



# Characterization of the key odorants in goji wines in three levels of sweetness by applications of sensomics approach

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## ABSTRACT

The correlations and differences of the key odorants were systematically conducted among three sweetness of goji wines by the sensomics approach. After aroma (extract) dilution analysis, 67, 67, and 66 odorants were screened in sweet goji wine, semi-dry goji wine, and dry goji wine, in which, 63 odorants were identified in all goji wines. Determination of 53 odorants revealed a total of 30 odorants with the concentrations surpassing their olfactory thresholds. Overall, the odor activity values (OAVs) of ketones decreased, while esters, alcohols, phenols, and aldehydes increased with the decrease in sweetness in goji wine samples. Nevertheless, (*E*)- $\beta$ -damascenone, *trans*- and *cis*-whisky lactones, and 3-methyl-2,4-nonanedione, evoked cooked apple-like, coconut-like, and hay-like odor impressions in goji wines and showed the highest OAVs. A reliable evaluation of the aroma contributions was executed as aroma recombinations and suggested a successful evaluation of key odorants in goji wines.

## 1. Introduction

Goji berry is world widely known as a “superfruit” and celebrated for its rich health benefits, including anti-oxidative, anti-inflammatory, and antitumor attributes, along with clinical applications for conditions such as non-alcohol fatty liver disease, cardiovascular disease, eye fatigue, etc. (Chang et al., 2019; Ma et al., 2022). Various goji products have been developed to enhance the goji market and create more economic value. Among these, goji wines (GWs), made from the fruit of goji berry, have emerged as functional alcoholic beverages and are increasingly favored by customers. Their phytochemicals, including carotenoids, polysaccharides, and flavonoids, were elaborated for years (Niu et al., 2017; Ren et al., 2018; Song & Xu, 2013).

Besides the nutrition, customers are also attracted by the distinctive flavor of GWs. Researchers aimed at understanding the molecular bases of the aroma profiles in GWs and thus identified about 200 volatile compounds for years (Liu et al., 2022; Lu et al., 2022; Niu et al., 2017, 2018; Ouyang et al., 2017; Ren et al., 2018; Shumin et al., 2021; Yuan et al., 2016). Ren et al. (2018) found that alcohols, esters, and acids were the predominant groups among 50 volatiles in GWs via headspace-solid

phase microextraction (HS-SPME). Often, contributions of volatiles to the aroma profile are not entirely presented by their concentrations. Considering the characteristics of each odorant in a specific matrix, the odor activity value (OAV) is introduced as the ratio of odorant concentration to its orthonasal odor threshold (OT). Accordingly, 16 of 98 volatiles in GWs detected after HS-SPME were found with concentrations exceeding their OTs (Yuan et al., 2016). Notably,  $\beta$ -ionone, 4-ethylphenol, isopentyl acetate, methionol, and other esters and acids were assigned as the key odorants and contributed the violet, phenolic, fruity, and cooked-potato odor notes (Yuan et al., 2016). Although earlier studies have investigated odorants with OAV > 1 in GWs, there is a lack of aroma recombinations and an effective combination of solvent and solvent-free extraction. It is still unclear whether these odorants could reflect the authentic aroma profiles of GWs.

It has been well documented that wine-making processing impacted GWs flavor. The maceration process resulted in more aroma compounds released from fruit skin and enriched the aroma profiles, especially increased the content of 2-methoxy-4-vinylphenol enhanced the fruity note in GW (Ouyang et al., 2017). After fermentation, numerous alcoholic products, such as wines, whiskies, rums, and cognacs, are generally

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stored in wood barrels for maturation and aging. Wood barrels could provide stable storage conditions, facilitate spontaneous clarification, and enrich the products' flavor. During GWs stored and aged in the barrels, the oak shavings improved esters formation and released oak-related compounds, including whisky lactones, vanillin, eugenol, and 2-methoxy-4-vinylphenol (Niu et al., 2017). Niu et al. (2018) pointed out that caramel-like, coffee-like, floral, and vanilla flavors, owned to the presence of vanillin, ethyl dihydrocinnamate, and eugenol, were highlighted in GWs after being treated with oak chips. According to the sweetness (represented as the total sugar contents), sweet GW (SGW), semi-dry GW (SDGW), and dry GW (DGW) were developed to fulfill customers' preferences. Sweetness has a major impact on wine flavor. Sugar in matrix imparts a distinct aroma-attractive profile and balances the acid, bitter, and astringent taste to a certain degree. Previous researches reported that the most marked odorant series for sweet wines were sweet, burnt-wood, and dried fruit-like (Figueiredo-González et al., 2014; González-Álvarez et al., 2013; Noguerol-Pato et al., 2012). Fewer flavors were detected in dry wines (Wang & Spence, 2018) and some volatiles, such as monoterpenes and norisoprenoids, were found in lower contents in dry wines (Azzolini et al., 2015). They were more like a fruity, spicy, and geranium-like flavor (Figueiredo-González et al., 2014; González-Álvarez et al., 2013; Noguerol-Pato et al., 2012). However, there is no available data related to the sweetness of GWs and their volatiles or key odorants.

This study aimed to provide comprehensive knowledge on overall aroma profile of SGW, SDGW, and DGW, and analyze the key odorants by applications of the sensomics approach. It is based on (i) relatively entire extraction of volatile compounds by the combined methods of static headspace (SHS) and solvent-assisted-flavor-evaporation (SAFE), (ii) carefully identification of odorants by a combination of gas chromatography-olfactometry (GC-O) and gas chromatography-mass spectrometry (GC-MS), (iii) screening of the odorants by aroma (extract) dilution analysis (A(EDA)), (iv) quantitation by stable isotope dilution assays (SIDAs) and semi-quantitation by internal standard (IS) methods, as well as (v) evaluation of the contributions of odorants by recombination experiments.

## 2. Materials and methods

### 2.1. Chemicals

The following references and chemicals were provided by the suppliers given in parentheses: methionol (98%), 2-methoxyphenol (98%), 3-methyl-1-butanol (99%), 2-methylbutanoic acid (98%), 6-methyl-5-hepten-2-one (98%), phenylacetic acid (99%), ethyl hexanoate (99%), ethyl 2-methylpropanoate (99%), methyl phenylacetate (99%), heptanoic acid (98%), pentanoic acid (99%), ethyl cinnamate ( $\geq 98\%$ ),  $\alpha$ -terpineol (96%), (*E*)-3-hexen-1-ol (97%), ethyl 2-phenylacetate (99%), ethyl 2-methylbutanoate (98%), butanoic acid (99%), decanoic acid (99%), ethyl 3-methylbutanoate (98%), ethyl 3-phenylpropanoate ( $\geq 98\%$ ), 1,1-diethoxyethane (99%) (Alfa Aesar, Karlsruhe, Germany); octanal (99%), 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone (99%), (*E,E*)-2,4-heptadienal (90%), octanoic acid ( $\geq 98\%$ ), (*E*)- $\beta$ -damascenone (1.1–1.4%),  $\gamma$ -nonalactone (97%), ethyl butanoate (99%), whisky lactone (98%), 2-phenylethyl acetate (99%), ethyl propanoate (99%) (Sigma-Aldrich Chemie, Taufkirchen, Germany); dimethyl trisulfide (98%), linalool (98%), 3-methylbutanoic acid (99%), 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone (97.5%), 2-methoxy-4-vinylphenol (98%), ethyl octanoate (99%) (J&K scientific Ltd., Shanghai, China); *m*-cresol ( $> 98\%$ ), 2-methoxy-5-methylphenol ( $> 98\%$ ), nonanal (95%), (*E*)-2-nonenal ( $> 95\%$ ), hexanoic acid ( $> 98\%$ ), 2-methylbutyl acetate ( $> 98\%$ ) (TCI, Eschborn, Germany); vanillin (99%), phenylacetaldehyde (98%), 3-phenylpropanoic acid (99%), 3-methylbutyl acetate (99%), *p*-cresol ( $> 99\%$ ) (Acros Organics; Thermo Fisher Scientific, Nidderau, Germany); 1-octen-3-one (97.7%), 3-isobutyl-2-methoxypyrazine (99.6%), (*E,Z*)-2,6-nonadienal (96% mixture isomers) (BLD pharm,

Karlsruhe, Germany); nonanoic acid ( $> 97\%$ ), acetic acid (Fluka, Neu-Ulm, Germany); hydrochloric acid (HCl; 37%), sodium chloride (NaCl; 99%), sodium sulphate (Na<sub>2</sub>SO<sub>4</sub>; specification as European Pharmacopoeia (reag. ph. Eur.)), decanal (97%), eugenol (reag. ph. Eur.), acetophenone (98%) (Merck, Darmstadt, Germany); diethyl ether (DEE;  $\geq 99.5\%$ ) (Fisher Scientific, Karlsruhe, Germany); ethanol (for HPLC), methanol (for HPLC) (Th.Geyer, Renningen, Germany); *n*-pentane ( $\geq 99.7\%$ ) (VWR, Leuven, Belgium); silica gel 60 (0.04–0.063 mm, Macherey-Nagel, Düren, Germany); 2-phenylethanol (99%) (Carl Roth, Karlsruhe, Germany); ethyl acetate (for pesticide residue analysis) (Honeywell, Hessen, Germany); liquid nitrogen, helium (99.9990%), hydrogen (99.999%), nitrogen (99.95%) (Westfalen, Münster, Germany). DEE was freshly distilled via a Vigreux column before use and stabilized by adding potassium hydroxide (VWR, Darmstadt, Germany).

