Solid-phase microextraction for headspace analysis of key volatile compounds in orange beverage emulsion

ABSTRACT

Headspace solid-phase microextraction (HS-SPME) gas chromatography was used to analyze target flavor compounds in orange beverage emulsion. The effects of SPME fiber (PDMS 100 lm, CAR/PDMS 75 lm, PDMS/DVB 65 lm and DVB/CAR/PDMS 50/30 lm), adsorption temperature (25–45 C), adsorption time (5–25 min), sample concentration (1–100%), sample amount (5–12.5 g), pH (2.5– 9.5), salt type (K2CO3, Na2CO3, NaCl and Na2SO4), salt amounts (0–30%) and stirring mode were studied to develop HS-SPME condition for obtaining the highest extraction efficiency and aroma recovery. For the head space volatile extraction, the optimum conditions were: CAR/PDMS fiber, adsorption at 45 C for 15 min, 5 g of diluted beverage emulsion (1:100), 15% (w/w) of NaCl with stirring and original pH 4. The main volatile flavor compounds were: limonene, 94.9%; myrcene, 1.2%; ethyl butyrate, 1.1%; c-terpinene, 0.41%; linalool, 0.36%; 3-carene, 0.16%; decanal, 0.12%; ethyl acetate, 0.1%; 1-octanol, 0.06%; geranial, 0.05%; b-pinene, 0.04%; octanal, 0.03%; a-pinene, 0.03%; and neral, 0.03%. The linearity was very good in the considered concentration ranges (R2 P0.97). Average recoveries ranged from 88.3% to 121.7% and showed good accuracy for the proposed analytical method. Average relative standard deviation (RSD) for five replicate analyses was found to be less than 14%. The limit of detection (LOD) ranged from 0.06 to 2.27 mg/l for all volatile flavor compounds and confirmed the feasibility of the HS-SPME technique for headspace analysis of orange beverage emulsion. The method was successfully applied for headspace analysis of five commercial orange beverage emulsions.

Keyword: Solid-phase microextraction, Gas chromatography, Flavor compounds, Orange beverage emulsion, Extraction efficiency, Headspace analysis