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Volatile flavor compounds, total polyphenolic contents and antioxidant activities of a China gingko wine

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ABSTRACT

The volatile compounds in gingko wine, a novel functional wine, were extracted by head-space solid phase micro-extraction (SPME) and analyzed by gas chromatography–mass spectrometry (GC–MS) coupled with odor activity value (OAV) and relative odor contribution (ROC) analyses. In addition, the total polyphenolic content of gingko wine was determined using the Folin–Ciocalteu reagent, and its antioxidant capacity was evaluated by 1,1-diphenyl-2-picrylhydrazyl (DPPH) and 2,2′-azinobis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) assays. Fifty-eight compounds were tentatively identified, including 13 esters, 10 alcohols, 11 acids, 12 carbonyl compounds, 2 lactones, 2 phenols, and 8 hydrocarbons. Ethyl hexanoate, ethyl pentanoate, nonanal, ethyl butyrate and ethyl heptanoate were the major contributors to the gingko wine aroma based on the results of OAV and ROC. The total phenols content of the gingko wine was 456 mg/L gallic acid equivalents, and its antioxidant capacity was higher than those of typical Chinese liquors analyzed in this paper.

1. Introduction

Gingko (*Gingko biloba* L.), an ancient plant, is well-known for the *G. biloba* extract, which can be extracted from its leaves and fruits. It contains high amounts of flavonoids and terpene lactones. Nowadays, gingko nuts and leaves have been used as liquor-making raw materials, to produce "gingko wine". In general, gingko nuts and leaves are initially milled and mixed with other raw materials. The production process of gingko wine follows typical manufacturing procedures for Chinese liquors, including fermentation, distillation and blending (Xiao et al., 2014).

It is well-recognized that wine aroma can greatly affect wine quality and consumer evaluation. There are numerous means to isolate and enrich the aroma compounds of wine, such as simultaneous distillation extraction (SDE) (Blanch, Reglero, & Herraiz, 1996; Bosch-Fusté et al., 2007), solvent extraction (Pino & Queris, 2011), head space-solid phase micro-extraction (HS-SPME) (Xiao et al., 2014). However, only a few fractions of wine volatiles contribute to the fragrance of a wine. Thus, it is very important to select the isolation method that can reflect the release of the significant volatile compounds from the matrix rather than determining the overall contents of those components of wine (Plutowska & Wardencki, 2008). Considering all of the above-mentioned factors, HS-SPME was selected since this approach can quickly, simply and accurately assess the essential volatile compounds from wine, with infinitesimal traces of the sample (Sagratini et al., 2012). Odor activity value (OAV) and relative odor contribution (ROC) are two common indicators for the evaluation of the contribution of volatile compounds to the overall aroma of the sample. ROC refers to the ratio of the OAV percentage of each compound and the sum of the OAV of compounds that showed OAV > 1 (Welke, Zanus, Lazzarotto, & Alcaraz Zini, 2014).

Free radicals containing unpaired electrons, are prone to actively react with biomolecules (DNA, lipid, et al.) and cause cellular injury and death (Hsu, Zhang, Peng, Travas-Sejdic, & Kilmartin, 2008). Researchers have paid much attention to the human health issues caused by free radicals. Many studies have focused on the free radical scavenging capacity of red wines, due to the presence of polyphenolic compounds (Cimino, Sulfaro, Trombetta, Saija, & Tomaino, 2007; Li, Wang, Li, Li, & Wang, 2009; Paixão, Perestrelo, Marques, & Câmara, 2007). To our best knowledge, the aroma profiles and free radical scavenging capacity of gingko wine have not been reported previously in the literature. Hence, the aim of this

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work was to investigate the gingko wine volatile flavor compounds, total polyphenolic contents and its free radical scavenging capacities. This work may provide useful information to the gingko wine industry to evaluate quality and potential consumer acceptability.

2. Materials and methods

2.1. Wine samples and chemicals

Gingko wine was provided by a domestic winery which was manufactured in 2013. The alcoholic content was 12% (v/v). The five typical Chinese liquors: *Luzhou Laojiao*, *Xinghuacun*, *Maotai wangzi*, *Xifengjiu*, *Haizhilan*, were purchased from the local market.

2-Octanol (internal standard) was obtained from Dr. Ehrenstorfer (Augsburg, Germany). *n*-Alkane standard (C7-C30) was purchased from Supelco (Bellefonte, PA, USA). Folin–Ciocalteu reagent, 1,1 diphenyl-2-picryl-hydrazyl (DPPH), 2, 2′-azinobis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS), potassium persulfate, gallic acid and trolox were obtained from Sigma–Aldrich (St. Louis, MO, USA). All reagents were of analytical grade.

2.2. Extraction of volatile compounds

A 75 μm CAR/PDMS fiber (Supelco, Bellefonte, PA, USA) was used to extract the volatile compounds in gingko wine. 5 mL of gingko wine was placed into a 15 mL vial, and was saturated with sodium chloride. Then, 50 μL of 2-octanol (400 mg/L in absolute ethanol) was added into the wine sample as internal standard. The vial was tightly capped with a silicon septum. The fiber was exposed in the headspace of the vial for 45 min at 50 °C. Subsequently, the fiber was inserted into the gas chromatography (GC) injection port for 4 min at 250 °C to desorb the analytes in splitless mode. Each sample was analyzed in triplicate.