### 2.2. Materials

SGW, SDGW, and DGW containing 12% alcohol (by vol.) were online purchased from the manufacturer in 2021. The raw materials (goji berries) of these GWs were from the same area (37°22'N, 105°40'E, Zhongwei, Ningxia, China) and collected in the same year. After the fermentation, the GWs were stored in the oak barrels. The final products were made in 2021. The arrived GW samples were stored at room temperature in the dark prior to the analysis.

### 2.3. Stable isotopically labeled references

Labeled references of [<sup>13</sup>C<sub>2</sub>]-2-methylpropyl acetate, [<sup>2</sup>H<sub>5</sub>]-ethyl butanoate, [<sup>2</sup>H<sub>5</sub>]-ethyl-3-methylbutanoate, [<sup>13</sup>C<sub>2</sub>]-3-methylbutyl acetate, [<sup>2</sup>H<sub>5</sub>]-ethyl hexanoate, [<sup>2</sup>H<sub>5</sub>]-ethyl octanoate, [<sup>13</sup>C<sub>2</sub>]-phenylacetic acid, and [<sup>2</sup>H<sub>3</sub>]-vanillin were obtained from Sarah Kramp and Prof. Dr. Michael Granvogl (Department of Food Chemistry and Analytical Chemistry, Institute of Food Chemistry, University of Hohenheim).

### 2.4. Isolation of the volatiles

#### 2.4.1. SAFE

GW (100 mL) was separated and extracted three times with an equal amount of DEE, followed by washing three times with aqueous NaCl solution (1 mol/L, 300 mL). After drying over anhydrous Na<sub>2</sub>SO<sub>4</sub>, the extract was filtered and isolated the volatiles from nonvolatiles using the SAFE technique (Engel et al., 1999). The distillate was carried on Vigreux column (50 × 1 cm i.d.) followed by microdistillation to concentrate to ~200  $\mu$ L.

#### 2.4.2. SHS

To detect the very high-volatile compounds that were co-eluted with the solvent by liquid extraction, the SHS analysis was performed. An aliquot (5 mL) of sample and NaCl (1 g) in a septum-sealed vessel (20 mL) were incubated for 30 min at 40 °C.

#### 2.4.3. HS-SPME

To a better determination of the concentrations of the high-volatile odorants, which detected with the concentrations below the limit of quantitation (LOQ) level by using SHS method, HS-SPME were executed according to the previous description (Zhang et al., 2023) with slight modification. A divinylbenzene/carboxen/polydimethylsiloxane fiber (50  $\mu$ m/30  $\mu$ m, 2 cm, Suplco Inc., Bellefonte, United States) was used for HS-SPME. A headspace vial (20 mL) with sample (10 mL) and NaCl (1 g) was extracted for 30 min with a magnetic stirring rate at 300 rpm, 70 °C, and incubated for 10 min for equilibrium.

### 2.5. Instrumentation

#### 2.5.1. GC-O

A type 5160 gas chromatography (Carlo Erba Instrument, Milano,

Italy) was operated with two fused silica capillary columns, either a DB-FFAP or a DB-5 (both 30 m × 0.32 mm i.d., 0.25 μm film thickness; both J&W Scientific; Agilent, Waldbronn, Germany), for GC-O analysis. The distillate was injected by the on-column technique. The oven temperature was set to be held for 2 min at 40 °C, then raised at 6 °C/min to 230 °C, and held for 5 min. At the end of the column, the effluent was 1:1 split with a Y-type quick-seal glass splitter (Agilent Technologies, California, USA) connected with two equal lengths of deactivated fused-silica capillaries. One part of the effluent was connected to a flame ionization detector (FID) (230 °C), and the other part of the effluent was connected to a sniffing port (230 °C). A series of *n*-alkanes was used for determining the linear retention indices (RIs).

#### 2.5.2. Static headspace–gas chromatography–olfactometry–mass spectrometry (SHS–GC–O–MS)

Due to the low boiling points, high-volatile odorants were identified and screened using Thermo Scientific trace 1300 gas chromatography (GC, Dreieich, Germany) with a sniffing port of static headspace. After extraction of SHS, the sample (2.5 mL) was injected by splitless injection at 35 °C as an initial temperature and flushed onto a DB-5 fused silica column (30 m × 0.25 mm i.d., 0.25 μm film thickness; J&W Scientific; Agilent, Waldbronn, Germany). After injection, oven temperature was held at 35 °C for 2 min, followed by ramped to 100 °C at 3 °C/min, ramped to 230 °C at 20 °C/min, then held for 5 min. The effluent was split into two equal parts at the end of the column. One part was flowed to a sniffing port (230 °C), and the other part was flowed to a Thermo ISO 7000 single quadrupole mass spectrometer (Dreieich, Germany). The helium served as the carrier gas and was maintained at 1.6 mL/min. The samples (2 μL) were analyzed in electron impact mode (MS-EI) at 70 eV and generated mass spectra in full scan mode (*m/z* 35–300).

#### 2.5.3. Gas chromatography–mass spectrometry (GC–MS)

The GC–MS was equipped with a Thermo TriPlus RSH autosampler and used with a polar or nonpolar fused silica capillary column depending on the analysis, as described in Section 2.5.2. The equal MS information was used as described above in Section 2.5.2 and the temperature program was as described above in Section 2.5.1.

An Agilent 7890 B GC was equipped with an MPS robotic autosampler (Gerstel, Mülheim, Germany) with either HS–SPME or liquid injection and an Agilent J&W DB–WAXms column (30 m × 0.25 mm i.d., 0.25 μm film thickness; Agilent, Waldbronn, Germany). The end of the column was linked with a 5977 B mass spectrometry detector (Agilent, Waldbronn, Germany). Helium was kept at a constant flow rate of 1.62 mL/min as carrier gas. The GC temperature was the same as it was mentioned above in Section 2.5.2.

#### 2.5.4. Two–dimensional gas chromatography–mass spectrometry (2D–GC–MS)

The co-eluted compounds or trace odorants were identified by 2D–GC–MS. The first GC (Thermo Scientific trace 1300 GC) with an FID and a sniffing port was equipped with a DB–FFAP column, and connected to a second GC equipped with a sniffing port and a DB–5 column via a cold trap. The end of the second column was connected to MS. The temperature program and MS information were as mentioned in Section 2.5.1 and 2.5.2.

### 2.6. AEDA

To achieve a better screening of odorants by GC–O, the SAFE distillate (~200 μL) was diluted to 1 mL with DEE and then stepwise diluted with DEE (1 + 1 by vol.) for the AEDA application (Schieberle, 1995). Flavor dilution (FD) factors, showing the highest detectable dilution, were obtained for each odor-active region. AEDA was the average of triplicate results and differed by ≤ two dilution steps (Grosch, 1994).

### 2.7. ADA

In terms of the screening of the high-volatile odorants, samples were 1:1 stepwise diluted with water in headspace vials (20 mL) prior to SHS–GC–O–MS analysis (Schieberle, 1995). The FD factors of ADA were analyzed as mentioned in Section 2.6.

### 2.8. (Semi-) quantitation of volatiles in GW

#### 2.8.1. Internal standard (IS) methods

The internal standards, 2-methylvaleric acid (IS1), thymol (IS2), and *α*-methyl- $\gamma$ -butyrolactone (IS3), were used for the semi-quantitative measurement. The relative standards and target compounds (2–50 μg/mL) were dissolved in DEE at five concentration ratios (1:5, 1:3, 1:1, 3:1, and 5:1). Solutions were obtained the peak areas at selected ion of internal standards and analytes via GC–MS (Table 1). The calibration curves were established according to the concentration ratios and respective peak areas (Table 1). IS (0.3–150 μg, considering of the results of the analytes estimated from preliminary semi-quantitation experiments) dissolved in DEE and GW (10–100 mL) was equilibrated for 30 min. The volatiles in GW were extracted as mentioned in Section 2.4.

