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Development and Optimization of a Solid Phase Dynamic Extraction (SPDE) Method for Volatile Compound Determination in Beer

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ABSTRACT

A headspace solid phase dynamic extraction coupled with gas chromatography mass spectrometry (HS-SPDE-GC/MS) method for the analysis of the volatile fraction of beer was optimized. Optimization was performed using a mixture of five volatile compounds commonly found in beer (isoamyl acetate, ethyl hexanoate, myrcene, benzaldehyde, linalool). A two-level full factorial design was used, with extraction temperature, extraction strokes, and fill/eject speed as the main factors. The results showed that extraction time and temperature significantly affected the peak area response of the volatile compounds. A second optimization of the significant factors was carried out using a Doehlert matrix design. An extraction temperature of 42 ° C and 46 extraction strokes provided the highest total peak area GC response for the five volatile compounds. This optimized method was applied for the analysis of five commercial beers. A variety of compounds from different chemical classes were extracted from each sample, reflecting the effective application of the SPDE method for volatile compound determination across different beers.

INTRODUCTION

The volatile compound fraction in beer is important as these compounds significantly contribute to the perceived flavor in the beer (1). In recent years, headspace solid phase microextraction (HS-SPME) coupled to Gas Chromatography has become the most widely used headspace sampling technique (2), and has been widely used in beer analysis (1,2,3). A further development of SPME is solid phase dynamic extraction (SPDE). SPDE is based on the same principles as SPME, but uses an internally coated steel needle instead of a fiber for the extraction and pre-concentration of compounds from the solution headspace (4). To the best of our knowledge the SPDE technique has not yet been applied for the analysis of the volatile fraction of beer.

The objectives of this study were to: 1) optimize a HS-SPDE method for the direct extraction of volatile compounds from beer through the use of a factorial design and a Doehlert matrix design, and 2) determine its suitability for the characterization of the volatile fraction of beer.

MATERIALS AND METHODS

Sample preparation. Beer samples were purchased from a local store and kept at 4 ° C until analysis. The beer samples selected included: Beer A (American lager), B (American light lager), C (American Pale Wheat Ale), D (Banana bread beer), and E (Raspberry wheat ale).

Volatile Compound Stock Solution. For method optimization, a volatile compound stock solution in 5% ethanol was prepared to compose a solution with final concentrations of: isoamyl acetate (0.84 mg/L), ethyl hexanoate (0.85 mg/L), benzaldehyde (1.03 mg/L), myrcene (0.71 mg/L) and linalool (1.03 mg/L).

Optimization

- Samples analyzed by HS-SPDE-GC/MS using preliminary 2 level full factorial design.

- Factors analyzed: extraction strokes, extraction temperature, extraction speed.

- Sum of GC peak areas was used as the response and analyzed using ANOVA.

- Significant factors optimized using Doehlert matrix design and response surface analysis.

Volatile Compound Analysis

- Beer Samples analyzed using optimized SPDE conditions
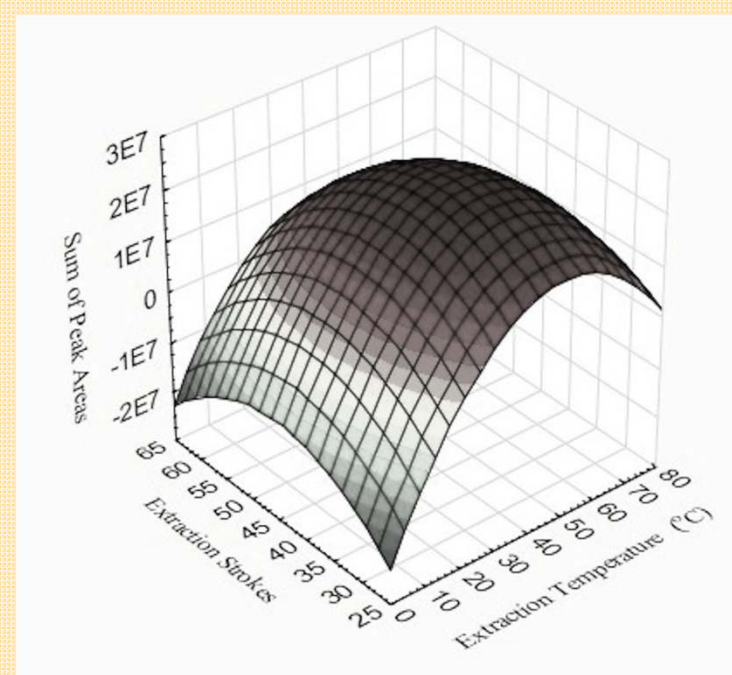
RESULTS AND DISSCUSSION

Fig.1 Response surface plot for extraction strokes and extraction temperature

- Extraction temperature and extraction strokes had a significant effect on volatile compound extraction. These factors were optimized using the Doehlert design.

- Fitted response surface for significant factors obtained from the Doehlert design is shown in figure 1.

- Extraction Temperature of 42° C and 46 extraction strokes provided the highest total GC area response.

- The technique allowed the identification of 19 volatile compounds of various chemical classes in the five commercial beer samples (Table 1).

Table 4. Peak identification and relative peak areas of compounds found in five commercial beers. Beer A (lager), B (light lager), C (Wheat Ale), D (Banana bread beer), and E (Raspberry wheat ale).

Compound	Chemical Class	Beer Samples				
		A	B	C	D	E
Ethyl acetate	Ester	6.49	1.45	0.98	0.25	1.12
1-Pentanol	Alcohol	-	-	66.00	-	46.30
2-ethyl-cyclobutanone	Ketone	31.43	-	-	20.73	-
Ethyl-2-methylbutyrate	Ester	-	-	-	-	8.50
Isoamyl aceate	Ester	50.37	37.40	9.69	60.86	30.94
β-myrcene	Terpene	-	-	-	-	1.86
Ethyl hexanoate	Ester	2.70	2.89	6.07	1.42	3.09
Limonene	Terpene	-	-	-	-	0.95
β-Ocimene	Terpene	-	-	-	-	0.58
(+)-3-Carene	Terpene	-	-	-	-	2.93
Isoamyl isovalerate	Ester	-	-	-	6.49	-
Phenylethyl alcohol	Alcohol	1.94	2.54	3.49	2.47	6.72
Ethyl decanoate	Ester	-	-	-	-	0.78
Ethyl heptanoate	Ester	-	-	2.12	-	-
Octanoic acid	Acid	-	0.13	1.23	0.44	2.98
Ethyl octanoate	Ester	2.52	5.40	7.05	3.28	6.81
Citronellol	Terpene	-	-	-	-	0.50
Ethyl laurate	Ester	-	-	5.04	-	-

CONCLUSIONS

➢ The Doehlert design was efficient as it enabled the simultaneous optimization of the parameters affecting the extraction of volatile compounds.

➢ In SPDE, extraction temperature and extraction strokes significantly influenced the extraction of volatile compounds from beer samples.

➢ The optimized HS-SPDE-GC/MS method enabled the extraction of a wide variety of compounds in the beer samples, showing promise for the analysis of the volatile fraction in beer.

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