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Short communication

Enhanced flavour extraction in continuous liquid–liquid extractors

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Abstract

Continuous liquid–liquid extraction is a versatile, reliable and robust sample preparation technique, but there is a tendency for the solvent to make channels through the sample, causing uneven and incomplete extraction. A simple, cheap magnetic stirrer that prevents channelling improves extraction efficiency by between 2.8 and 17.6 times.

Keywords: Sample preparation; Liquid–liquid extraction; Solvent extraction; Aroma; Flavour; Fruit; Guava; Pineapple; Gas chromatography; Mass spectrometry

1. Introduction

“Sample preparation is one of the most critical aspects of the analysis of complex matrices for trace components and can also be the most time consuming.” [1].

Sample preparation techniques for flavour and off-flavour analysis in foods and beverages are as diverse as the analytes and matrices to which they are applied [2,3]. While solid-phase microextraction (SPME), stir bar sorbtive extraction and solid phase extraction are finding increasing application in the field of flavour and aroma analysis, continuous liquid–liquid extraction (CLLE) which dates back at least to the 1960s [4], remains a rugged and reliable workhorse that provides good recoveries of analytes over a wide range of volatilities and functional groups [5]. Compared to other systems [6] the glassware is simple and the procedures straightforward. It requires no gas chromatographic inlet hardware beyond a split–splitless injector, and it can run unattended for long periods [2,7,8]. Because a small volume of solvent [5] is continuously recycled, solvent purity is less critical than with the larger volumes used in batch extractions [9]. The extract from a continuous liquid–liquid extraction can be concentrated by evaporation to a high enough concentration for the detection of trace (sub μg/L) components [10], and a single extraction yields sufficient concentrated extract for repeated analyses under different conditions, as required for example by aroma dilution analysis [11,12]. CLLE is especially valuable in exploratory flavour and fragrance research where the composition of samples in terms of viscosity, suspended solids and flavour compound concentration is variable and unpredictable.

Published applications of CLLE are limited to samples that are free-running liquids. Viscous or heterogeneous samples such as fruit or vegetable pulps can lead to incomplete and uneven extraction because the solvent repeatedly follows the same channel through the sample. Filtering or centrifuging the pulp to remove suspended solids easily solves this problem [8,12], but assumes that the flavours are all in the liquid fraction. Excessive high-speed blending in an attempt to reduce viscosity or make samples very homogenous can change flavour profiles [9]. Sufficient dilution of viscous samples to provide a free-running solution or suspension with no tendency to channelling necessarily also dilutes the analytes of interest and compromises their recovery in the extract.

If the classical design for an extractor using heavier than water solvents [4] is modified by moving the solvent return arm to the side of the sample cylinder (Fig. 1), a conventional magnetic stir bar can easily be placed in the bottom of the extractor, but for the stirring to extend up through the column of viscous sample the stirrer has to be run at high speed, which prevents separation of the solvent and sample at the

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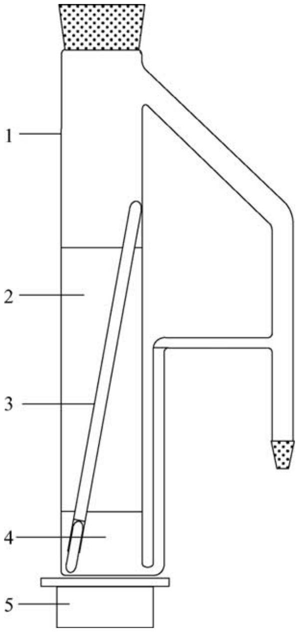

Fig. 1. Deployment of the stirring rod. (1) Extractor body (the condenser and solvent flask are not shown); (2) sample; (3) stirring rod; (4) heavier-than-water solvent; and (5) magnetic stirrer driver.

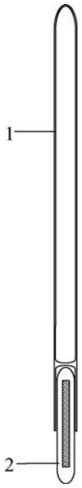

Fig. 2. Construction of the basic stirring rod. (1) Thin-walled glass tube and (2) Teflon-coated magnetic stir bar which is a sliding fit into the bottom of the glass tube.

bottom of the extractor, and leads to carryover of sample into the solvent flask. Rather than high speed mixing, what is required is a slow churning action extending throughout the column of sample. While a low speed shaft mixer would

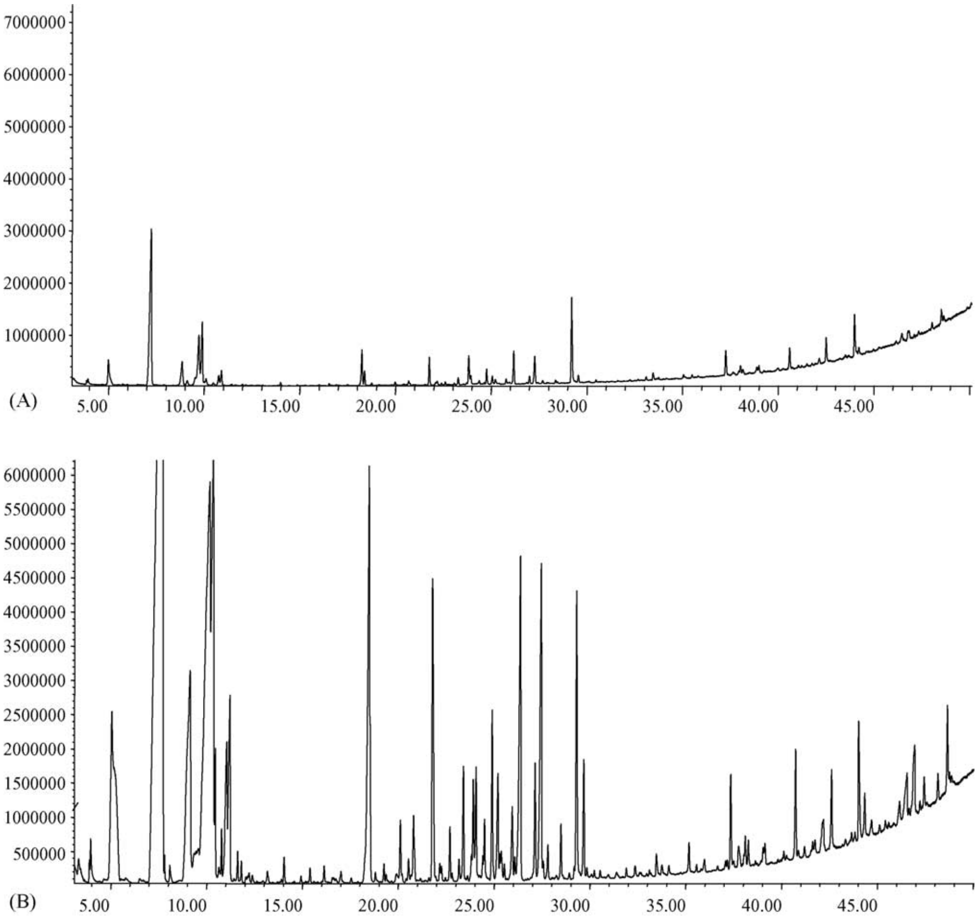

Fig. 3. TIC chromatograms from liquid–liquid extracts of guava pulp extracted without stirring (A) and with stirring (B).