#### 2.8.2. SIDA

The labeled standard (2–50 μg, depending on determined data from preliminary experiments) in DEE was equilibrated for 30 min with different amounts of GW (5–100 mL). Calibration factors were determined by analyzing peak area ratios and responding concentration ratios (1:5, 1:3, 1:1, 3:1, and 5:1) of labeled compounds and analytes (Table 2). The high-volatile compounds were quantitated by HS–SPME, in which case labeled references or internal standards in methanol were equilibrated with GWs.

### 2.9. Sensory analyses

#### 2.9.1. Aroma profile analysis (APA)

A sensory panel with 15 experienced assessors was involved in sensory analyses (Czerny et al., 2008). The odor attributes were selected as cooked apple-like (*E*- $\beta$ -damascenone), coconut-like (whisky lactone), fatty (*E*-2-nonenal), fruity (ethyl 2-methylbutanoate), hay-like (3-methyl-2,4-nonanedione), floral (phenylacetaldehyde), malty (3-methyl-1-butanol), and green (octanal). The standards were dissolved in ethanol and then diluted at the concentration of 100-fold of their OTs in a 12% ethanol/water solution. GW (15 mL) was put into a Teflon vessel at room temperature. For the sensory analyses, the panelists rated each odor intensity in GW using a linear scale in increments of 0.5 from 0 (not perceivable) to 3 (very strong).

#### 2.9.2. Aroma recombination

According to the concentrations that were determined in the original GWs, all the analyzed odorants with OAV ≥ 1 were mixed and dissolved in the respective odorless matrix. The isomers (2- and 3-methylbutyl acetate) were quantitated as a sum because of the presence of their corresponding ethyl esters (EEs) (ethyl 2- and 3-methylbutanoates) in a similar ratio of 1:1. For *trans*- and *cis*-whisky lactone, the sum was used for recombination by the commercial reference which contained ~1:1 of these two isomers. To imitate the original aroma profiles, the matrixes, ethanol/water (12% by vol.) solution that was adjusted to pH 1.5 (for SGW) or pH 3 (for SDGW and DGW) with tartaric acid (5 g/L), were considered. The sugar contents in matrixes were set with consideration of the sugar content occurred in original GWs samples (Table S2). The matrixes also contained 30 g/L fructose and 20 g/L glucose (for SGW), 6 g/L fructose and 4 g/L glucose (for SDGW), and no sugar (for DGW). The recombinants (15 mL) were evaluated by panelists via the same procedure described in Section 2.9.1 at room temperature.

**Table 1**Selected internal standards, quantified mass ions ( $m/z$ ), and calibration curve equations used in the semi-quantitation by internal standard methods.

Odorant	Internal standard <sup>d</sup>	System <sup>b</sup>	Ion ( $m/z$ ) <sup>c</sup>		Calibration curve <sup>d</sup>	R <sup>2</sup>
			an.	is.		
octanoic acid	IS1	I	60	74	$y = 0.6641 \times -0.1051$	0.9921
nonanoic acid	IS1	I	73	74	$y = 0.3587 \times -0.0176$	0.9994
3-phenylpropanoic acid	IS1	I	91	74	$y = 0.7304 \times -0.0358$	1
acetic acid	IS1	II	60	74	$y = 0.0712 \times -0.0182$	0.9979
hexanoic acid	IS1	I	60	74	$y = 0.8255 \times -0.1081$	0.9979
decanoic acid	IS1	I	73	74	$y = 0.4132 \times -0.0052$	0.9999
heptanoic acid	IS1	I	60	74	$y = 0.8113 \times -0.1317$	0.9905
pentanoic acid	IS1	I	60	74	$y = 0.8403 \times -0.105$	0.9984
nonanal	IS1	I	57	74	$y = 0.3731 \times +0.0121$	0.9975
( <i>E</i> )-2-nonenal	IS1	I	55	74	$y = 1.5951 \times -0.3383$	0.9994
octanal	IS1	II	84	74	$y = 2.2023 \times -0.6122$	0.9944
decanal	IS1	II	57	74	$y = 3.5894 \times -0.0213$	0.9916
butanoic acid	IS1	I	60	74	$y = 0.4285 \times -0.0311$	0.9999
2-methylbutanoic acid	IS1	I	60	74	$y = 1.3181 \times -0.3865$	0.9932
3-methylbutanoic acid	IS1	I	60	74	$y = 1.3181 \times -0.3865$	0.9932
phenylacetaldehyde	IS1	I	91	74	$y = 9.5791 \times -1.2803$	0.9957
<i>p</i> -cresol	IS2	I	107	135	$y = 0.579 \times +0.0415$	0.9992
2-phenylethanol	IS2	II	91	135	$y = 0.3628 \times -0.0769$	0.9990
3-methyl-1-butanol	IS2	II	55	135	$y = 0.0857 \times -0.014$	0.9987
2-methoxyphenol	IS2	I	109	135	$y = 0.2871 \times -0.0204$	0.9970
2-methoxy-4-vinylphenol	IS2	I	135	135	$y = 0.397 \times +0.0204$	0.9967
eugenol	IS2	I	164	135	$y = 0.2958 \times -0.021$	0.9975
linalool	IS2	I	71	135	$y = 0.2949 \times +0.0217$	0.9907
$\alpha$ -terpineol	IS2	I	59	135	$y = 0.3071 \times +0.0025$	0.9994
methionol	IS2	I	106	135	$y = 0.2148 \times -0.0295$	0.9976
<i>m</i> -cresol	IS2	I	107	135	$y = 0.4492 \times -0.0096$	0.9950
( <i>E</i> )-3-hexen-1-ol	IS2	I	67	135	$y = 0.2472 \times -0.0121$	0.9990
( <i>E</i> )- $\beta$ -damascenone	IS3	I	69	56	$y = 0.4754 \times -0.0035$	0.9998
<i>trans</i> -whisky lactone	IS3	I	99	56	$y = 3.8783 \times -0.0946$	0.9990
<i>cis</i> -whisky lactone	IS3	I	99	56	$y = 3.8796 \times -0.0848$	0.9988
4-hydroxy-2,5-dimethyl-3(2 <i>H</i> )-furanone	IS3	I	128	56	$y = 1.0518 \times -0.2044$	0.9901
$\gamma$ -nonalactone	IS3	I	85	56	$y = 5.1889 \times -1.1375$	0.9976
6-methyl-5-hepten-2-one	IS3	I	108	56	$y = 1.0647 \times +0.0651$	0.9986
acetophenone	IS3	I	105	56	$y = 3.3856 \times +0.5768$	0.9965
ethyl 2-phenylacetate	IS3	I	91	56	$y = 5.6704 \times +0.7411$	0.9979
ethyl cinnamate	IS3	I	131	56	$y = 4.3711 \times -0.3358$	0.9973
3-methyl-2,4-nonanedione	IS3	I	99	56	$y = 3.6796 \times -0.6399$	0.9893
2-phenylethyl acetate	IS3	I	104	56	$y = 5.2772 \times +0.0435$	0.9994
ethyl 3-phenylpropanoate	IS3	I	104	56	$y = 3.3492 \times -0.0115$	0.9978
3-hydroxy-4,5-dimethyl-2(5 <i>H</i> )-furanone	IS3	I	83	56	$y = 1.201 \times -0.1502$	0.9976

<sup>a</sup> IS1, 2-methylvaleric acid; IS2, thymol; IS3,  $\alpha$ -methyl- $\gamma$ -butyrolactone.<sup>b</sup> System I, GC-MS equipped with liquid injection; System II, HS-SPME-GC-MS equipped with a DB-WAXms capillary column.<sup>c</sup> Quantifier ions used for quantitation in electron impact (EI) mode; an., analyte; is., internal standard.<sup>d</sup> The curve was fitted by peak area ratios and concentration ratios of analyte and the relevant internal standard.**Table 2**Stable isotopically labeled standards, quantified mass ions ( $m/z$ ), and calibration curve equations used in the quantitation by stable isotope dilution assays (SIDAs).

