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Original Article

Analysis of volatile compounds from Malaysian durians (*Durio zibethinus*) using headspace SPME coupled to fast GC-MS

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Abstract

Headspace solid phase microextraction (SPME) coupled to fast gas chromatography-mass spectrometry (GC-MS) was applied to analyze the volatile compounds of durian (*Durio zibethinus*) varieties D2, D24, and D101 from Malaysia. Sampling sensitivity was improved by evaluation of sample matrix, sampling size, headspace volume, salt addition and sampling duration. A total of 39 volatile compounds were identified including 22 esters, 9 sulphur-containing alkanes, 3 thioacetals, 2 thioesters, 2 thiolanes and 1 alcohol. The relative amount of volatiles estimated using 1 ppm internal standard (IS) revealed the differences in the volatile composition among varieties. Further classification and characterization of each durian variety was successfully conducted using principal component analysis (PCA).

Keywords: Durian; *Durio zibethinus*; Solid phase microextraction (SPME); Fast gas chromatography; Principal component analysis (PCA)

1. Introduction

Durian (*Durio zibethinus*), a member in the *Bombaceae* family, is one of the most well-known tropical fruits in southeast Asia. Complete morphological details have been given elsewhere (Wong and Tie, 1995; Moser et al., 1980). Durian species growth originated in the Malay Peninsular and now over 20 durian varieties can be found in the Malaysian market; only certain varieties, however, are highly favoured (Berry, 1981) and prized over others because of specific flavour as well as their particular odour. Studies have been carried out to attempt to verify the volatile and non-volatile substances that are responsible for the durian flavour. A total of 137 durian volatile constituents were reported by previous works (Jaswir et al., 2005; Weenen et al., 1996; Naf and Velluz, 1996; Wong and Tie, 1995; Moser et al., 1980; Baldry et al., 1972). Jaswir et al. (2005) had identified 38 volatile compounds comprising mostly esters and acids in fresh durian aril variety D24 using *n*-hexane solvent in simultaneous steam distillation extraction. Wong and Tie (1995) examined three Malaysian durian varieties (D15, D28 and D74) and discovered 63 volatile constituents overall, whereas higher sulphide volatiles content were present in durian clone D28. Among various durian varieties, sulphur-containing volatiles such as thiols, disulfides and trisulfides were reported as the major volatile constituents that possessed the distinct strong onion-like odour, while esters were the predominant volatiles that corresponded to the fruity odour (Baldry et al., 1972; Moser et al., 1980; Weenen et al., 1996). Nevertheless, information regarding the volatile composition of the flavours which distinguishes the most popular and preferred durian varieties is limited.

Solid phase microextraction (SPME) combines procedures that include analyte isolation from sample matrix, concentration and introduction of analytes into gas chromatography in an analysis. A short, thin, solid rod

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of fused silica which is coated with an absorbent polymer was utilized as the tool in SPME to absorb or adsorb the target analytes from sample matrix until equilibrium was reached in the system. SPME provides advantages such as eliminating the need for solvent; it is inexpensive, rapid and easy to use; it is compatible with a wide range of analytical instruments; and the detection limits are improved (Zhang and Pawliszyn, 1993; Workhoff et al., 2002). To date, SPME has been successfully applied in the qualitative and quantitative analysis of volatiles in fruits such as kiwi, strawberry, blackcurrant, cantaloupe and banana (Wan et al., 1999; Schulbach et al., 2004; Ruiz del Castillo and Dobson, 2002; Beaulieu and Grimm, 2001; Liu and Yang, 2002). However, a calibration or optimization of SPME conditions is always required for increasing the sensitivity and accuracy of the analysis (Penton, 1999).

