



## Pressurised liquid extraction of volatile compounds in coffee bean



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### ABSTRACT

In this work, we reported a novel application of pressurised liquid extraction (PLE) on coffee bean. The condition of PLE was carefully optimised with the aid of response surface methodology (RSM) including adjustment of experimental parameters (solvent type and sample to hydromatrix ratio) and other operating parameters (i.e. temperature (50–100 °C), pressure (1000–2000 psi) and static extraction time (5–15 min)). The coffee extracts obtained under three different extraction conditions were evaluated through descriptive sensory analysis. Then, the results showed that those targeted compounds obtained from PLE were nearly three times higher (1473 ppm) than conventional solvent extraction (571 ppm). Thus, PLE demonstrated the feasibility of producing a series of coffee extracts under controllable extraction conditions in correlation with desirable sensory attributes. This approach has not previously reported to characterise the aroma of coffee bean.

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### 1. Introduction

Recent advancement in developing rapid and sensitive extraction techniques has become increasingly important [1–6]. Pressurised liquid extraction (PLE), also known as accelerated solvent extraction (ASE), is developed based on Soxhlet extraction except the solvents used close to their supercritical region [4]. At elevated pressure and temperature, the solubility and diffusion rate of solvents is enhanced, and thereby improves the mass transfer of analytes [5,6]. With this improvement, extraction yield from complex matrices could be significantly increased with the decrease of extraction time. Moreover, extraction procedure becomes versatile through tuning different operating parameters (e.g. temperature, pressure, time, extraction cycles and solvent) [7]. Another detailed optimisation indicated the possibility of manipulating the composition of the extract by adjusting the extraction parameters [8]. On the other hand, even minor adjustments of PLE parameters could affect the composition of extracted compounds.

Multivariate statistical approach, namely response surface methodology was applied effectively to determine optimum extraction parameters [5,9–11]. With the linear or square polynomial functions obtained from RSM, the significant effects of main factors as well as their interaction effects were identified and predicted. In order to comprehend the interactions among PLE parameters, multiple responses of targeted compounds with different scaling can be transformed into a desirability function [10,12]. Furthermore, under

elevated temperature and pressure during PLE process, the composition of delicate aroma is easily distorted majorly due to thermal degradation of labile compounds and some side-reactions. Hence, besides the optimisation of operating parameters, a thorough sniffing is required to identify and monitor the desirable flavour profiles of different extracted products that are concomitant to the analytical work [13,14]. In contrast to other common PLE applications on environmental aspects [6,11,15–20], PLE has been found very limited reports in flavour isolation [7,8]. For example, PLE was applied to study volatile compounds in turmeric leaves, in which simultaneous optimisation of several responses was carried out based on the desirability function and evaluated the flavour intensity of turmeric leaf extracts [8]. However, high extraction yields as well as desirable flavour profiles are still challenging, especially dealing with complex food matrices.

Underlying the unique aroma of coffee is a profound complexity that involves more than 800 different chemical compounds inherited in roasted coffee beans [21–24]. Although the extensive studies have been carried out for decades, the determination of volatiles in roasted coffee bean is still a challenging task as many of the important odorants are present in trace amounts and/or are reactive and unstable [3,23,25,26]. To the best of our knowledge, there is only one study reported on the PLE of polycyclic aromatic hydrocarbon from coffee [20], and has yet to be applied on any other coffee volatiles.

In this work, the objective was to extract volatiles in the coffee bean using PLE. Initially, the feasibility of PLE on extracting coffee volatiles was evaluated through comparison to solvent extraction. Furthermore, the factors affecting PLE of volatiles in coffee beans were systematically optimised using RSM. Finally, the aromatic profiles of these coffee extracts were evaluated through descriptive sensory analysis.

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## 2. Materials and methods

### 2.1. Coffee bean and chemicals

In this study, Boncafé International Pte. Ltd. (Singapore) provided the roasted Sumatra Mandheling coffee (*Coffea arabica* L. cv. Catimor), which underwent a roasting process for 14 min with an initial temperature of 160 °C and discharged at 223 °C. Coffee beans were grounded (Café Select Classic KMM 30, Braun, Germany) and sieved (Coffee Grind Sizer, Coffee Chemistry, CA, USA) into the size range of 1.77–2.36 mm. The coffee powder was sealed in the aluminium pouch and stored at –20 °C until use.

Acetone, dichloromethane, hexane and methanol from Fisher Scientific UK Limited (Loughborough, UK) were of analytical grade. Anhydrous sodium sulphate purchased from Merck (Darmstadt, Germany) was used as a drying agent while hydro super gel diatomaceous earth (hydromatrix) obtained from Sigma Aldrich (Missouri, USA) was applied as drying agent and dispersing agent during extraction.

