



Flavor and chiral stability of lemon-flavored hard tea during storage



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ABSTRACT

Flavor stability of hard tea beverage was investigated over eight weeks of storage. The volatile compounds were analyzed using solid-phase microextraction-gas chromatography-mass spectrometry (SPME-GC–MS) and two-dimensional GC–MS. Quantitative analysis showed that the concentrations of linalool, citronellol, geranial, neral, geraniol, and nerol decreased dramatically during storage, whereas α -terpineol showed an increasing trend during storage. Heart-cut two-dimensional GC–MS (2D-GC–MS) chirality analysis showed that (*R*)-(+)-limonene, (*R*)-(–)-linalool, (*S*)-(–)- α -terpineol and (*S*)-(–)-4-terpineol dominated in the fresh hard tea samples, however, the configuration changed during storage for the terpene alcohols. The storage conditions did not change the configuration of limonene. A conversion of (*R*)-(–)-linalool to (*S*)-(+)-form was observed during storage. Both (*S*)- α -terpineol and (*S*)-4-terpineol dominated at beginning of the storage, but (*R*)-(+)- α -terpineol became dominated after storage, suggested in addition to isomerization from (*S*)- α -terpineol, other precursors could also generate α -terpineol with (*R*)-isomer preference.

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1. Introduction

Hard iced tea is a malted ready-to-drink (RTD) tea beverage containing about 5% alcohol. Hard tea is less studied compared to other alcoholic beverages such as beer, wine, and distilled spirits. Hard iced tea is typically formulated with tea extract (Yuan, He, Qian, Zheng, & Qian, 2016). Both green tea extract and black tea extract are commonly used for the manufacturing of hard tea. Hard tea aroma varies greatly and is largely dependent on the tea extract used in the formulation. Green tea aroma is complex, both the aroma and aroma precursors are affected by tea variety and growing conditions (Ho, Zheng, & Li, 2015). The aromas of semi-fermented tea (oolong tea) and fermented tea (black tea) are even more complex, as many glycoside bounded aroma compounds in fresh tea leaves are released by endogenous glycosidases during the manufacturing processes (Wang, Yoshimura, Kubota, & Kobayashi, 2000). Linalool was reported to play an important role in the quality of green teas (Kato & Shibamoto, 2001). α -Terpineol is reported to be important in black tea (Alasalvar et al., 2012).

Hard tea is typically flavored with artificial or natural flavorings. Lemon is the most popular flavor among flavored iced tea beverages. It has been reported that the key aroma compounds contributing to lemon flavor are terpenoids such as linalool, citral, neral, geranial and citronellol (Schieberle & Grosch, 1988).

Flavor instability is a long existing concern in RTD beverage because of the decomposition of the flavor compounds, especially at a low-pH environment and under oxidative stress. RTD tea beverages are acidic and therefore highly susceptible to flavor loss during storage due to the acid-catalyzed reactions. It has been reported that citral decomposes rapidly at low pH via cyclization and oxidation reactions (Piorkowski & McClements, 2014), and resulting in loss of the lemon-like aroma, as well as generating other undesired off-flavors (Choi, Decker, Henson, Popplewell, & McClements, 2009). Limonene is the most abundant components in lemon oil, and is also very susceptible to oxidation. Limonene oxidation leads to the loss of lemon-like odors and the formation of compounds imparting flowery, piney, and minty off-flavor (Djordjevic, Cercaci, Alamed, McClements, & Decker, 2007). Other components in the tea can also influence the aroma stability. Free amino acids have been reported responsible for the loss of α , β -unsaturated carbonyl aroma compounds (i.e. 1-octen-3-one and geranial) in Longjing green tea by the formation of 2-butyl-2-octenal upon storage at 50 °C (Cheng, Huynh-Ba, Blank, & Robert, 2008).