2.3. Gas chromatography–mass spectrometry (GC–MS) analysis

The extracted compounds were separated and identified by a 7890 gas chromatography coupled with a 5975C mass selective detector (MS) (Agilent Technologies, USA) equipped with a HP-INNOWAX capillary column (60 m × 0.25 mm ID, 0.25 μm film thickness), or a DB-5 MS column (60 m × 0.25 mm ID, 0.25 μm film thickness). The carrier gas (He) flow rate was 1 mL/min. The temperature of the injector was 250 °C. The oven temperature was 50 °C for 2 min (HP-INNOWAX) or 4 min (DB-5 MS), then rose to 230 °C at 6 °C/min (HP-INNOWAX) or 4 °C/min (DB-5 MS) and was held for 10 min. The electron impact (EI) ion energy was 70 eV, and the chromatograms were obtained by recording the total ion currents within 30–450 *m*/*z*.

Tentative identification of the volatile compounds was achieved by comparing mass spectrum and retention indices (RI) with the Wiley7n.l Database (Hewlett–Packard, Palo Alto, CA) and Nist05a.l Database and previously reported RI. Some compounds were identified by the injection of the authentic compounds into the GC–MS system, while the RI of the compounds was calculated using an *n*-alkane series under the same conditions according to van den Dool and Kratz equation (van Den Dool & Dec. Kratz, 1963). Semiquantitative determinations were performed according to the reference (Xiao et al., 2014).

2.4. Odor activity value (OAV) and relative odor contribution (ROC)

OAV of volatile compound was the ratio of the content of each compound to its detection threshold concentration (González Álvarez, González-Barreiro, Cancho-Grande, & Simal-Gándara, 2011). ROC was calculated through dividing the OAV of each compound by the sum of the OAV of compounds (OAV > 1).

2.5. Determination of total polyphenolic content

The content of total polyphenolic compounds in gingko wine and the typical Chinese liquors was determined using the Folin–Ciocalteu reagent (Paixão et al., 2007; Zhang et al., 2010). Briefly, 1 mL of wine sample was put into a 15 mL test tube containing 5 mL of deionized water. 1 mL of Folin–Ciocalteu reagent and 3 mL of 20% Na_2CO_3 solution was added in the tube and mixed thoroughly. After standing in dark place at room temperature for 30 min, the absorbance of the mixture was measured at 760 nm using a 7200 spectrophotometer (UNICO (Shanghai) Instruments Co., Ltd). All results were compared to the standard curve of gallic acid which was prepared at a concentration range from 0 to 500 mg/L and expressed as gallic acid equivalent (mg GAE/L wine). Each sample was performed in triplicate.

2.6. DPPH and ABTS free radical scavenging capacity

For evaluating the DPPH free radical (DPPH·) scavenging activity, the well-established method was used, based on Li et al. (2009) with slight modifications. The DPPH· stock solution was prepared by dissolving 20 mg of DPPH· in 50 mL of ethanol, and then 50 mL of deionized water was added. The DPPH· working solution was obtained by diluting stock solution with water/ethanol (50/50, v/v) mixture to an absorbance of 0.75–0.80 at 515 nm. 1 mL of sample was added into 9 mL of DPPH· working solution. After reaction in dark place for 30 min, the absorbance at 515 nm was measured. The absorbance of 1 mL of water/ethanol (50/50, v/v) mixture under the same condition was regarded as the control.

The method described by Re et al. (1999) was adopted with slight modifications to determine the ABTS free radical ($ABTS^{\cdot+}$) scavenging capacity. The $ABTS^{\cdot+}$ stock solution was prepared by reacting ABTS solution (7 mmol/L) with potassium persulfate (2.45 mmol/L) solution in dark place at room temperature for 12–16 h before use. The $ABTS^{\cdot+}$ working solution was prepared by diluting stock solution with water/ethanol (50/50, v/v) mixture to an absorbance of 0.75–0.80 at 734 nm. A 1 mL of sample was added into 9 mL of $ABTS^{\cdot+}$ working solution. The absorbance of the mixed solution at 734 nm was recorded after 6 min in the dark.

The inhibition percentage of DPPH· ($ABTS^{\cdot+}$) was calculated as follow:

$$\text{Inhibition}_{\text{sample}}\ (\%) = (\text{Abs}_{\text{control}} - \text{Abs}_{\text{sample}})/\text{Abs}_{\text{control}} \times 100$$

where $\text{Abs}_{\text{control}}$ is the absorbance of the control sample, and $\text{Abs}_{\text{sample}}$ is the absorbance of the sample.

All the results for the DPPH· ($ABTS^{\cdot+}$) scavenging capacity of wine samples were expressed as trolox equivalent antioxidant capacity (TEAC). The standard solutions of trolox ranged from 0 to 400 mg/L. The trolox standard curve was built by plotting inhibition percentage versus trolox concentration at 515 nm (734 nm) for DPPH (ABTS) assays. Every sample was analyzed in triplicate.

2.7. Statistical analysis

Experimental results were means ± SD of replicates. The antioxidant capacity analysis was performed by analysis of variance (ANOVA) using SAS V8 (SAS Institute Inc., Cary, NC).