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Research Article

Discrimination of Laoshan Green Tea with Ultrasound Processing Method by SPME-GC-MS of the Aromatic Composition

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Abstract: This study suggests that the ultrasound processing method for Laoshan green tea affects the final flavor quality of the product. The results indicated that the 75 μm Carboxen/PDMS was the most effective for aromatic components extraction of Laoshan green tea. The aroma of ultrasound processing tea and control was compared by both SPME-GC/MS analysis and sensory evaluation. It was found that ultrasound processing tea and control shared similar aroma component species, but differed in their contents. Organoleptic evaluation indicated that the sensory quality of tea infusion with ultrasound processing method was better than that of tea infusion with conventional extraction.

Keywords: Aromatic composition, Laoshan green tea, SPME-GC-MS, ultrasound processing method

INTRODUCTION

Tea was known as the most widely popular beverage in the world, for its wide beneficial effects to humans' health. And their pharmaceutical and industrial applications are also in development (Someswararao and Srivastav, 2012). It has been said that drinking green tea brings relaxation because of L-theanine, that is one of the stimulant neurotransmitters in brain. Furthermore, some studies suggest that theanine may be clinically useful to preventing Parkinson's disease symptoms (Boudaoud and Eveleigh, 2003).

Laoshan green tea produced in Qingdao, is the highest latitude tea tree planting areas in China. Region according to mountain alongside sea (Quanzhen *et al*., 2011; Gao *et al*., 2011), the unique climate and geological conditions of given laoshan green tea excellent quality (Ruizhan *et al*., 2012; Farid *et al*., 2011), is known as "the first famous tea in the north". The traditional tea products form is simple, in recent years a variety of new technology were used for deep processing of tea, promoting industrial added value, such as ultrasonic, microwave (Pan *et al*., 2003; Tao and Sun, 2015; Zhou *et al*., 2015) enzymolysis and so on. It has been said that the improvement of solvent extraction from plant by ultrasound is due mainly to the effects of acoustic cavitation, which enhances solvent penetration into the plant material and the intracellular product release by disrupting the cell walls (Mason *et al*., 1996).

The quality of the tea flavor compounds depends on sensory evaluation and aromatic components analysis. At the same time distillation and SDE method were commonly used for aroma substances extraction, but the aroma composition loss. The aroma of tea material is mainly of volatile and semi volatile compounds, the analysis depends on appropriate extraction adsorption and analysis methods. Solid Phase Micro Extraction (SPME) is put forward by Pawliszyn technology in 1989, without complicated sample preparation, fast and convenient (Sun *et al*., 2015), can be directly on the GC and GC-MS and HPLC analysis, has become internationally recognized detection method suitable for tea aroma matter. The factors affecting the effects of SPME extraction is more, the most important is extracted first choice. Different extraction head coated different chromatographic stationary phase, according to the analysis of molecular weight and polarity selection of objects. In addition, should also be designed according to different objects of different reaction system, design the appropriate conditions of GC-MS, including parameter setting, temperature program of exploration, etc., (Nwaneshiudu *et al*., 2014; Wang *et al*., 2015).

However, no report has been done on the effect of ultrasound processing method on the aromatic components analysis of Laoshan green tea. In this study, the aroma of ultrasound processing tea and conventional extraction as control was compared by both SPME-GC-MS analysis and sensory evaluation.

MATERIALS AND METHODS

Materials: Laoshan green tea is from the Qingdao Xiao Yangchun company.

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Instruments: Extraction bottle, 65 μm PDMS/DVB, 100 μm PDMS, 75 μm Carboxen/PDMS fibers (Supelco company, USA), constant temperature magnetic stirrer (PC-420), Agilent GC 6890 n-5973 Mass Selective Detector (MSD), ultrasonic cleaning bath (40 kHz, 250 W).

The sample processing method: Six gram tea powder sample, to join the extraction bottle, built-in magnetic stirring rotor, with 60 mL 100°C hot water (containing sodium chloride concentration is 0.32 g/mL) brewing, airtight bottle, prevent volatile component spillover, balance at 60°C constant temperature water bath for 5 min. Then insert the solid phase micro extraction, open temperature 60°C magnetic stirring device, headspace extraction for 60 min (Dilek *et al*., 2012). The experimental ultrasonic processing at the same time, power 150 w, 40 min. Micro-extraction head out immediately after insert chromatograph injection port, parsing 3.5 min, for data collection and analysis.

SPME and GC analysis: After SPME fibre screening, 75 μm fiber was carried out in order to select the best type in terms of extraction efficiency and reproducibility.

