87 ^{aA}	0.262±0. 000 ^A	0.254±0. 061 ^a
46	α-Terpineol	Pine, terpenic, li lac	250	(Li et al., 2008)	--	--	0.535±0.0 00 ^a	--	0.506±0. 003 ^a
55	Nerol	Neroli, citrus, magnolia	300	(Javed et al., 2018)	0.087±0. 000 ^a	--	0.109±0.0 17 ^{aA}	0.092±0. 003 ^A	0.099±0. 015 ^a
	Green								
22	(Z)-2- Hexen-1- ol**	Vegetable, fatty, fusel,	400	(Jiang & Zhang, 2010)	1.012±1. 082 ^a	0.534±0. 331 ^B	4.960±1.9 07 ^{aA}	1.187±1. 192 ^{AB}	1.619±1. 915 ^a
27	1-Heptanol	Musty, pungent, leafy, green	1000	(Jiang & Zhang, 2010)	<0.001	<0.001	0.013±0.0 18	<0.001	<0.001
40	(E)-2-Octen- 1-ol**	Green, citrus, ve getable	18	(Javed et al., 2018)	221.923 ±8.540 ^a	79.400±1 37.290 ^A	286.271±4 7.006 ^{aA}	248.022± 10.455 ^A	263.801± 36.144 ^a
41	Phenylacetal dehyde**	Honey, floral, e arthy	4	(Javed et al., 2018)	57.204± 15.345 ^b	19.210±1 0.495 ^B	218.486±1 00.071 ^{aA}	91.380±2 4.661 ^{AB}	70.639±1 8.003 ^{ab}
	Minty								
42	Ethyl benzoate	Camomile, flower, celery	500	(Zea et al., 2001)	0.031±0. 002 ^a	0.029±0. 000 ^A	0.030±0.0 02 ^{aA}	0.030±0. 001 ^A	0.030±0. 001 ^a
48	Carvone**	Minty, licorice	2.7	(Ahmed et al., 1978)	--	--	52.536±5. 781 ^a	--	32.664±2 8.365 ^a
	Herbal								
19	1- Hexanol**	Ethereal, fusel, oily	8000	(Li et al., 2008)	0.526±0. 340 ^a	0.198±0. 137 ^B	1.156±0.5 25 ^{aA}	0.541±0. 123 ^{AB}	0.588±0. 327 ^a
	Earthy								
26	1-Octen-3- ol**	Earthy, green, o ily	1	(Javed et al., 2018)	22.918± 15.743 ^a	15.834±1 3.433 ^A	47.306±40 .39 ^{aA}	27.139±8 .295 ^A	29.778±2 2.092 ^a
	Tropical								
39	β- Cyclocitral	Tropical, saffro n, herbal	19	(Young & Suffet, 1999)	--	--	0.221±0.0 00 ^{aA}	0.221±0. 000 ^A	0.221±0. 000 ^a
	Others								
32	(S)-3-Ethyl- 4- methylpenta nol	--	--	--	--	--	--	--	--

58	Nonadecane	Alkane	--	--	--	--	--	--	--
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* = Odor descriptor were obtained from TGSC (2019)

** = Odor-active compound (OAV >1 under at least one of the treatments)

a-b = Values with different superscripts in the same row are significantly different ($P \leq 0.05$)

(Pre-treatment)

A-B = Values with different superscripts in the same row are significantly different ($P \leq 0.05$)

(Hydrolysis time)

Table 4 Effects of ethanol concentration and distillation time on odor active values of the volatile compounds in Chardonnay grape pomace extracts

P e a k N o.	Compo und	Odor descriptor	Odor threshold ($\mu\text{g/L}$)	Referenc e	Ethanol concentration in the extraction solution (%)							
					0		30		50		70	
					Distillation time (min)							
					20	40	20	40	20	40	20	40
	Fruity											
22	(E)-2-Hexen-1-ol*	Green grass, herb	400	(Jiang & Zhang, 2010)	5.063 $\pm$ 1.116 ^{aa}	2.701 $\pm$ 1.504 ^{Aa}	3.860 $\pm$ 0.988 ^{aa}	1.991 $\pm$ 0.571 ^{Ab}	0.951 $\pm$ 0.297 ^{bA}	0.549 $\pm$ 0.751 ^{AA}	5.015 $\pm$ 0.741 ^{aA}	2.995 $\pm$ 1.497 ^A
2	Hexanal*	Green, cut grass	4.5	(Guth, 1997)	--	--	73.80 $\pm$ 7 $\pm$ 43.284 ^b	<0.001	--	--	396.51 $\pm$ 1 $\pm$ 121.705 ^{aA}	52.692 $\pm$ 33.460 ^B
5	3-Methylbutyl acetate*	Sweet, banana, estery	30	(Guth, 1997)	1.551 $\pm$ 0.479 ^{aa}	1.738 $\pm$ 0.592 ^{Aa}	0.861 $\pm$ 0.403 ^{aa}	0.468 $\pm$ 0.073 ^{Ba}	--	--	0.720 $\pm$ 0.100 ^{aA}	0.579 $\pm$ 0.130 ^B
8	Methyl hexanoate	Fresh (Pome)	70	(Li et al., 2008)	--	--	<0.001	<0.001	--	--	0.124 $\pm$ 0.052 ^A	0.001 $\pm$ 0.012 ^B
1	Ethyl	Apple peel,	5	(Li et al.,	<0.00	--	2.506	<0.00	<0.00	--	17.575	2.846 $\pm$

