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Simultaneous Quantification of Furaneol and Sotolon in Alcoholic Beverages by GC-MS after In-solution Silylation

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Abstract

In this study, a reliable and robust method was developed to analyze furaneol and sotolon based on *N,O*-bis(trimethylsilyl) trifluoroacetamide (BSTFA) silyation coupled with GC-MS. Various derivative parameters were evaluated, and the results demonstrated that the ratio of sample, BSTFA (5% trimethyl chlorosilane, TMCS) and dichloromethane of 1:1:1 , derivatization temperature of 50˚ C, and reaction time of 30 min were the best for the reaction. Recovery of furaneol and sotolon was 92.5 and 85.2%, respectively. Limit of detection (LOD) and limit of quantification (LOQ) were 7.3 and 9.1 μg/L for furaneol and 24.3 to 30.4 μg/L for sotolon, respectively. The optimal method resulted in good repeatability and reproducibility with the inter- and intra-day relative standard deviations less than 11%. The developed method was applied to Pinot noir wine, Chardonnay wine, beer and hard iced tea, the results showed that the highest furaneol and sotolon concentrations was determined in beer, ranging from 101 μg/L to 487 μg/L, which was 200-400 times higher than in Pinot noir wines and 20-40 times compared with the hard iced tea.

Introduction

Furaneol and sotolon have very low odor threshold of 5 μg/L and 0.3 μg/L in water, respectively. They are widely used as caramel, fruity and sweet aroma enhancers in fruit jams, beverages and sweets. However, owing to the presence of furanone-ring and active hydrogen group in their molecules, they are unstable in basic condition and in apolar organic solvent. Moreover, due to the thermal instability, the sensitivity of GC-MS method is usually very low and often below detect limit. Thus, it requires the development of sophisticated method to obtain reliable quantification results.

Therefore, in this study, we proposed a BSTFA-based silylation method to determine concentrations of furaneol and sotolon for the first time. And we have successfully applied it in various alcoholic beverages including Pinot noir wine, Chardonnay, beer and hard iced tea.

Conclusions

Results of this study suggested that silylation coupled with GC/MS is a robust method to quantify furaneol and sotolon in different kinds of alcoholic beverages. Simplicity and efficiency of BSTFA silylation makes the quantification process quite attractive when coupled with the Lichrolut EN SPE purification. This method was successfully applied to different alcoholic beverages. Recoveries of furaneol-TMS and sotolon-TMS were 92.5% and 86.5%, respectively. Repeatability and reproducibility values (RSDs) were less than 11%. Limit of detection (LOD) and limit of quantification (LOQ) were 7.3 and 9.1 μg/L for furaneol and 24.3 to 30.4 μg/L for sotolon, respectively.

Methods

- The reaction temperature, time, percentage of catalyst, and silylation solvent was systematically evaluated to obtain optimal silylation condition.
- The linearity of the developed method was evaluated by preparing calibration curves.
- The LODs and LOQs of furaneol-TMS and sotolon-TMS were determined as 3 and 10 times of signal to noise (S/N) of certain concentration of standards, respectively.

Results

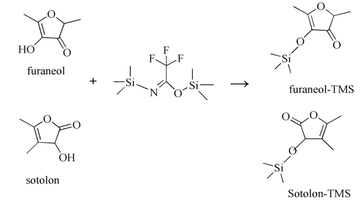

Fig. 1 Scheme of BSTFA-based silylation.

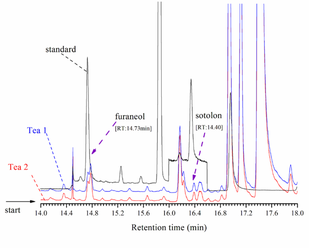

Fig. 2 SIM chromatograms of derivate products for pure standards and related compounds in hard ice tea.

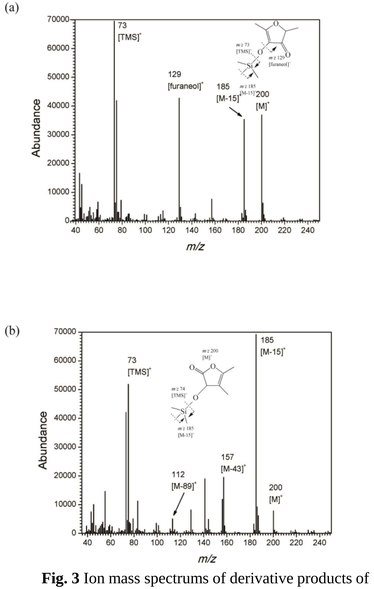

Fig. 3 Ion mass spectrums of derivative products of furaneol and sotolon.

Results

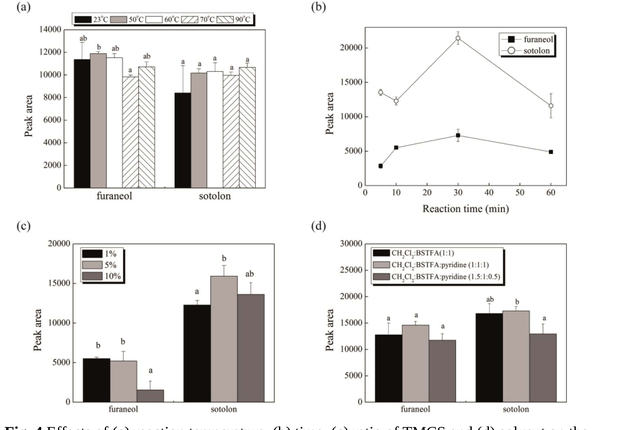

Fig. 4 Effects of (a) reaction temperature, (b) time, (c) ratio of TMCS and (d) solvent on the silylate reaction. Different letters represented the significant difference (one-way ANOVA with Tukey's test, $P < 0.05$).

Tab. 1 Concentrations of furaneol and sotolon in different alcoholic beverages.

Samples	furaneol	sotolon
	μg/L	
Pinot noir wine 1	1.99±0.06	n.d.
Pinot noir wine 2	1.92±0.03	n.d.
Pinot noir wine 3	2.09±0.08	n.d.
Pinot noir wine 4	1.78±0.02	n.d.
Pinot noir wine 5	1.83±0.06	n.d.
Chardonnay	1.10±0.00	n.d.
Beer 1	487±98.8	177±5.49
Beer 2	285±25.4	101±7.29
Tea 1	11.7±0.50	17.8±0.13
Tea 2	12.2±0.56	n.d.