GC conditions: HP-5 ms quartz capillary column (30 m*0.25 mm*0.25 microns). Injection port temperature 250°C, ECD detector temperature 250°C; Carrier gas for high purity helium, purity>99.999%, the flow rate of 1 mL/min; Starting column temperature is 50°C, keep 5 min, at the rate of per min 6 to 160°C, keep 3 min, then to 20°C/min to 230°C, don't tap into the sample. The EI MS conditions: ion source; Ion source temperature 230°C; 70 eV electron energy; Emission current of 100 mu; Quadrupole temperature 150°C; Turn the interface temperature: 280°C; Electron multiplier voltage 350 V. Ainu quality scan range: 50-550.

Data analysis: GC-MS data collected by the NIST98. L standard spectral library retrieval, by mass spectrometry materials, refer to the literature (Pukale *et al*., 2015), analysis the base peak, mass ratio, relative abundance, etc., determine the components of each peak, peak area percentage of total peak of components in relative content. The repeat 3 times, average.

RESULTS AND DISCUSSION

SPME fibre screening: Sixty five μm PDMS/DVB suit for volatile and amine, extraction of nitro aromatic compounds, 75 μm Carboxen/PDMS suit for VOC organic volatile sulfur compounds, 100 μm PDMS is suitable for the extraction of volatile and semi volatile compounds. By using three different extraction head to aroma composition analysis of laoshan green tea, 75 μm fiber was carried out in order to select the best type in terms of extraction efficiency and reproducibility.

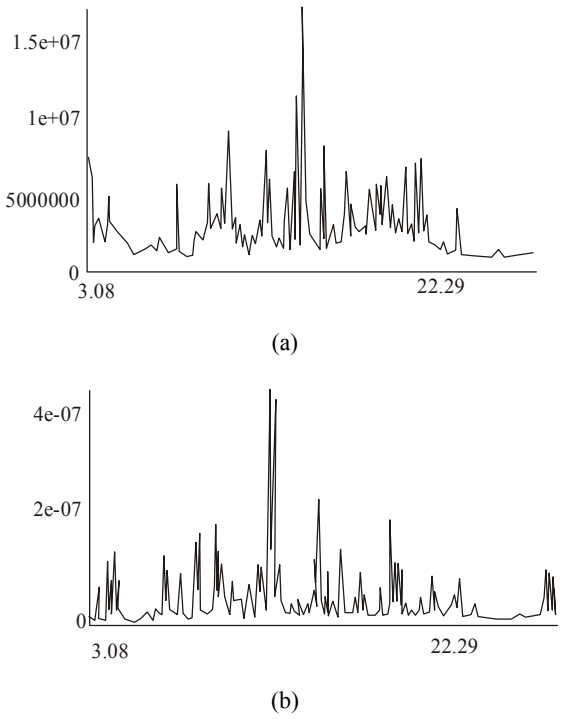

Fig. 1: The ion chromatogram of control and ultrasonic sample (a) control, (b) ultrasonic sample

Characteristic flavor substance: Figure 1 shows the ion flow chart of control and ultrasonic sample. The 75 μm Carboxen/PDMS solid-phase micro-extraction head detected 102 kinds of laoshan green tea aroma substances, while the ultrasonic processing sample 117 kinds. The flavor type is similar, but the ultrasonic processing samples have more flavor substances.

Contrast with the ultrasonic treatment sample and the control, the same aroma components has 71 kinds: Cyclohexene (Flowers light incense), 1, 6-Octadien-3-ol (Rose fragrance), Hexanoic acid, 3-hexenyl ester (Diffuse pear fragrance), Naphthalene, 1, 2, 3, 4-tetrahydro-1, 6-dimethyl-4- (1-methylethyl)-, (1S-cis) - (sweet), Limonene (Fresh citrus), 3-Buten-2-one, 4- (2, 6, 6-trimethyl-1-cyclohexen-1-yl)-, (E) -beta- (Violet sweet), 3-Penten-2-one, 4-methyl- (Honey taste), 3-Cyclohexen-1-carboxaldehyde, 3, 4-dimethyl- (Strong green grass and sweet breath), Phenylethyl Alcohol (Rose fragrance), alpha.-Cubebene (Spicy, herbal), Hexanoic acid, hexyl ester (With green wax is sweet, sweet, herbs, sweet and tropical fruits, berries like nuances), 3-Buten-2-one, 4- (2, 6, 6-trimethyl-2-cyclohexen-1-yl) - (The sweet fragrance of flowers), 5, 9-Undecadien-2-one, 6, 10-dimethyl-, (E) - (fruity), Nerolidol (Rose and apple aromas), etc.

