Introduction

Aroma is one of the key indicators for evaluation of the quality of Oolong tea. The formation and characteristics of aroma from Oolong tea are accompanied by a series of biological changes during the manufacturing process. However, conventional techniques including sensory tests or a few targeted compound tests cannot reflect the overall biological changes during the manufacturing process. Therefore, a more effective method is highly desired. The technique of gas processing.

Method

Samples of tea leaves at various stages of the manufacturing process were analyzed by solid-phase micro-extraction and Triple Quadrupole GC-MS in MS scan mode. Compounds were then extracted based on deconvolution using Unknowns Analysis software. The extracted compounds were exported as a compound and subsequently imported into Mass Profiler Professional (MPP) software, a chemometric software for data alignment and analysis. The resulting data were then subjected to identification through the integrated ID Browser in MPP. In this study, 40 feature compounds were identified as the key flavoring compounds in tea by assigning against the NIST library, the compounds were mainly alcohols, ketones, terpenes, esters, etc. (Fig. 3). The charts in Fig. 4 demonstrate the clear variation in abundance for the selected four compounds in the different processing steps.

Experimental

Tea Sample

Four groups of tea leaf samples with seven biological replicates each were collected at each step of tea processing, including fresh leaves, leaves after sun withering, leaves after rocking green, and leaves after deactivation of enzymes.

Data Extraction

A total of 943 entities were obtained after data alignment on the frequency of occurrence and abundance cutoff of 0.01. Further filtration by fold change (FC>2) using fresh leaves as a reference led to a reduction of entities from 149 to 54.

 principality Component Analysis (PCA)

Based on PCA analysis of the 46 identified entities by ID Browser, an excellent separation of tea samples from different processing stages was observed (Fig. 2). Data filtering

mass Profiler Professional(MPP)

Basic statistical and multivariate analyses were carried out using MPP software. Chromatographic peak deconvolution using the Unknowns Analysis tool was used to find more than 400 compounds in each sample (Fig. 2). Deconvolution was able to extract clean spectra from background noise based on both retention time and peak shape. For each sample, compound lists were obtained and imported to MPP software for analysis.

SPME Conditions

3.5 cm tea was placed in a vial and infused with 10 ml of boiling water. Then 10 μl internal standard (p-tert-Butyphenol) and a SPME fiber were added into the vial. Immediately after the sample was cooled and placed in a 50°C oven.

GC Conditions

GC system: Agilent 7890A; Column: DB-5MS (60 m×0.32 mm×0.25 μm); Column temperature: from 50°C hold 5 min; from 150°C to 250°C hold 5 min; Injection port temperature: 270°C; Carrier gas: Helium; Flow rate: 1.0 ml/min; Injection mode: manual, SPME Fiber Injection port temperature: 370°C

MS Conditions

MS system: Agilent 5975C; Ion source: EI; temperature 330°C; Ion energy 70 eV; Scanning range: 40-500 m/z; Quadrupole temperature 150°C.

Results and Discussion

Data Filtering

Fig. 1. The total ion chromatograms of tea at four different stages of processing (a) Chromatographic peak deconvolution using the Unknowns Analysis tool (b) Chromatographic peak deconvolution using the Unknowns Analysis tool

Fig. 2. D and 5-S-phenylcynamide analysis (PCA) of the four processing stages of tea leaf samples

Fig. 4. The clear variations in abundance for the selected four compounds in different processing steps

Fig. 3. ID Browser function in MPP for compound identificatio

Conclusions

• A GC-MS method for profiling of Oolong tea under different processing steps has been developed.

• The acquired data has been subjected to chemometric analysis using MPP, and principle components analysis has shown excellent separation among four groups of tea leaf samples.

• Feature compounds, such as alcohols, ketones, terpenes, esters, etc., were combined well with the aroma variation of the tea samples.

• The finding is potentially beneficial for guidance of tea processing and control of tea quality.

The finding is potentially beneficial for guidance of tea processing and control of tea quality.

Study of the Aroma Variation in Oolong Tea Processing by Solid-Phase Micro-Extraction and Gas Chromatography-Mass Spectrometry

Chengying Ma1, Shan Zhou1, Wenwen Wang2, Aiqing Miao1, Junxi Cao1

1 Tea Research Institute, Guangdong Academy of Agricultural Sciences, Guangdong, China; 2 Agilent Technologies Company, Ltd., Beijing, China

ASMS 2018 WP 242

Study of the Aroma Variation in Oolong Tea Processing by Solid-Phase Micro-Extraction and Gas Chromatography-Mass Spectrometry

Chengying Ma1, Shan Zhou1, Wenwen Wang2, Aiqing Miao1, Junxi Cao1

1 Tea Research Institute, Guangdong Academy of Agricultural Sciences, Guangdong, China; 2 Agilent Technologies Company, Ltd., Beijing, China

ASMS 2018 WP 242