

Analysis of menthol optical isomers in tobacco products

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1 Abstract

Menthol has long been an important flavorant in tobacco products. Menthol can exist in **four enantiomeric pairs**, each of which has D-type and L-type, respectively. Different menthol isomers possess different sensory properties, among which **L-menthol has obvious mint flavor and cool taste characteristics**, and its cooling threshold is far lower than other isomers, while other menthol isomers have obvious bad or irritating odor just like the smell of mildew. Establishment configuration analysis technology of menthol isomer is of great value for the quality evaluation and application of menthol additives in tobacco.

Based on the detailed study of the separation efficiency of 8 menthol optical isomers by different chiral and non-chiral capillary columns, a method for the separation of 8 menthol optical isomers in tobacco products by tandem chiral capillary columns and gas chromatography-mass spectrometry was established.

2 Materials and methods

Instrument: Clarus 600 GC/MS (PerkinElmer).

Tandem chiral column-GC-MS method: The separation of menthol isomers was carried out with cyclosil-B + BGB-175 tandem chiral capillary column. The first column was a 30 m × 0.22 mm ID × 0.25 mm column containing heptakis(2,3-di-O-methyl-6-O-t-butyl dimethylsilyl)-β-cyclodextrin (Agilent, USA), and the second column was a 30 m × 0.22 mm ID × 0.25 mm column containing 2,3-diacetyl-6-tert-butyl dimethylsilyl-γ-cyclodextrin (BGB, Switzerland).

GC/MS condition: GC oven was programmed from 45 °C to 100 °C at 10 °C /min and held for 16 min, then raised to 200 °C at 10 °C /min and held for 10 min. The MS was operated in selected ion monitoring mode (SIM), and the monitoring ions were 71, 81 and 95 m/z.

Sample: 13 kinds of menthol-flavored traditional cigarettes (C1-C13) and 13 kinds of menthol-flavored heated cigarettes (hc1-hc13).

3 Results and Discussions

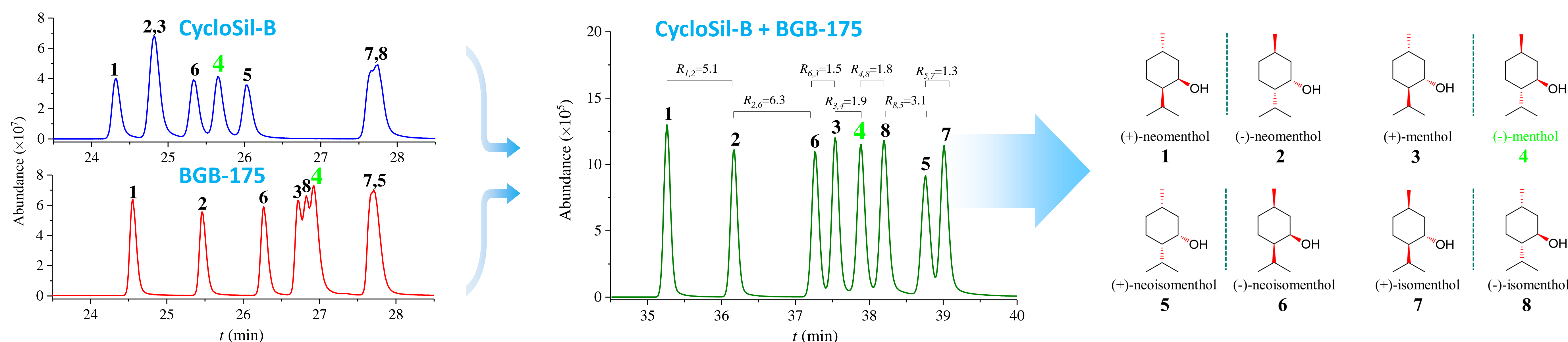


Fig.1 Chromatogram of eight menthol optical isomers separated by single and tandem chiral column

3.1 Enantioseparation of menthol isomers on different GC columns

(1) **Non-chiral DB-5MS, DB-624, DB-ALC1 and HP-INNOWAX columns:** Four enantiomeric pairs of menthol can be separated, but the enantiomers of D-type and L-type can not be separated.

(2) **Chiral CycloSil-B and BGB-175 columns:** D-type and L-type enantiomers from 4 pairs of menthol enantiomers can be separated, but the chromatographic peaks of D-type and L-type isomers of different enantiomers overlapped.