There has been growing interest in enantiomeric compositions of flavor compounds in foods. Enantiomer purity has been used to evaluate adulteration of food products or to differentiate natural compounds from synthetic origin (Ruiz del Castillo, Caja, & Herraiz, 2003). Individual enantiomer of chiral compound usually has different flavor characteristics and odor threshold. It is known that the odor threshold of (*R*)-(–)-linalool (woody, lavender-like) is

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nearly 9 times lower than (*S*)-(+)-linalool (sweet, floral-like) (0.8 vs 7.4 ppb) (Barba, Flores, & Herraiz, 2010). Linalool enantiomer distribution has been studied in coffee (Bonnländer, Cappuccio, Liverani, & Winterhalter, 2006), beer (Steinhaus, Fritsch, & Schieberle, 2003), honey (Wilkins, Lu, & Tan, 1993), fruit juices (Jordán, Margaría, Shaw, & Goodner, 2003) and hard ice tea (Yuan et al., 2016).

The aromas of fresh and aged hard tea have been investigated by aroma extract dilution analysis (AEDA) (Yuan et al., 2016), and it was observed that the flavor dilution value changed substantially in the aged samples, particularly for terpenoids. Therefore, the purpose of this study was to further investigate the stability of key aroma compounds in hard iced tea, especially terpenoids that are mainly responsible for lemon-flavor, under different storage conditions. The enantiomeric transformation of important aroma compounds during storage was further investigated by two-dimensional GC–MS.

2. Materials and methods

2.1. Tea samples

Two types of RTD hard iced tea (pH 3.2) containing 5% alcohol by volume with tea extract and flavoring were obtained commercially. Sample 1 is formulated with tea extract and natural lemon flavoring, and sample 2 is formulated with tea extract, natural lemon flavoring and lemonade. Tea samples were aged at four different temperatures (room temperature, 35 °C, 40 °C, 45 °C) and one bottle from each storage temperature was sampled each week up to 8 weeks and kept at 4 °C before analysis.

2.2. Chemicals

Methanol (HPLC grade) was from EM Science (Gibbstown, NJ, U.S.A.). Geranyl acetate (90%), and α -terpinene were purchased from TCI America (Cambridge, MA, U.S.A.). Ethyl octanoate were purchased from Eastman (Rochester, NY, U.S.A.). Ethyl butanoate (>98%), 3-methylbutyl acetate (isoamyl acetate, >95%), octyl acetate (98%), ethyl hexanoate (>98%), ethyl decanoate (>98%), phenethyl acetate (>98%), isoamyl alcohol (>98%), 1-octanol, phenethyl alcohol (99.0%), eucalyptol (>99%), *p*-cymene, α -terpineol (90.0%, mixture of enantiomers), terpinen-4-ol, linalool, limonene, geranial, neral, β -citronellol (95.0%), geraniol (98.0%), nerol (98.0%), β -damascenone (\geq 90.0%), β -ionone (90.0%), (*S*)-(-)-limonene (96%), (*R*)-(+)-limonene (97%) were purchased from Sigma-Aldrich (St. Louis, MO, U.S.A.). (*R*)-(-)-linalool (\geq 95%) was purchased from Fluka (Buchs, Switzerland). Citric acid was purchased from J.T. Baker (Center Valley, PA, U.S.A.). Sodium chloride (ACS grade) was purchased from EMD Millipore (Billerica, MA, U.S.A.). Pure water was obtained from a Milli-Q purification system (Millipore, Bedford, MA, U.S.A.) All standard stock solutions were prepared in methanol individually and then mixed to make the standard solution.