The unique elements of ultrasonic treatment sample are as follow: Cyclobutanol, 4-Pentenal, 2-methylene-, 2-Ethyl-3-vinyloxirane, 1-Octene, 2-Butyn-1-ol, 2, 3, 4-Trimethyl pyrrole, Oxirane, ethenyl-, Chloroacetic acid propyl ester, 2-Pentene, 5-(pentyloxy)-, (E)-, 7-Octen-2-one, Furan, 2-pentyl-, 1-Methyl-3-nitro-1 H-pyridin-2-one, D-Limonene,

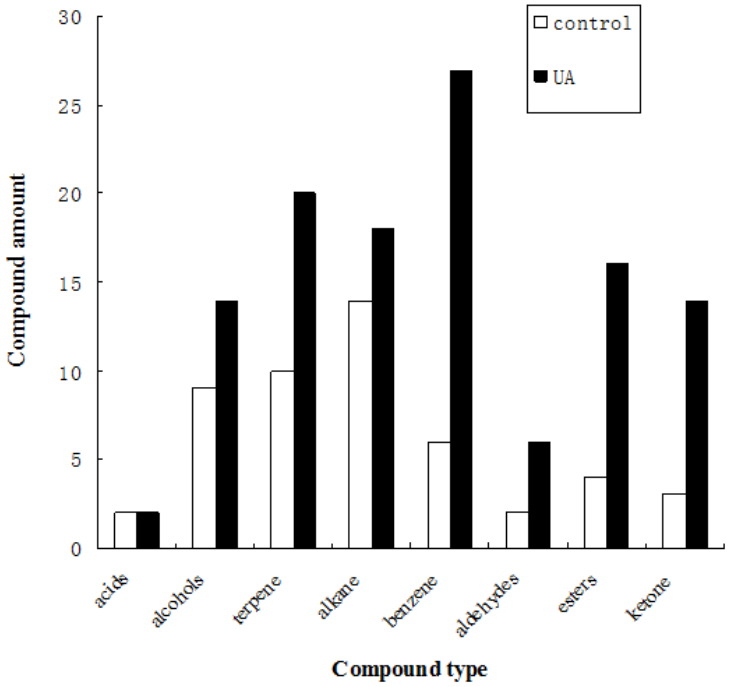

Fig. 2: Comparison of compound amount of aroma components identified in conventional extraction and ultrasonic sample

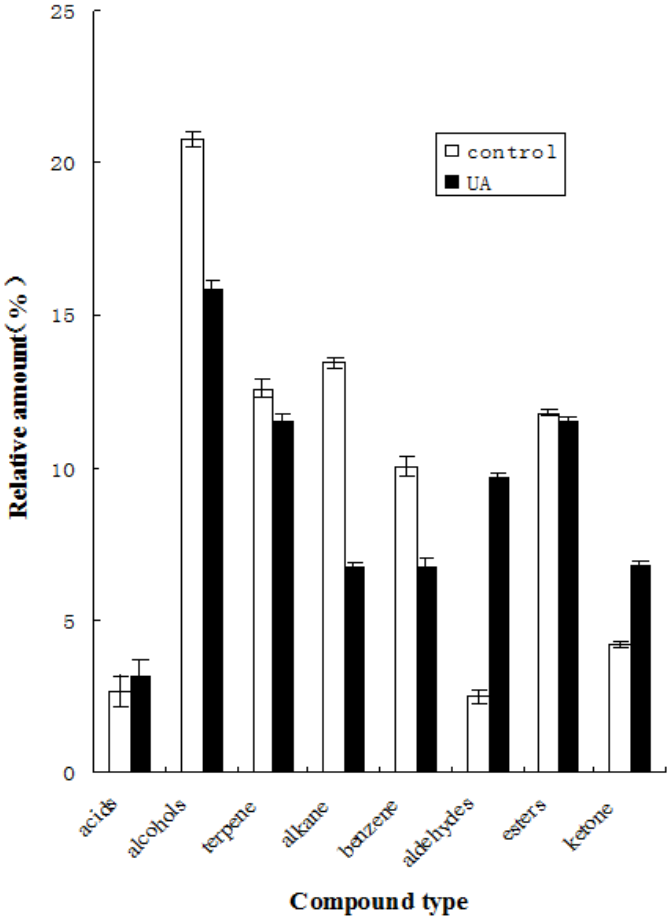

Fig. 3: Comparison of relative amount of aroma components identified in conventional extraction and ultrasonic sample