2	hexanoat e*	fruit (Pome)		2008)	1		±1.60 3 ^b	1	1		±6.75 7 ^{aA}	1.187 ^B "
1 4	Hexyl acetate	Pleasant, fruity, pear	1500	(Li et al., 2008)	--	--	<0.00 1	<0.00 1	--	--	0.003 ±0.00 1	<0.001
2 5	Ethyl octanoat e*	Apricot, banana, pear	2	(Guth, 1997)	2.664± 0.018 ^a	--	2.775 ±0.03 6 ^{aa}	2.616 ±0.02 2 ^{Aa}	--	--	3.522 ±0.34 3 ^{aA}	2.654± 0.076 ^A B ["]
4 4	Diethyl succinat e	Light fruity	200000	(Guth, 1997)	--	--	--	<0.00 1	--	<0.001	--	<0.001
3 8	2- Undecan one*	Fallow, musty	5.5	(Giri et al., 2010)	2.482± 0.507 ^{aa} '	1.979 ±0.34 7 ^{Aa}	1.905 ±0.70 5 ^{aa}	1.377 ±0.10 8 ^{Aa}	1.781 ±0.19 9 ^{aA}	1.571±1 .552 ^{AA}	2.394 ±0.26 3 ^{aA}	1.746± 0.278 ^A A ["]
3 1	Benzald ehyde	Almond, cherry	2000	(Dragone et al., 2009)	0.005± 0.003 ^{ca} '	0.001 ±0.00 3 ^{Ba}	0.068 ±0.01 0 ^{ba}	0.030 ±0.01 6 ^{Ab}	0.005 ±0.00 6 ^c	<0.001	0.114 ±0.01 9 ^{aA}	0.053± 0.027 ^A B ["]
1 1	2- Pentylfu ran	Green bean like	5.8	(Giri et al., 2010)	<0.00 1	--	<0.00 1	<0.00 1	<0.00 1	<0.001	<0.00 1	<0.001
	Floral											
4 3	1- Nonanol	Rose, oran ge, dusty	50	(Javed et al., 2018)	0.341± 0.001 ^b	--	0.403 ±0.01 6 ^{ba}	0.340 ±0.00 6 ^{Ab}	0.338 ±0.00 6 ^b	<0.001	0.732 ±0.06 4 ^{aA}	0.440± 0.072 ^A B ["]
5 2	Ethyl benzene acetate	Honey, ros e, balsamic	650	(Dragone et al., 2009)	--	--	0.001 ±0.00 0 ^a	<0.00 1	--	--	0.004 ±0.00 0 ^{aA}	0.001± 0.001 ^B '
5 6	Geranyl acetone*	Rose, wood y, magnolia	60	(Javed et al., 2018)	--	--	--	--	--	--	4.349 ±0.15 3	--
5 7	Benzyl alcohol	Rose, phenolic, balsamic	200000	(Jiang & Zhang, 2010)	0.003± 0.001 ^{aa} '	0.004 ±0.00 1 ^{Aa}	0.002 ±0.00 1 ^{aba}	0.002 ±0.00 0 ^{BCa}	0.001 ±0.00 0 ^{ba}	0.001±0 .001 ^{CA}	0.003 ±0.00 1 ^{abA}	0.003± 0.000 ^A BA ["]
5 9	2- Phenylet hanol	Bready, ros e, honey	1104	(Guth, 1997)	0.479± 0.078 ^{aa} '	0.612 ±0.15 9 ^{Aa}	0.304 ±0.19 2 ^{aa}	0.267 ±0.06 6 ^{Ba}	0.146 ±0.03 8 ^{aA}	0.142±0 .126 ^{BA}	0.467 ±0.12 7 ^{aA}	0.558± 0.063 ^A A ["]

