Solid-phase microextraction for determining twelve orange flavour compounds in a model beverage emulsion

ABSTRACT

Solid-phase microextraction (SPME) coupled to gas chromatography has been applied for the headspace analysis (HS) of 12 target flavour compounds in a model orange beverage emulsion. The main volatile flavour compounds studied were: acetaldehyde, ethyl acetate, α-pinene, ethyl butyrate, β-pinene, myrcene, limonene, γ -terpinene, octanal, decanal, linalool and citral (neral plus geranial). After screening the fibre type, the effect of other HS-SPME variables such as adsorption temperature (25–55°C), extraction time (10–40 min), sample concentration (1–100% w/w), sample amount (5–10 g) and salt amount (0–30% w/w) were determined using a two-level fractional factorial design (25−2) that was expanded further to a central composite design. It was found that an extraction process using a carboxen−polydimethylsiloxane fibre coating at 15ºC for 50 min with 5 g of diluted emulsion 1% (w/w) and 30% (w/w) of sodium chloride under stirring mode resulted in the highest HS extraction efficiency. For all volatile flavour compounds, the linearity values were accurate in the concentration ranges studied (r 2 > 0.97). Average recoveries that ranged from 90.3 to 124.8% showed a good accuracy for the optimised method. The relative standard deviation for six replicates of all volatile flavour compounds was found to be less than 15%. For all volatile flavour compounds, the limit of detection ranged from 0.20 to 1.69 mg/L.

Keyword: Solid-phase microextraction, headspace analysis, orange beverage emulsion, fractional factorial design, central composite design, extraction efficiency