Odorant	Internal standard <sup>d</sup>	Ion ( $m/z$ ) <sup>b</sup>		Calibration curve <sup>c</sup>	R <sup>2</sup>
		an.	is.		
ethyl 2-methylpropanoate <sup>d</sup>	[ <sup>13</sup> C <sub>2</sub> ]-2-methylpropyl acetate	71	75	$y = 0.9868 \times +0.0734$	0.9999
2-methylpropyl acetate	[ <sup>13</sup> C <sub>2</sub> ]-2-methylpropyl acetate	73	75	$y = 1.2963 \times +0.0263$	0.9971
ethyl propanoate <sup>e</sup>	[ <sup>2</sup> H <sub>5</sub> ]-ethyl butanoate	57	93	$y = 1.3602 \times -1.023$	0.9951
ethyl butanoate	[ <sup>2</sup> H <sub>5</sub> ]-ethyl butanoate	88	93	$y = 0.9882 \times +0.1198$	0.9910
ethyl 3-methylbutanoate	[ <sup>2</sup> H <sub>5</sub> ]-ethyl-3-methylbutanoate	88	93	$y = 0.9542 \times -0.2954$	0.9948
ethyl 2-methylbutanoate <sup>d</sup>	[ <sup>2</sup> H <sub>5</sub> ]-ethyl-3-methylbutanoate	57	93	$y = 1.2184 \times -0.4144$	0.9929
3-methylbutyl acetate <sup>f</sup>	[ <sup>13</sup> C <sub>2</sub> ]-3-methylbutyl acetate	41	43	$y = 1.3012 \times -0.4094$	0.9922
2-methylbutyl acetate <sup>f</sup>	[ <sup>13</sup> C <sub>2</sub> ]-3-methylbutyl acetate	41	43	$y = 1.3012 \times -0.4094$	0.9922
ethyl hexanoate	[ <sup>2</sup> H <sub>5</sub> ]-ethyl hexanoate	88	93	$y = 0.7284 \times -0.1836$	0.9907
ethyl octanoate	[ <sup>2</sup> H <sub>5</sub> ]-ethyl octanoate	88	93	$y = 0.556 \times -0.0159$	0.9948
phenylacetic acid <sup>g</sup>	[ <sup>13</sup> C <sub>2</sub> ]-phenylacetic acid	91	92	$y = 0.944 \times +0.0425$	0.9957
vanillin <sup>g</sup>	[ <sup>2</sup> H <sub>5</sub> ]-vanillin	151	154	$y = 0.9657 \times -0.0243$	0.9990

<sup>a</sup> Stable isotopically labeled internal standards were used for quantitation by SIDAs.<sup>b</sup> Quantifier ions used for quantitation in electron impact (EI) mode; an., analyte; is., internal standard; the analysis was carried on HS-SPME-GC-MS equipped with a DB-WAXms capillary column.<sup>c</sup> The curve was fitted by peak area ratios and concentration ratios of analyte and the relevant internal standard.<sup>d</sup> Analyte was determined by using its isotopically labeled isomer as the internal standard.<sup>e</sup> Analyte was determined by using its isotopically labeled homologue as the internal standard.<sup>f</sup> Analyte was determined the sum of both the 2- and 3-methyl isomers, 3-methylbutyl acetate and [<sup>13</sup>C<sub>2</sub>]-3-methylbutyl acetate was used for calibration.<sup>g</sup> Analytes were determined by a GC-MS equipped with liquid injection and DB-WAXms capillary column.

### 3. Results and discussion

#### 3.1. APA results of GWs

Sensory evaluations were executed to get the first look of overall GWs aroma profiles (Fig. 1). The most intense odor impression was cooked apple-like, and the rates of this odor note were quite similar among the three sweetness levels of GWs. While the aroma differences among GWs were mainly shown in the perceivability of fatty, green, and malty odor impressions. The fatty and malty odor impressions were strongest in DGW, while the green odor notes were weakest in DGW.

#### 3.2. Identification of the odorants in GWs

After being subjected to GC-O and screening odorants via AEDA, the first impressions of the odor-active regions in GWs can be displayed by the FD chromatograms (Fig. 2). In SGW, 61 regions were aroma-perceivable in the SAFE distillate. Among these, 18 regions were in the  $FD \geq 64$ . In the SDGW distillate, 22 of the total 59 odor-active areas were verified in the FD factor range of 64–8192. Three lacked odorants were found in SDGW compared with those found in SGW. The DGW SAFE-distillate was found to have 60 odor-active regions, 20 of which were confirmed in the FD factor range from 64 to 16,384. Two additional odorants were detected compared to SGW. Surprisingly, the minimal number ( $n = 6$ ) of most intense odor regions (with  $FD \geq 512$ ) was found in SGW.

To determine the odorants, the corresponding odor quality, odor intensity, and the RIs of odor-active regions on both polar and nonpolar capillary columns were referred to an in-house database consisting of over 1000 aroma references. Then, the mass spectra in EI mode were further used to compare the potential structure for each odor-active region in the sample and the authentic reference. The most intense smells with  $FD \geq 512$  were assigned as 3-hydroxy-4,5-dimethyl-2(5H)-furanone (58), (*E*)- $\beta$ -damascenone (38), *trans*-whisky lactone (42), 3-methyl-1-butanol (7), and 2-methoxy-4-vinylphenol (57), which contributed seasoning-like, lovage-like, cooked apple-like, coconut-like, malty, and smoky, clove-like odor notes, respectively (Table 3). However, due to the low concentration and weak MS signal in GWs, 22, inciting bellpepper-like odor impression, could be identified as 3-isobutyl-2-methoxypyrazine only based on comparing odor quality and the RIs on both capillary columns to the authentic reference. Additional important odorants with high FD factor in SDGW and DGW were identified as butanoic acid (27), 2-, and 3-methylbutanoic acid (30), acetic acid (18), 2-phenylethanol (43), and 4-hydroxy-2,5-dimethyl-3(2H)-furanone (49) (Table 3). Among these, 27, 30, and 18 were responsible for the sweaty, and vinegar-like smell, which might also be responsible for the character flavor in SDGW and DGW overall profile.

Headspace coupled with GC equipped with a sniffing port was performed to assign the high-volatile odorants. It enabled the identification of six additional compounds in SGW. Ethyl acetate (HS-2) was the

compound with the highest FD factor (32) (Table 4). HS-ADA revealed two additional odorants were found in SDGW. In DGW, no additional odorant was found compared to SGW, and ethyl 2-methylpropanoate (HS-6), eliciting a fruity odor note, was found as the odorant with FD factor of 16.

The major structural group in known odorants of GWs was observed as esters ( $n = 18$ ), followed by acids ( $n = 11$ ), aldehydes ( $n = 10$ ), ketones, alcohols, and phenols ( $n = 7$ ) (Tables 3 and 4). The previous investigations on GWs' flavor identified the volatiles via headspace extraction methods (Liu et al., 2022; Lu et al., 2022; Niu et al., 2017, 2018; Ouyang et al., 2017; Ren et al., 2018; Shumin et al., 2021; Yuan et al., 2016). Without relatively complete extraction methods (e.g., SAFE) or any human-perceivable detector (e.g., GC-O), we would miss the trace compounds that might be crucial to the overall aroma profiles of GWs. In our study, 35 of the total 71 odorants were first found in GWs, and 28 of these 35 compounds were found in all GW samples.

#### 3.3. Concentrations of the odorants in GWs and the OAVs

Semi-quantitation of 40 further compounds was analyzed by IS methods. IS1 (2-methylvaleric acid) was used for the semi-quantitation of acids and aldehydes; IS2 (thymol) was used for the semi-quantitation of phenols and alcohols; IS3 ( $\alpha$ -methyl- $\gamma$ -butyrolactone) was used for the semi-quantitation of esters and ketones (Table 1). Quantitation of twelve odorants with high FD factors ( $\geq 32$ ) or high volatility was performed by SIDA (Table 2). Ethanol was semi-quantified with *tert*-butanol as IS by using GC-FID (Table S1).

##### 3.3.1. Concentrations of the odorants in GWs

With labeled 12% alcohol content in the GWs, it is no wonder ethanol showed the highest concentrations in all GWs ( $8.8 \times 10^7$ – $9.9 \times 10^7$   $\mu\text{g/L}$ ). With the decrease of the sweetness of GWs, the contents of alcohols and acids increased with the representation of 3-methyl-1-butanol (7) ( $1.8 \times 10^5$ – $2.8 \times 10^5$   $\mu\text{g/L}$ ), 2-phenylethanol (43) ( $0.84 \times 10^4$ – $8.2 \times 10^4$   $\mu\text{g/L}$ ), and acetic acid (18) ( $1.5 \times 10^4$ – $7.0 \times 10^4$   $\mu\text{g/L}$ ) in our study. 3-Methyl-1-butanol and 2-phenylethanol are well-known higher alcohols produced during yeast fermentation, and they contribute to the flavor complexity of alcoholic products. They are predominately formed from  $\alpha$ -keto acids, which are generated from the deamination of their respective parent amino acids (leucine, isoleucine, valine, and 2-phenylalanine) or glucose metabolism via pyruvate. Afterward, the higher alcohols are synthesized from the decarboxylation of the  $\alpha$ -keto acids and the reduction of the formed fusel aldehydes following the Ehrlich mechanism (Cordente et al., 2012; Ehrlich, 1904; Hazelwood et al., 2008; Hernández-Orte et al., 2006; Vidal et al., 2015). Acetic acid was previously reported to occupy up to ~48% peak ingredient in red goji berry fruit (Chen et al., 2015; Lu et al., 2017). Another possible transformation pathway of acetic acid is postulated to be generated during aging or fermentation, as reported in kiwi wine (Li et al., 2022) and grape wine (Contreras et al., 2014). In consistent, the previous

