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COMPARISON OF DIFFERENT EXTRACTION METHODS TO ISOLATE AROMA VOLATILES FROM CASHEW APPLE JUICE

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Of the methodologies available for extracting the volatiles of greater importance for the aroma and flavour of beverages, liquid-liquid extraction (LLE) is one of the most traditional, but consumes large amounts of expensive, generally toxic, solvents. Dynamic headspace methodology (DHS) is also widely used, but recently solid phase micro-extraction (SPME) has gained popularity, being easy to use, quick, requiring no solvents and using a small sample volume. Thus the objective of the present research was to compare the efficiency of LLE, HS-SPME and DHS on the profile of the odour volatiles isolated from fresh cashew-apple juice. Initially, DHS was used to strip the volatiles from the headspace of 300mL fresh cashew apple juice (cv CCP76) for 2 h to a PorapakQ® trap using vacuum at 70mmHg and room temperature and then elute with 300µL acetone. SPME extraction was carried out for 45min in the headspace of 5ml of fresh cashew juice at 22±2°C using a DVB/Carboxen/PDMS fibre. After extraction, the SPME device was introduced into the splitless injector of a Gas Chromatograph (GC), and maintained at 200°C for 5min. Finally, the volatiles present in 40mL of fresh cashew juice were extracted by LLE with 15mL of methylene dichloride using 3 consecutive extractions. A second extraction was carried out in the same way, and the extract concentrated to 0.5mL under a nitrogen flow, generating a concentrated LLE extract. The four isolates were evaluated by gas chromatography coupled to mass spectrometry (GC-MS), and GC-olfactometry (1) was carried out on both the DHS and the two LLE isolates. Less volatiles were isolated by HS-SPME (n=72 volatiles) as compared to DHS (n=100 volatiles), and LLE with (n=116 volatiles) and without concentration (n=100 volatiles). Slightly more odoriferous volatiles were detected in the DHS isolate (n=44) as compared to isolates generated by LLE with (n=38) and without concentration (n=39). Ten volatiles, all esters obtained from the DHS isolate showed average odour intensity equal or above 4.0 on a 10-point scale, indicating that the panel members perceived their intensity as between moderate and high. Six volatiles from the concentrated LLE isolate were perceived with an average odour intensity $\geq$ 4, including 4 esters, 1 alcohol and 1 acid. Finally, 7 volatiles from the non-concentrated LLE isolate showed average odour intensity $\geq$ 4, including 3 esters, 3 terpenes and 1 aldehyde. The GC-MS technique confirmed that the esters were best isolated using the DHS technique, representing 33% of the total chromatogram area of the DHS isolate, 16% of that of the concentrated LLE isolate and 11% of that of the non-concentrated one. Esters represented 39.53% of the total chromatogram area of the

HS-SPME isolate but ethyl hexanoate alone corresponded to 31% of the total area. GC-MS also indicated that terpenes were best isolated by the non-concentrated LLE extract where they represented 41% of the total chromatogram area, whereas for the DHS isolate they represented 11% of the total area, less than 2% for the concentrated LLE extract, and less than 1% for the HS-SPME isolate.

References:

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