5	Geraniol	Sweet, rose	40	(Javed et	5.239±	5.236	5.392	5.210	5.174	5.168±5	6.035	5.632±
6	*	, citronella		al., 2018)	0.026 ^b	±0.08	±0.11	±0.09	±0.01	.164 ^{AA'}	±0.17	0.319 ^A
					^{a'}	^{5Aa'}	^{4ba''}	^{1Aa''}	^{0ba'}		^{6aA''}	^{A''}
	Fermen											
	ted											
7	1- Butanol	Fusel, balsam, whiskey	150000	(Jiang & Zhang, 2010)	0.011± 0.002 ^{aa}	0.008 ±0.00	0.008 ±0.00	0.006 ±0.00	0.005 ±0.00	0.005±0 .000 ^{AA'}	0.009 ±0.00	0.006± 0.001 ^A
						^{3Aa'}	^{1aba''}	^{0Ab''}	^{0ba'}		^{0aba''}	^{B''}
1	3- Methylb utan-1- ol	Fusel, whiskey, banana	30000	(Guth, 1997)	0.177± 0.061 ^{aa}	0.062 ±0.07	0.019 ±0.00	<0.00 1	<0.00 1	<0.001	0.029 ±0.00	<0.001
						^{3a'}	^{7b}				^{6b}	
1	1- Pentanol	Bready, ye ast, fusel	64000	(Zea et al., 2001)	0.005± 0.005	<0.00 1	<0.00 1	<0.00 1	<0.00 1	<0.001	<0.00 1	<0.001
	Waxy											
4	2- Methyl- 1- propanol	Ethereal, winey, cortex	65000	(Dragone et al., 2009)	<0.00 1	<0.00 1	--	--	--	--	--	--
3	2- Nonanol	Creamy, orange, cheesy	58	(Javed et al., 2018)	<0.00 1	--	0.037 ±0.00	0.028 ±0.00	--	--	0.062 ±0.00	0.039± 0.006 ^A
							^{2ba''}	^{0Bb''}			^{5aA''}	^{B''}
3	1- Octanol	Aldehydic, rose, mushroom	900	(Li et al., 2008)	0.009± 0.001 ^b	0.002 ±0.00	0.024 ±0.00	0.005 ±0.00	0.004 ±0.00	<0.001	0.088 ±0.01	0.013± 0.019 ^A
					^{ca'}	^{0Bb'}	^{5ba''}	^{2ABb''}	^{2c}		^{5aA''}	^{B''}
3	Ethyl nonanoa te	Cognac, estery, fruity	1300	(Li et al., 2008)	--	--	0.006 ±0.00	0.006 ±0.00	--	--	0.006 ±0.00	0.006± 0.000 ^A
							^{0aa''}	^{0Aa''}			^{0aA''}	^{A''}
	Citrus											
1	2- Heptano l	Lemongras s, herbal, sweet	70	(Jiang & Zhang, 2010)	0.531± 0.134 ^a	<0.00 1	0.213 ±0.02	<0.00 1	<0.00 1	<0.001	0.640 ±0.09	0.103± 0.172 ^{B'}
							^{6b}				^{0aA''}	
3	2-Ethyl- 1- hexanol	Citrus, fresh, floral	8000	(Jiang & Zhang, 2010)	<0.00 1	<0.00 1	<0.00 1	<0.00 1	<0.00 1	<0.001	0.001 ±0.00	<0.001