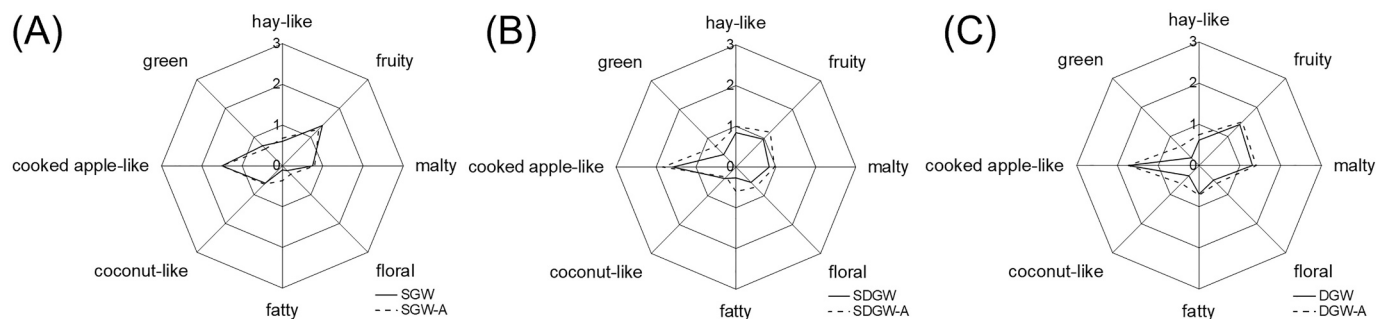
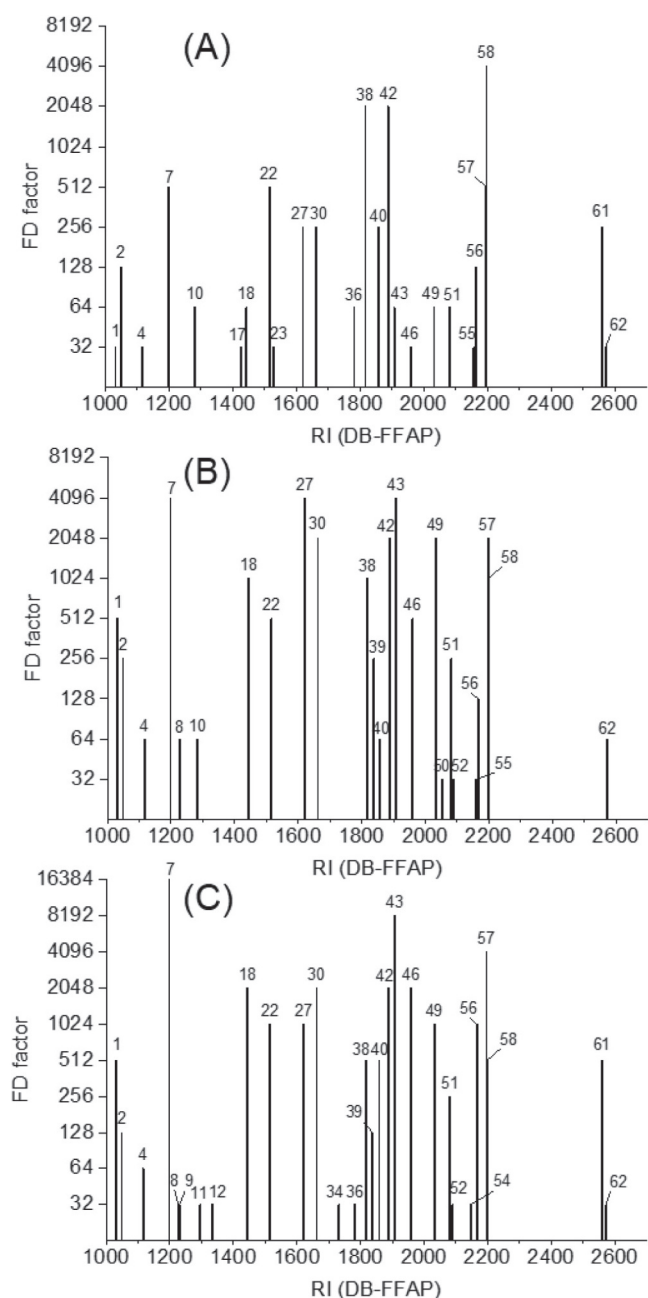


Fig. 1. The overall aroma profiles of (A) sweet goji wine, (B) semi-dry goji wine, and (C) dry goji wine were in solid line, and their aroma recombinants were in dashed line.



**Fig. 2.** Flavor dilution (FD) chromatograms of the distillate obtained by AEDA on the SAFE distillation of (A) sweet goji wine, (B) semi-dry goji wine, and (C) dry goji wine (compounds with RI (DB-FFAP)  $\geq$  1000 and FD factors  $\geq$  32 were displayed).

investigation reported the increment of acetic acid in GWs after aging in oak barrels (Niu et al., 2017, 2018).

### 3.3.2. OAVs of the odorants

However, AEDA and quantitation data ignore the interaction between odorants and matrix. The release ability of each odorant is not taken into consideration. Therefore, OAV of each odorant was calculated compared to its OT. In this study, 22, 25, and 26 odorants were found with OAVs  $\geq$  1 in SGW, SDGW, and DGW, respectively. (*E*)- $\beta$ -Damascenone (38, OAVs range: 980–3600) and *trans*-/*cis*-whisky lactone (42 and 46, OAVs range: 280–690) were presented the highest OAVs in all GWs. Higher OAVs were also detected for 3-methyl-2,4-nonanedione (32, 170) in SGW, (*E*)-nonenal (23, 290), and ethyl octanoate (17,

230) in SDGW, and (*E*)-2-nonenal (23,300) in DGW.

The co-eluted compounds, 3-methylbutyl acetate and 2-methylbutyl acetate, were in similar presence of MS spectra, odor quality, and retention time on both DB-FFAP and DB-5 capillary columns. Their concentrations and OAVs were calculated together as a sum. In agreement with that, 3-methylbutyl acetate was previously found in high OAVs in GWs (Ouyang et al., 2017; Yuan et al., 2016). However, earlier investigations only assigned  $< 16$  compounds with OAV  $\geq 1$  in GWs, ten (4, 7, 8, 17, 37, 42, 43, 46, 50, and 56) of these were also confirmed with OAV  $\geq 1$  in our study (Niu et al., 2017, 2018; Ouyang et al., 2017; Ren et al., 2018; Yuan et al., 2016).  $\beta$ -Ionone, which was reported to have the highest OAVs in GWs (Ren et al., 2018; Yuan et al., 2016), was undetectable in our study. Eight (HS-6, 10, 12, 23, 28, 32, 37, and 58) of the key odorants in our study were unmentioned previously.

### 3.4. Recombination test

The sugar and acid compositions and contents in food matrix greatly affected the release ability and perceived intensity of aroma compounds (Arvisenet et al., 2019). Considering the interaction of odorants and food matrixes, the matrix for each recombinant was set to resemble sugar composition, sugar content (Table 5), and acidic atmosphere to the respective original GW sample. All the odorants with OAVs  $\geq 1$  were diluted to the determined concentrations in the original samples. Compared to the respective recombinants, although the original GW samples lacked of hay-like, malty, floral, and fatty odor impressions, the sensory evaluation value of the original GW samples and respective recombinants showed a good similarity. All in all, these data successfully confirmed that the odorants with OAV  $\geq 1$  were the key odorants for GWs in different sweetness, and the resemble replicas had been created in recombinants.

### 3.5. Comparison of key odorants in three sweetness of GWs

The amounts of (*E*)- $\beta$ -damascenone (38) were previously reported to be impacted by the maturity of raw materials and the addition of raw materials, pectin compound enzyme, and *Saccharomyces cerevisiae* in GWs (Lu et al., 2022; Ren et al., 2018). In our study, the amounts of (*E*)- $\beta$ -damascenone were also influenced by the sweetness (Fig. 3B). As a C<sub>13</sub>-norisoprenoid compound, (*E*)- $\beta$ -damascenone evoked a flowery, cooked apple-like, and woody smell. It highlights the contributed flavor to the overall GWs aroma profile. The previous investigations demonstrated that (*E*)- $\beta$ -damascenone revealed the original fruit material aroma and played a role as the key odorant in wines (Noguerol-Pato et al., 2012; Pineau et al., 2007). Interestingly, (*E*)- $\beta$ -damascenone can promote the sensory effects in wines and serve as a flavor enhancer in wine (Pineau et al., 2007). The transformation route of (*E*)- $\beta$ -damascenone is known to be related to the degradation of grape glyco-conjugated precursors (Parker et al., 2018; Winterhalter & Schreier, 1995). In natural plants, (*E*)- $\beta$ -damascenone has been well demonstrated to be generated from carotenoids, especially neoxanthin (Parker et al., 2018; Yang et al., 2019). In wine processing, norisoprenoids are enzymatic or nonenzymatic oxidized from carotenoids, which are unstable under oxygen, sun exposure, and elevated temperatures (Cejudo-Bastante et al., 2013; Mendes-Pinto, 2009). This might be the reason for the increment of (*E*)- $\beta$ -damascenone after a longer wine aging and storage (Cejudo-Bastante et al., 2013). The phenomenon that dried wines contained lower contents and lower FD factors of (*E*)- $\beta$ -damascenone compared to sweet wines was found in GWs, which was consistent with other wines, such as grape wines (Lan et al., 2019). However, the sensory impressions of cooked apple-like odor notes were quite similar among the three sweetness levels of GWs. The sensory effect might not only depend on the concentration of (*E*)- $\beta$ -damascenone but also result from some interactions between some compounds in GWs and (*E*)- $\beta$ -damascenone.