Cyclohexane, (1-methylpropyl)-, 2-Pentyn-1-ol, Nonanal, 1, 6, 10-Dodecatriene, 7, 11-dimethyl-3-methylene-, Bicyclo (3.3.1) non-6-en-2-one, Cyclohexene, Dicyclopropylmethanimine, N-cyano-, 4H-Cyclopenta (3, 4) cyclobuta (1, 2-b) furan-4-one, 3a, 3b, 5, 6, 6a, 6b-hexahydro-, Cyclodecane, 2-Dodecanone, Cyclooctane, 3-Hexenoic acid, 5-hydroxy-2-methyl-, methyl ester, (R*, R*-(E))-, Epoxy-.alpha.-terpenyl acetate, 2, 4-Hexadiene, (E, E)-, Propanoic acid, 2, 2-dimethyl-, hexyl ester, 5H-Inden-5-one, 1, 2, 3, 6, 7, 7a-hexahydro-, Pyrimidine, 4, 6-dimethyl-, 2, 6-Octadien-1-ol, 3, 7-dimethyl-, (E)-, 2-Butene, 1-chloro-2-methyl-, 3-Buten-2-one, 4- (2, 2-dimethyl-6-methylenecyclohexyl)-, 2-Methyl-6-propylphenol, Pyrazine, 2-butyl-3, 5-dimethyl-, 2, 6, 10-Dodecatrienoic acid, 3, 7, 11-trimethyl-, methyl ester, (E, E)-, Hexanoic acid, hexadecyl ester, 2, 3-Dimethyl dodecane, Cyclopentanone, oxime, Nonanoic acid, ethyl ester, (Z, Z)-.alpha.-Farnesene, 2- (3, 3-Dimethyl-but-1-ynyl) -1, 1-dimethyl-3-methylene-cyclopropane, Formic acid, 3, 7, 11-trimethyl-1, 6, 10-dodecatrien-3-yl ester, Aspidospermidin-17-ol, 1-acetyl-19, 21-epoxy-15, 16-dimethoxy-, Aromadendrene oxide- (1), 1, 2-Benzenedicarboxylic acid, butyl 2-ethylhexyl ester, 1H-Purin-6-amine, N-methyl-, Bicyclo (2.2.2) octane-1, 4-diol, monoacetate, n-Hexadecanoic acid, Oxirane, tetradecyl-, 1-Nonadecene, Hexadecane, Estra-1, 3, 5 (10) -trien-17.beta.-ol, Docosanoic acid, ethyl ester, Octadecanal, etc.

Flavor components classification relative amount comparison between conventional extraction and ultrasonic sample: The determination of comparison with ultrasonic processing group shows that classification of the aroma components types are the same, both including terpenes, alcohols, aldehydes, ketones, acids, esters, alkane and methoxyl benzene, eight types, but all kinds of specific composition and relative content are different, ultrasonic processing sample is more abundant.

Figure 2 shows that ultrasonic sample has more kinds of compounds than conventional extraction (which is control) in most of compound type. That could confirm the opinion: the improvement by ultrasound is due mainly to the effects of acoustic cavitation, which enhances solvent penetration into the intracellular product release by disrupting the cell walls (Mason *et al*., 1996).

Perhaps because ultrasonic sample has more kinds of compounds, its relative amount of aroma components in each type is not more than that of conventional extraction, just as it shows in Fig. 3.

Sensory evaluation: The sensory profiles of conventional extraction and ultrasonic sample were evaluated by six experienced flavorists. A set of coded tasting cups containing 5 mL sample was presented to the panellists for sniffing and sipping and then the intensity of each attribute was ranked between ‘0’ and ‘5’. Experiments were carried out in triplicate while results were reported as the mean values. After reaching consensus, ultrasonic sample was more popular because of its rich flavor and more levels.

CONCLUSION

In this study, evaluation of tea at present is mainly the sensory evaluation of tea liquor, due to the limitations of evaluator who makes result more subjective, lack of stability and accuracy. Volatile composition analysis of the tea soup can reflect the real fragrance of the tea after soaking, HS-SPME method combined with GC-MS analysis of tea aroma component is a sensory evaluation, which is more objective and accurate. We examine the effect of ultrasound processing method on the volatile flavor compounds and sensory quality of tea infusion. This study provides scientific basis for the evaluation and classification of Laoshan green tea and the improvement of tea quality.

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