17	Sulcaton e*	Apple, musky, ketonic	50	(Javed et al., 2018)	0.562±0.037 ^{bc}	0.394±0.00 ^{0ABb}	0.652±0.06 ^{4ba}	0.388±0.01 ^{1Bb}	0.387±0.02 ^{2cA}	0.351±0.000 ^{BA}	1.353±0.12 ^{6aA}	0.731±0.130 ^B
36	Linalool	Citrus, floral, blueberry	25	(Guth, 1997)	0.247±0.015 ^a	--	0.311±0.02 ^{0aa}	0.233±0.00 ^{0Ab}	--	--	0.597±0.05 ^{5aA}	0.324±0.079 ^A
46	α-Terpinol	Pine, terpene, lilac	250	(Li et al., 2008)	--	--	--	--	--	--	--	--
55	Nerol	Neroli, citrus, magnolia	300	(Javed et al., 2018)	--	--	0.097±0.00 ^{7a}	0.093±0.00 ^{0Aa}	--	--	--	0.108±0.016 ^A
Green												
22	(Z)-2-Hexen-1-ol*	Vegetable, fatty, fusel	400	(Jiang & Zhang, 2010)	0.239±0.192 ^{ba}	0.059±0.33 ^{0Ca}	0.806±1.36 ^{1ba}	0.611±1.42 ^{2Ba}	0.534±0.33 ^{1b}	<0.001	4.969±0.78 ^{7aA}	2.004±2.452 ^A
27	1-Heptanol	Musty, pungent, leafy, green	1000	(Jiang & Zhang, 2010)	0.055±0.010 ^{aa}	0.003±0.01 ^{8Ab}	0.020±0.00 ^{5b}	<0.001	<0.001	<0.001	0.040±0.00 ^{8aA}	0.008±0.013 ^B
40	(E)-2-Octen-1-ol*	Green, citrus, vegetable	18	(Javed et al., 2018)	244.16±8.465 ^{ba}	229.43±14.4 ^{52Aa}	310.7±21.5 ^{23aba}	249.76±18.5 ^{74Ab}	79.40±137 ^{290bA}	221.697±5.795 ^A	444.23±57.9 ^{28aA}	328.33±71.76 ^{3Aa}
41	Phenylacetaldehyde*	Honey, floral, earthy	4	(Javed et al., 2018)	16.347±11.39 ^{0ba}	12.945±13.7 ^{16Ba}	107.4±69 ^{±72.7}	83.720±3.54 ^{0ABa}	19.21±10.4 ^{95bA}	30.743±32.992 ^{BA}	135.82±37.4 ^{03aA}	208.89±101.4 ^{47AA}
Minty												
42	Ethyl benzoate	Camomile, flower, celery	500	(Zea et al., 2001)	0.028±0.000 ^b	--	0.033±0.00 ^{1ba}	0.028±0.00 ^{0Aa}	0.029±0.00 ^{0b}	--	0.064±0.00 ^{5aA}	0.037±0.005 ^B
48	Carvone	Minty, licorice	2.7	(Ahmed et al., 1978)	--	--	--	--	--	--	--	--
Herbal												
1	1-	Ethereal,	8000	(Li et al.,	2.907±	1.213	1.517	0.469	0.198	0.016±0	2.239	1.049±

9	Hexanol	fusel, oily		2008)	0.741 ^{aa}	±1.01	±0.30	±0.13	±0.13	.085 ^{AA}	±0.26	0.500 ^A
	*					7 ^{Aa'}	9 ^{ba''}	3 ^{Ab''}	7 ^{cA'}		2 ^{abA''}	B''
	Earthy											
2	1-Octen-	Earthy, gre	1	(Javed et	53.116	10.185	113.8	19.012	15.83	3.633±3	375.43	151.48
6	3-ol*	en, oily		al., 2018)	±5.446	±7.86	13	±7.84 ^B	4	.332 ^{BA'}	8	±77.26
					bca'	1 ^{Bb'}	±21.0	b''	±13.4		±62.5	6 ^{AB''}
							14 ^{ba''}		33 ^{cA'}		52 ^{aA''}	
	Tropical											
3	β-	Tropical,	19	(Young &	--	--	--	--	--	--	0.277	0.185±
9	Cyclocit	saffron,		Suffet,							±0.02	0.161 ^{A'}
	ral	herbal		1999)							9 ^{A''}	
	Others											
3	(S)-3-		--	--	--	--	--	--	--	--	--	--
2	Ethyl-4-											
	methylp											
	entanol											
5	Nonadec	Alkane	--	--	--	--	--	--	--	--	--	--
8	ane											

* = Odor-active compound (OAV >1 under at least one of the treatments)

a-c = Values with different superscripts in the same row are significantly different ($P \leq 0.05$)

(20 min distillation time)

A-B = Values with different superscripts in the same row are significantly different ($P \leq 0.05$)

(40 min distillation time)

a'-b'= Values with different superscripts in the same row are significantly different ($P \leq 0.05$)

(0% ethanol concentration)

a''-b''= Values with different superscripts in the same row are significantly different ($P \leq 0.05$) (30% ethanol concentration)

A'-B'= Values with different superscripts in the same row are significantly different ($P \leq 0.05$) (50% ethanol concentration)

A"-B"= Values with different superscripts in the same row are significantly different ($P \leq 0.05$) (70% ethanol concentration)

Fig. 1. Flow diagram of optimization of aroma compounds extraction from Chardonnay grape pomace

Fig. 2. GC-MS chromatograms showing the volatile compound profile of Chardonnay grape pomace extracted under three extraction conditions of (a) 24 hr of enzymatic hydrolysis, 30% ethanol/water, 20 min of distillation; (b) 48 hr of enzymatic hydrolysis, 50% ethanol/water, 20 min of distillation; (c) 24 hr of enzymatic hydrolysis, 50% ethanol/water, 20 min of distillation. Peak numbers are referred to Table 1-4; peak No. 21 is the internal standard 4-octanol.

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