Another representative ketone in GWs was 3-methyl-2,4-

**Table 3**  
Odor-active compounds in SGW, SDGW, and DGW SAFE distillates.

No. <sup>a</sup>	Odorant <sup>b</sup>	Odor quality <sup>c</sup>	RI <sup>d</sup>		FD factor <sup>e</sup>			refs <sup>f</sup>
			DB-FFAP	DB-5	SGW	SDGW	DGW	
1	ethyl butanoate	fruity	1032	801	32	512	512	i,ii,iii,iv,v
2	ethyl 2-methylbutanoate	fruity	1049	847	128	256	128	i
3	ethyl 3-methylbutanoate	fruity, blueberry-like	1066	853	16	16	4	i,iv
4	2, and 3-methylbutyl acetate	sweet, fruity	1117	877	32	64	64	ii,iii
5	unknown	fatty	1125	nd <sup>h</sup>	8			
6	unknown	citrus-like, hop-like	1157	nd <sup>h</sup>	16	8	16	
7	3-methyl-1-butanol	malty	1198	<800	512	4096	16,384	i,ii, iii,iv,v,vi,vii
8	ethyl hexanoate	fruity, pineapple-like	1227	998	16	64	32	i,ii,iv,v,vi,vii
9	unknown	fruity, nutty	1231	nd <sup>h</sup>	1	4	32	
10	octanal	citrus-like, green	1283	1004	64	64	16	
11	1-octen-3-one <sup>g</sup>	mushroom-like	1295	978	16	8	32	
12	6-methyl-5-hepten-2-one	mushroom-like, pepper-like	1334	984	8	1	32	
13	(E)-3-hexen-1-ol	green, grassy	1357	849	4			ii,iii
14	dimethyl trisulfide <sup>g</sup>	cabbage-like	1375	973	16	2	1	
15	nonanal	citrus-like, soapy	1386	1106	4	8	4	i,ii,iii,v
16	unknown	butter-like	1403	nd <sup>h</sup>	4		1	
17	ethyl octanoate	fruity, green	1426	1195	32	16	16	i,ii,v,vi
18	acetic acid	vinegar-like	1444	<700	64	1024	2048	i,ii,iii,iv,v,vi,vii
19	methional	cooked potato-like	1453	908	4	1	2	
20	(E,E)-2,4-heptadienal <sup>g</sup>	fatty, flowery	1490	1013	8	8	2	
21	decanal	soapy, citrus-like	1491	1208	1	4	1	ii,v
22	3-isobutyl-2-methoxypyrazine <sup>g</sup>	bell pepper-like	1516	1177	512	512	1024	
23	(E)-2-nonenal	fatty, green	1529	1161	32	4	4	
24	linalool	citrus-like, flowery	1536	1112	4	8	16	iii
25	(E,Z)-2,6-nonadienal <sup>g</sup>	cucumber-like	1581	1155	4		8	
26	unknown	roasted	1601	nd <sup>h</sup>	4	8	2	
27	butanoic acid	sweaty	1621	<800	256	4096	1024	ii,iii
28	phenylacetaldehyde	flowery, honey-like	1642	1047	16	16	4	
29	acetophenone	foxy, bitter almond-like, rubber-like	1650	1069	16	4	4	
30	2-, and 3-methylbutanoic acid	sweaty	1662	841	256	2048	2048	ii,iii,v
31	methionol	cooked potato-like	1688	983	4	8	4	i,ii,v
32	$\alpha$ -terpineol	flowery, citrus-like	1690	1199	1	2	1	iii
33	3-methyl-2,4-nonanedione	hay-like, aniseed-like, fishy	1715	1262	16	2	1	
34	pentanoic acid	sweaty, fruity	1731	881	1	8	32	
35	methyl phenylacetate <sup>g</sup>	fruity, flowery	1758	1177	8	8	2	
36	ethyl 2-phenylacetate	beeswax-like	1782	1244	64	8	32	
37	2-phenylethyl acetate	honey-like, flowery	1816	1256	1	2	4	
38	(E)- $\beta$ -damascenone	cooked apple-like	1817	1382	2048	1024	512	i,iii
39	hexanoic acid	sweaty	1838	980	16	256	128	i,iii,vi,vii
40	2-methoxyphenol	smoky, gammon-like	1858	1087	256	64	512	viii
41	ethyl 3-phenylpropanoate	cinnamon-like, fruity	1884	1349	8	1	1	i,ii,v,vii
42	trans-whisky lactone	coconut-like	1889	1301	2048	2048	2048	i,ii,vi,vii,viii
43	2-phenylethanol	floral, honey-like	1908	1115	64	4096	8196	i,ii,iii,v
44	2-methoxy-5-methylphenol <sup>g</sup>	smoky, clove-like	1941	1198	16	2	1	
45	heptanoic acid	rancid, sweaty	1945	1075	16	16	4	
46	cis-whisky lactone	coconut-like	1960	1323	32	512	2048	i,ii,vi,vii
47	trans-4,5-epoxy-(E)-2-decenal <sup>g</sup>	metallic	2014	1382	8	4	1	
48	$\gamma$ -nonalactone	coconut-like	2033	1362	2	8	2	viii
49	4-hydroxy-2,5-dimethyl-3(2H)-furanone	caramel-like	2033	1158	64	2048	1024	
50	octanoic acid	carrot-like, musty	2053	1166	8	32	2	i,ii,iii,v,vii
51	p-cresol	faecal, phenolic, horse stable-like	2081	1072	64	256	256	viii
52	m-cresol	smoky, phenolic	2089	1130	16	32	32	viii
53	ethyl cinnamate	cinnamon-like	2131	1471	2	8	16	ii,v,vi
54	unknown	fruity	2147	nd <sup>h</sup>			32	
55	nonanoic acid	moldy, pungent	2158	1282	32	32	16	vi
56	eugenol	clove-like	2166	1357	128	128	1024	i,vi,vii
57	2-methoxy-4-vinylphenol	smoky, clove-like	2197	1311	512	2048	4096	ii,v,vi,vii,viii
58	3-hydroxy-4,5-dimethyl-2(5H)-furanone	seasoning-like, lovage-like	2198	1100	4096	1024	512	
59	unknown	sweet, roasted	2208	nd <sup>h</sup>	8	8		
60	decanoic acid	soapy, musty	2266	1360	2	16	16	vi,ii,iii,v
61	phenylacetic acid	honey-like, beeswax-like	2561	1241	256	16	512	
62	vanillin	vanilla-like, sweet	2572	1401	32	64	32	vi,vii
63	3-phenylpropanoic acid	flowery, cinnamon-like	2629	1346	4	16	8	

<sup>a</sup> The number was given according to the order of the RIs on DB-FFAP capillary column.

<sup>b</sup> Odorants were identified based on comparing their odor qualities, odor intensities, RIs on both DB-FFAP and DB-5 capillary columns, and their mass spectra (EI mode) to the data of authentic references.

<sup>c</sup> Odor quality was perceived at the sniffing port during GC-O measurement.

<sup>d</sup> Retention index (RI), linear-interpolated calculated according to the retention times of reference and adjacent *n*-alkanes; determinations of RIs on DB-FFAP and DB-5 capillary columns were based on the mixture of *n*-alkanes C<sub>6</sub>-C<sub>26</sub> and C<sub>6</sub>-C<sub>19</sub> series, respectively.

<sup>e</sup> Flavor dilution (FD) factor is the highest aroma-perceivable dilution of the SAFE distillate during GC-O measurement on the sniffing port on a DB-FFAP capillary column and determined by AEDA; average of triplicate SAFE distillate samples and three parallel measurements.