2.3. Volatile compounds quantification by solid-phase microextraction (SPME)-GC–MS

Volatile compounds were quantitated using solid phase microextraction (SPME)-GC–MS (Fan & Qian, 2005) with minor modification. Volatile compounds in tea samples were extracted using a 2 cm, 23-gauge SPME fiber coated with 50/30 μ m divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) (Supelco Co., Bellefonte, PA, U.S.A.). Fibers were conditioned prior to use according to the manufacturer's recommendations. Two milliliter of iced tea sample was mixed with 8 mL of 0.1 M citric acid buffer (pH 2.5,

saturated with NaCl) in a 20 mL glass vial, and 20 μ L of 34 mg/L 4-octanol was added as internal standard. Samples were incubated at 50 °C for 5 min, with an agitation speed of 250 rpm. After equilibration, the SPME fiber was exposed to the headspace for 50 min at the same agitation speed and temperature. The SPME fiber was then injected to the GC at 250 °C for 4 min in splitless mode.

The analysis of volatile compounds was carried out using an Agilent 6890 GC equipped with a 5973 mass selective detector (MSD) (Agilent Technology, Palo Alto, CA, U.S.A.), a Gerstel MPS multipurpose autosampler (Gerstel, Linticum, MD, U.S.A.). The carrier gas was helium at a constant flow rate of 1.2 mL/min. A DB-wax column (30 m \times 0.25 mm i.d., 0.5 μ m film thickness, Agilent Technology) was used for separation. The oven temperature was programmed at 60 °C for a 2 min holding, then increased to 220 °C at a rate of 3 °C/min with 5 min holding. The MS transfer line and ion source temperature were 280 and 230 °C, respectively. Electro ionization mass spectrometry data from *m/z* 35–350 were collected using a scan rate of 5.27/s, with an ionization voltage of 70 eV. The volatile compounds were identified by comparing their mass spectra with those in the Wiley 275.L Database (Agilent Technologies Inc.) and retention indices (RIs) with those of authentic standards available in the laboratory using the same instrument. RIs were calculated after analyzing C6–C20 *n*-alkane series (Supelco, Bellefonte, PA, U.S.A.) under the same chromatographic conditions. Triplicate analysis was performed on each sample.

Calibration curves were built up for quantitative analysis of selected volatile compounds. Individual standard stock solution was diluted in methanol to make the first level mixed standard solution, and then diluted with methanol to a serial concentration. A synthetic tea solution was prepared using 0.25 g/L citric acid solution saturated with NaCl, and pH was adjusted to 3.5 with 1 M NaOH. A set of synthetic tea (10 mL) containing 5 different concentrations of standard mixture solution was placed into 20 mL vial with Teflon-faced silicon septa to build calibration curve. Twenty microliter of 35 mg/L 4-octanol was added to each standard solution as internal standard. Standards and the internal standard were extracted with the SPME fiber and analyzed using the same GC–MS condition as described previously for sample analysis. Selective mass ion was used to quantify the volatile compounds. The calibration curves were built up and calculated through GC/MSD ChemStation software (Agilent Technologies).

2.4. Chiral analysis by two-dimensional GC–MS

The samples were extracted using the same procedure as described previously. Volatile compounds were first separated on a DB-WAX column (30 m \times 0.25 mm i.d., 0.5 μ m film thickness, Agilent Technologies), then the targeted compounds were heart-cutted and on-line transferred to a Cyclosil-B column (30 m \times 0.25 mm i.d., 0.25 μ m film thickness, Agilent Technologies) for further separation. The first column oven temperature was programmed at 80 °C for a 2 min hold and then increased to 230 °C at a rate of 4 °C/min, with a 20 min hold at the final temperature. The second column oven temperature was programmed at 80 °C for a 20 min hold and then increased to 150 °C at a rate of 2 °C/min, with a 3 min hold at the final temperature. The “heart-cut” window was set as 9.5–10.5 min, 19–22 min, 23–24.5 min and 26–30 min for cutting limonene, linalool, 4-terpineol and α -terpineol respectively. In addition, a cutting interval of 15–16 min was chosen to cut internal standard. The *R*- and *S*-enantiomers of limonene, linalool and terpinen-4-ol were identified by comparing retention time with authentic standards. α -Terpineol enantiomers were identified by comparing with published elution order from the same column (Martin & Bohlmann, 2004). The isomeric ratio was determined using the relative total

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