Multivariate analysis is employed in food quality evaluation in order to investigate the underlying relationships that exist between numerous variables and consequently to interpret the sample classification on the basis of these variables (Chien and Peppard, 1993). Thus, application of headspace SPME and fast gas chromatograph mass spectrometer (GC-MS) with multivariate statistical analysis would exist as a rapid, convenient and reliable tool for routine analysis of the volatile profile in durian fruit. The present work aims to study the composition of volatile constituents in fresh durian pulp using headspace SPME coupled to fast GC-MS. The effect of several variables in SPME conditions on the extraction sensitivity was evaluated. The concentration of volatile compounds from three well-known clones, D24, D2 and D101, were estimated, and the chromatographic data obtained were processed using multivariate analysis to verify and discriminate among these varieties.

2. Materials and methods

2.1. Materials

Fruit samples of durian varieties D2, D24 and D101 were obtained from orchards located in Pahang State, Malaysia during the fruiting season in December 2004. Three to five durians of each variety were collected and only fully ripened fruits without any split on the husk were selected for the study. A manual SPME sampling unit, SPME fibre with 50/30 μm divinylbenzene/Carboxen on polydimethylsiloxane coating (DVB/CAR/PDMS), was purchased from Supelco (Bellefonte, PA). All GC chemical standards with purity grade higher than 98% were purchased from Aldrich Chemical Co. (Milwaukee, WI) while sodium chloride (NaCl), sodium sulfate ($NaSO_4$) and calcium chloride ($CaCl_2$) salts were purchased from Merck (Darmstadt, Germany). Stock solution of the internal standard (IS), thiophene was prepared in methanol at a concentration of 1000 mg/L and stored at 4 °C prior to use within 2 weeks.

2.2. Sample preparation and SPME conditions

One hundred grams of fresh durian pulp were taken and macerated with 200 g distilled water in a Waring blender for 1 min to form homogenate. Fifteen grams of durian homogenate were transferred to 30 mL vial together with a magnetic stirring bar (8×25 mm) and 5 g of NaCl. An amount of 15 μg thiophene was spiked into the sample to a concentration of 1 μg/g in the durian homogenate. The vial was crimped-sealed with 20 mm diameter aluminium seal and a Teflon septum. The fruit homogenate was kept under constant vigorous stirring at 30 °C for 15 min initially and the SPME syringe was then manually inserted into the headspace of the vial with the fibre coating exposed for 30 min.

2.3. GC-MS Conditions

A HP 5890 gas chromatograph coupled to a HP 5973 mass spectrometer (Hewlett-Packard, Avondale, PA) was utilized for this analysis. Once extraction was completed, the SPME fibre coating was immediately introduced into the GC injection port at 250 °C with a split ratio of 10:1 and maintained for 5 min. Separation of analytes was achieved with a Supelcowax-10 capillary column ($10\,m \times 0.10\,mm$ i.d. $\times\, 0.10\,\mu m$ film thickness) (Supelco, PA) under oven temperature program as follows: 40 °C initially, held for 1.5 min, then increased to 240 °C at rate 50 °C/min and held for 2 min. Purified helium (purity 99.999%) was used as the carrier gas at 0.4 mL/min constant flow rate. The mass spectrometer was operated in scan mode from m/z 33 to 400 at 2.05 scans/s, with 70 eV electron ionization at 230 °C, quadrupole at 150 °C.

Data was collected with HP ChemStation software (A.03.00) and identified according to National Institute of Standards and Technology (NIST) v2.0 library (Palisade Corp., Newfield, NY; to download see http://www.nist.gov/srd/nist1a.htm). Compounds were then confirmed by GC retention time (RT), MS spectra, and authentic standard compounds. The RTs from a series of aliphatic alkanes (C8–C22) were used to calculate retention indices (RIs) for all identified compounds. The relative amounts of volatile compounds were expressed as their peak area ratio to peak area of the IS (1 μg/g thiophene), using a response factor of 1.

2.4. Statistical analysis

The chromatographic data obtained by SPME-GC-MS was statistically tested using one-way ANOVA, principal component analysis (PCA) with Minitab software release 13. Correlation matrix was applied in multivariate analysis so that the data was autoscaled by variable to give the same weight to all components.