<sup>f</sup> Previous references previously reported relevant odorants in GWs; i, Lu et al., 2022; ii, Ouyang et al., 2017; iii, Ren et al., 2018; iv, Shumin et al., 2021; v, Yuan et al., 2016; vi, Niu et al., 2017; vii, Niu et al., 2018; viii, Liu et al., 2022.

<sup>g</sup> The MS signal was too weak to get an unequivocal interpretation, the identification was according to other criteria in the footnote b.

<sup>h</sup> Not determined.

**Table 4**  
High-volatile odor-active detected during static headspace analysis of SGW, SDGW, and DGW.

No. <sup>a</sup>	Odorant <sup>b</sup>	Odor quality <sup>c</sup>	RI <sup>d</sup>	FD factor <sup>e</sup>			refs <sup>f</sup>
				SGW	SDGW	DGW	
HS-1	ethanol	ethanolic	<600	4	8	4	–
HS-2	ethyl acetate	solvent-like	619	32	32	4	(Lu et al., 2022; Ouyang et al., 2017; Ren et al., 2018; Shumin et al., 2021; Yuan et al., 2016)
HS-3	3-methylbutanal	malty	652		1		
HS-4	ethyl propanoate	fruity, glue-like	712	1	2	2	Shumin et al., 2021
HS-5	1,1-diethoxyethane	fruity	726	4	4	2	
HS-6	ethyl 2-methylpropanoate	fruity	756	1	8	16	
HS-7	2-methylpropyl acetate	fruity	772	1	1	1	Ouyang et al., 2017; Yuan et al., 2016
HS-8	unknown <sup>g</sup>	fruity	830		1		

<sup>a</sup> The number was given according to the order of the RIs on a DB-5 capillary column.

<sup>b</sup> Odorants were identified based on comparing their odor qualities, odor intensities, RIs on a DB-5 capillary column, and their mass spectra (EI mode) to the data of authentic references.

<sup>c</sup> Odor quality perceived at the sniffing port during GC-O measurement.

<sup>d</sup> Retention index (RI) was determined on a DB-5 capillary column using a mixture of *n*-alkanes series (C<sub>5</sub>–C<sub>9</sub>) diluted in water.

<sup>e</sup> Flavor dilution (FD) is the highest aroma-perceivable dilution of the headspace extract during GC-O-MS measurement on the sniffing port on a DB-5 capillary column and determined by ADA; average of triplicate headspace samples and three parallel measurements.

<sup>f</sup> Previous references previously reported relevant odorants in GWs; – indicated that no mention in this study.

<sup>g</sup> The MS signal was too weak to get an unequivocal interpretation; no reference was available to match the odor quality, odor intensity, or RI.

nonanedione (**32**), which evoked a strong hay-like, fishy, and dried parsley-like smell in food materials and wines. Its precursor is known to be furan fatty acids (FAs) and undergoes oxidation degradation (Masa-netz et al., 1998). 3-Methyl-2,4-nonanedione reflected the oxidation in wines during the maceration of fruits and was presented as a keto-enol tautomerism (Pons et al., 2013). The contents were also related to the wine materials, lees, and aging process in oak barrels (Thibaud et al., 2021). With the decrease in the sweetness of GWs, the contents of 3-methyl-2,4-nonanedione decreased. The trend differed from the trend of its sensory perception levels in overall aroma profiles (Fig. 1). It might be due to the salting effect in matrix resulting in solubility changes in high carbohydrate level solution. Alternatively, it could result from the interaction between odorants and other solute components.

The OAVs of esters, which are dominant in wines, increased with the decrease in sweetness of GWs (Fig. 3A). One important group of esters in GWs was lactones, represented by whisky lactone (5-*n*-butyl-4-methyl-4,5-dihydro-2(3*H*)-furanone), which mainly presented as the (4*S*,5*S*) *cis*- and (4*S*,5*R*) *trans*-diastereomers. It is the key wood-derived odorant and associated with a coconut-like descriptor. It was reported to be presented in oak wood and additionally generated from glycoconjugates of 3-methyl-4-hydroxyoctanoic acid under acidic hydrolysis or pyrolysis conditions in wood (Parker et al., 2018; Wilkinson et al., 2004). In general, *cis*-whisky lactone is more abundant than its *trans*-isomer because *cis*-whisky lactone is prone to be released at the early maturation period during the wine process (Ortega-Heras et al., 2007; Pérez-Prieto et al., 2003). Afterwards, the *cis/trans*-isomerization occurs during storage in barrels (Ortega-Heras et al., 2007; Pérez-Prieto et al., 2003). The other formation pathway of whisky lactone is to be degraded from glycosidic and other precursors via enzymatic hydrolysis with the interaction of wood barrels (Bloem et al., 2008).

Esters could also be originated from fruit materials or alcoholic fermentation. In GWs, the EEs of C<sub>6</sub> and C<sub>8</sub> FAs (ethyl hexanoate, **8**, and

ethyl octanoate, **17**) elicited the fruity flavor, and detected the higher concentrations above their thresholds. In raw materials, these C<sub>8</sub> and C<sub>10</sub> esters are typically formed by oxidation of long-chain polyunsaturated FAs (Heinz & Jennings, 1966). During wine processing, these EEs of medium-chain FAs (C<sub>6</sub>–C<sub>12</sub>) are metabolized from the reaction of ethanol and acyl-coenzyme A derived from medium-chain FAs with the assistance of yeast (Cordente et al., 2012). Their contents vary from the effects of yeast strain and aging conditions, and are balanced by the esterification/hydrolysis process (Cejudo-Bastante et al., 2013; Cordente et al., 2012). The higher concentrations of the two esters were detected in dry wines (such as base wine) than in sweet wines (such as sweet fortified wine and icewine) (Bowen & Reynolds, 2012; Lan et al., 2019; Noguerol-Pato et al., 2012). They were detected in the same phenomenon with their precursors, hexanoic acid and octanoic acid, in grape wines (Lan et al., 2019; Noguerol-Pato et al., 2012). Similarly, the concentrations of ethyl hexanoate, ethyl octanoate, and their precursors showed the same trends in GWs with different sweetness.

Ethyl 2-methylbutanoate (**2**) and ethyl 3-methylbutanoate (**3**) also evoked the fruity flavor. Their precursors are known as 2-methylbutanoic acid and 3-methylbutanoic acid. Different from the EEs of straight-chain FAs, these EEs of branch-chain FAs are transformed from the esterification of ethanol and acids. The EEs of branch-chain FAs are also formed from the corresponding amino acids (e.g., isoleucine) or amino acid derivatives (e.g.,  $\alpha$ -ketoacids) by Strecker degradation or biosyntheses pathway in nonoxidative aging (Díaz-Maroto et al., 2005; Matich & Rowan, 2007). The previous investigations demonstrated that these compounds were less abundant in sweet wines (such as botrytized wines and icewines) than in their corresponding dry wines (including base wines or table wines) (Bowen & Reynolds, 2012; Lan et al., 2019; Noguerol-Pato et al., 2012), which is consistent with our study.

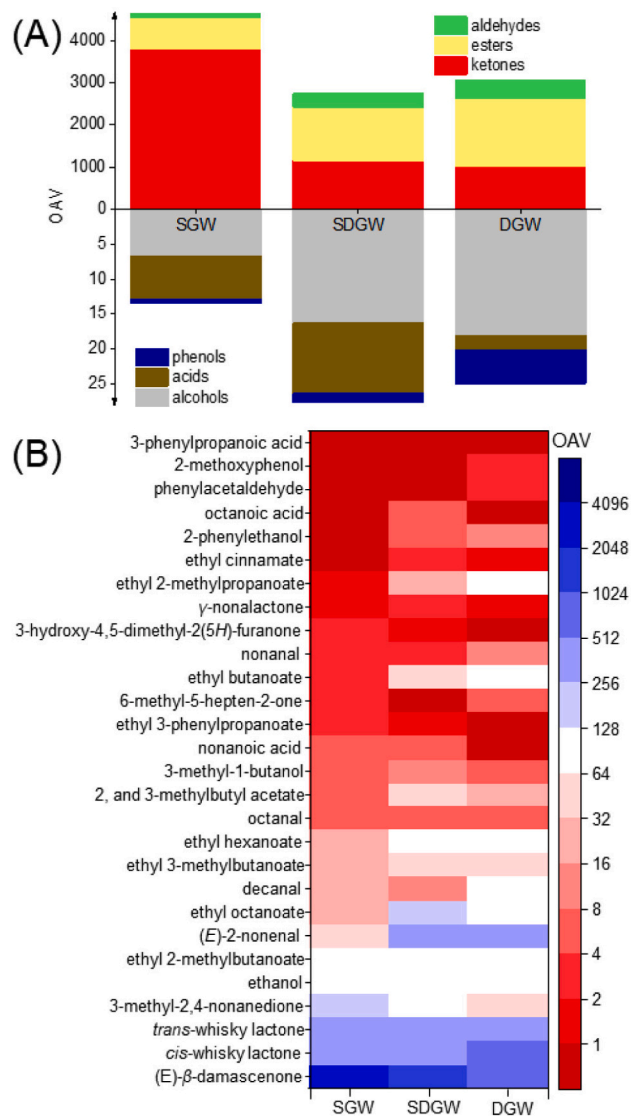
The volatile aldehydes, which increased with the decrease of the sweetness of GWs (Fig. 3a), were reported to be formed from alcohols or