(3) **Tandem chiral CycloSil-B + BGB-175 column:** 8 menthol optical isomers were successfully separated. The chromatographic peaks of 8 menthol optical isomers all met the requirements for separation, and the peaks were all sharp and symmetrical. The response value and retention time had good daytime stability.

3.2 Method validation

A quantitative method for the determination of 8 menthol optical isomers was established, with the limit of quantification (LOQ) lower than **72.9 µg/L**, the relative standard deviations (RSDs) below **3.4 %** and the recoveries ranging from **92.1 % to 109.5 %** at three spiked levels.

3.3 Quantification of menthol isomers in tobacco products

The results showed that the dominant form of menthol in all samples was L-menthol, and the isomer purity of L-menthol was **higher than 97.9 %**, except that the purity in one filter tow was 92.0 % and that in one breakable capsule was 95.7 %.

Trace other menthol isomers were also detected in most samples and demonstrated their **distinct isomer distribution characteristics**, which may provide the basis for verifying the product source, distinguishing diverse tobacco samples and identifying the authenticity.

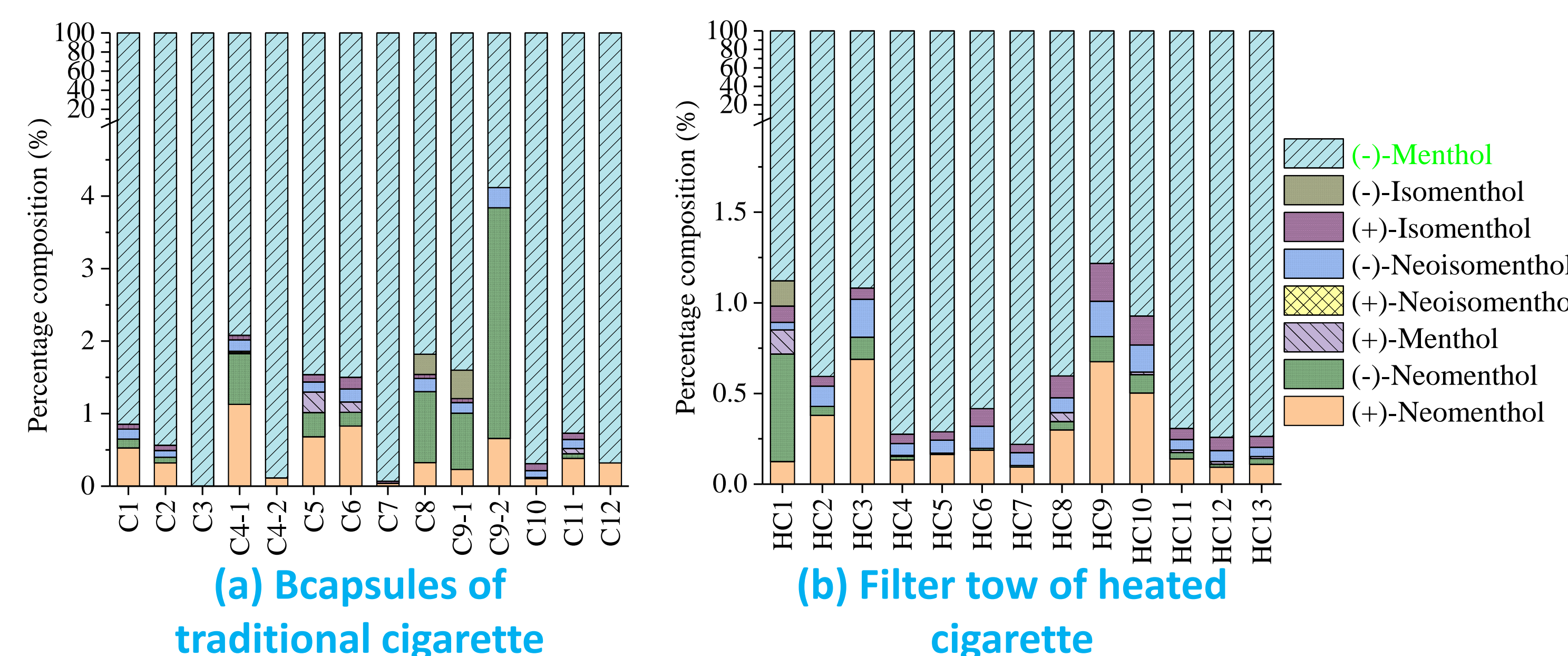


Fig.2 Disribution of menthol isomers in samples