**Table 5**  
Concentrations and odor activity values (OAVs) of odorants in SGW, SDGW, and DGW.

Odorant	Concentration ( $\mu\text{g/L}$ ) <sup>a</sup>			OAV <sup>b</sup>		
	SGW	SDGW	DGW	SGW	SDGW	DGW
( <i>E</i> )- $\beta$ -damascenone	182	52.8	48.8	3600	1100	980
<i>cis</i> -whisky lactone	3.15	3.50	6.93	320	350	690
<i>trans</i> -whisky lactone	2.80	3.99	4.91	280	400	490
3-methyl-2,4-nonanedione	7.75	3.84	1.97	170	83	43
ethanol	$8.8 \times 10^7$	$8.8 \times 10^7$	$9.9 \times 10^7$	89	89	100
ethyl 2-methylbutanoate	69.9	93.0	98.1	70	93	98
( <i>E</i> )-2-nonenal	24.4	176	180	41	290	300
ethyl octanoate	57.9	466	148	29	230	74
decanal	28.1	8.19	106	28	8	120
ethyl 3-methylbutanoate	72.6	116	124	24	39	41
ethyl hexanoate	111	339	334	22	68	67
octanal	21.1	20.9	20.2	6	6	6
2, and 3-methylbutyl acetate	177	985	549	6	33	18
3-methyl-1-butanol	$1.8 \times 10^5$	$2.8 \times 10^5$	$2.0 \times 10^5$	6	9	7
nonanoic acid	130	106	11	5	4	<1
ethyl 3-phenylpropanoate	5.75	3.17	1.25	4	2	<1
6-methyl-5-hepten-2-one	0.57		0.97	4	<1	6
ethyl butanoate	62.0	723	$1.4 \times 10^3$	3	36	69
nonanal	2.59	3.75	11.3	3	4	11
3-hydroxy-4,5-dimethyl-2(5 <i>H</i> )-furanone	12.5	8.05	2.78	3	2	<1
$\gamma$ -nonalactone	9.47	24.2	7.91	1	3	1
ethyl 2-methylpropanoate	15.5	459	974	1	31	65
ethyl cinnamate	0.93	3.23	1.44	<1	3	1
2-phenylethanol	$8.4 \times 10^3$	$5.6 \times 10^4$	$8.2 \times 10^4$	<1	6	8
octanoic acid	391	$2.5 \times 10^3$	428	<1	5	<1
phenylacetaldehyde	0.59	0.69	2.54	<1	<1	3
2-methoxyphenol	4.03	7.60	23.8	<1	<1	2
3-phenylpropanoic acid	14.5	22.9	6.12	<1	<1	<1
2-methoxy-4-vinylphenol	4.06	8.08	17.74	<1	<1	<1
eugenol	0.47	1.37	9.20	<1	<1	2
acetic acid	$1.5 \times 10^4$	$6.4 \times 10^4$	$7.0 \times 10^4$	<1	<1	<1
ethyl propanoate	221	753	563	<1	<1	<1
<i>p</i> -cresol	0.30	0.83	0.65	<1	<1	<1
vanillin	5.80	9.01	7.38	<1	<1	<1
butanoic acid	285	$1.4 \times 10^3$	754	<1	<1	<1
<i>m</i> -cresol	1.48	1.54	1.83	<1	<1	<1
2, and 3-methylbutanoic acid	51.2	543	616	<1	<1	<1
methionol	8.40	688	$1.3 \times 10^3$	<1	1	3
$\alpha$ -terpineol	3.82	5.00	5.48	<1	<1	<1
acetophenone	7.16	7.77	6.07	<1	<1	<1
4-hydroxy-2,5-dimethyl-3(2 <i>H</i> )-furanone	6.78	20.6	16.5	<1	<1	<1
decanoic acid	188	725	399	<1	<1	<1
hexanoic acid	33.5	172	158	<1	<1	<1
2-methylpropyl acetate	15.4	78.3	50.2	<1	<1	<1
ethyl 2-phenylacetate	6.20	12.4	8.64	<1	<1	<1
heptanoic acid	12.3	9.86	6.58	<1	<1	<1
2-phenylethyl acetate	1.50	174	681	<1	<1	3
pentanoic acid	57.2	37.1	26.6	<1	<1	<1
( <i>E</i> )-3-hexen-1-ol	0.97			<1	<1	<1
phenylacetic acid	3.80	4.05	3.62	<1	<1	<1
linalool		0.83	4.42	<1	<1	<1

<sup>a</sup> The concentrations were calculated by the mean concentration in 2–4 samples with relative standard deviations <15%.

<sup>b</sup> Odor activity values, calculated according to the measured concentrations in GWs and the orthonasal odor thresholds (see Supporting Information, Table S2).



**Fig. 3.** The OAVs of key odorants in GWs. The data showed (A) according to the structural groups or (B) individually.

other precursors by oxidation degradation that this kind of (*E*)-2-alkenals was detected in large amounts in wines exposed to oxygen (Culleré et al., 2007), such as during aged-in-barrel processing. Unsaturated FAs, such as linoleic acid, are also introduced as the precursor of (*E*)-2-nonenal (23) during the lipid oxidation pathway (Chatonnet & Dubourdieu, 1998). (*E*)-2-Nonenal is widely found in wines at level above threshold (Culleré et al., 2007) and causes the “sawdust” character flavor (Chatonnet & Dubourdieu, 1998). SDGW and DGW contained higher (*E*)-2-nonenal content than SGW in our study, which is consistent with the APA results.

During wine processing, the natural sugar was consumed and transformed into ethanol and carbon dioxide, and the end products were mostly dry wines. The common ways for elevating sugar content to produce sweet wines were (i) using the raw materials that contained less water but higher sugar content, such as overripe or dehydrated fruits (naturally sweet wines), (ii) adding additional ethanol to avoid or end

the fermentation and further keep the final sugar content (fortified or liquor wines) (Reboredo-Rodríguez et al., 2015). The higher contents of (*E*)- $\beta$ -damascenone, 3-methyl-2,4-nanonedione, and lower esters contents in SGW might indicate that more raw materials were used/consumed. The possible processing to gain SGW was using overripe or dehydrated raw materials or adding goji syrup, which could be confirmed by the brown color in SGW (Reboredo-Rodríguez et al., 2015). While the higher contents of odorants, such as whisky lactones and (*E*)-2-nonenal, in DGW might be resulted from a more complete sugar consumption or longer stored-in-barrel time in processing DGW.

#### 4. Conclusion

Numerous factors affect the final overall aroma profile of GWs. One of the factors is the sweetness. A total of 71 odorants were characterized in three GWs, including esters, ketones, phenols, and aldehydes, and 35 of which were never been previously reported in goji wines. With OAV  $\geq 1$ , (*E*)- $\beta$ -damascenone, whisky lactones, and (*E*)-2-nonenal were assigned as the key odorants, and cooked apple-like odor impression was the predominant among the three sweetness levels of GWs. The differences were elucidated among SGW, SDGW, and DGW, and DGW showed a more intense malty and fatty odor impression, represented by the alcohols and aldehydes.

In our study, even though the sweetness was different among these three GWs, many odorants and the key odorants were in common. The odorant differences with different sweetness levels of GWs were not significant on the compositions, but mainly on the concentrations. Although the key odorants in GWs were impacted by the sweetness, raw materials, and fermented conditions, which often remain a trade secret, the detailed analysis of aroma profiles and the common odorants among different sweetness levels of GWs in our study can provide a long-time reference value on GWs flavor and be applicable to other wines with different sweetness. Studies on the raw materials and the fresh knowledge for improving flavor quality and developing diverse goji products will be continued in more detail.

#### CRedit authorship contribution statement

**Yan Zheng:** Writing – original draft, Visualization, Methodology, Investigation. **Claudia Oellig:** Writing – review & editing, Conceptualization. **Youfeng Zhang:** Writing – review & editing, Methodology. **Yuan Liu:** Writing – review & editing. **Yanping Chen:** Writing – review & editing. **Yanyan Zhang:** Writing – review & editing, Supervision, Resources, Conceptualization.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

Data will be made available on request.

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#### Appendix A. Supplementary data

The supporting information (pdf.) provided the detailed determination of the concentrations of ethanol and sugar contents, and the odor threshold information of the used odorants.

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