All standard compounds used in the identification of the volatiles were obtained from the Firmenich Asia Pte. Ltd., Singapore.

### 2.2. PLE procedure

Ten grams of coffee powder was evenly mixed with 5 g of diatomaceous earth, and then packed into a 40-mL stainless steel cell secured with a neoprene filtration end cap. Extraction was performed with a Power-Prep PLE (Fluid Management Systems, MA, USA). The automated extraction cycle was operated using DMS6000 software as follows: the cell containing coffee powder was prefilled with extraction solvent (i.e. methanol, hexane, and dichloromethane), pressurised and heated for a static period (see Table 1). The cell was then flushed with fresh extraction solvent and purged with a flow of nitrogen gas and the extracts were eluted out of the extraction cell into the collection bottle placed in an ice bath. The extract was cooled for 30 min and dried by 10 g of anhydrous sodium sulphate before being concentrated to 1.0 ml using TurboVap II (Caliper Life Science, Massachusetts, USA). Finally, the extract was transferred to a 2-ml vial and stored at –30 °C until further analysis by GC–MS/FID and sensory evaluation.

### 2.3. Solvent extraction

Coffee extract was prepared from 10 g coffee powder with a volume of 40 mL dichloromethane. The suspension was stirred by vortex shaker (Heidolph Rotamax 120, Schwabach, Germany) at 200 rpm for 1 h. The extract was then filtered from the ground coffee beans and dried over anhydrous sodium sulphate. The solvent was removed under purified nitrogen stream using TurboVap II until the volume of sample was reduced to 1 mL. The experiment was performed in triplicate and stored at –30 °C until used for analysis.

**Table 1**  
Face-centered central composite design (CCD).

Factor	Low (-)	High (+)	Centre
Temperature, $x_1$	50	100	75
Pressure, $x_2$	1000	2000	1500
Static extraction time, $x_3$	5	15	10

The design was a two-level full factorial design with 8 cube points, 6 centre points in cube, 6 axial points and alpha value 1.

### 2.4. GC–MS/FID analysis

GC–MS/FID analysis was carried out using Agilent 6890 N GC coupled with FID and a 5975 inert MS (Agilent Technologies, Palo Alto, CA, USA). In order to quantify the volatiles in the coffee bean extracts, 199  $\mu$ L of the sample extracts were spiked with 1  $\mu$ L of 20,000 ppm 5-methyl-2-hexanone (Sigma-Aldrich, Missouri, USA) as internal standard. One  $\mu$ L of the spiked extract was directly injected by an auto sampler (Gerstel Multi Purpose Sampler MPS, Mülheim an der Ruhr, Germany) into the GC injector under splitless mode, which was connected to a fused silica capillary column (60 m  $\times$  0.25 mm  $\times$  0.25  $\mu$ m DB-FFAP, Agilent Technologies, Woodbridge, USA) coated with 0.25  $\mu$ m film thickness of nitroterephthalic acid modified with polyethylene glycol. The injector temperature was 250 °C. The GC oven temperature was programmed from initially 50 °C for 5 min, then was raised to 230 °C at 5 °C/min and held for 30 min. FID temperature was set at 250 °C. Helium was used as carrier gas at a flow rate of 1.2 mL/min. The ionisation mode in the MS was electron impact (EI) mode at the ionisation energy of 70 eV, with turning performed using perfluorotributylamine. Identification of the eluted compounds was achieved by matching the mass spectra against NIST commercial library (Scientific Instrument Services, USA). Linear Retention Indices (LRI) values on a DB-FFAP column were determined by using a series of alkanes (C8–C40) (Fluka, Missouri, USA) run under identical conditions. LRI were further confirmed with the values of standard compounds and those reported in the literature [27–29].

The concentration of the compounds was expressed as parts per million (ppm) based on the relative FID peak area of each compound against internal standard with the response factor, which was previously determined with standard compounds under the same conditions. The relative response factors (RRFs) were calculated as:  $RRF = (M_{\text{compound}} \times A_{\text{IS}}) / (M_{\text{IS}} \times A_{\text{compound}})$ , where  $M_{\text{compound}}$  and  $A_{\text{compound}}$  are the mass and corresponding GC peak area of the compounds,  $M_{\text{IS}}$  and  $A_{\text{IS}}$  are the mass and GC peak area of the internal standard. For the commercially unavailable compounds, the RRFs were assumed to be 1.00.

### 2.5. RSM and statistical analysis

A face-centered central composite design (CCD) was constructed and analysed using the Design Expert Version 6.0.10 software (Stat-Ease, MN, USA), where the effects of three independent parameters on the selected key odorants [13,23–26] were taken as the response variables. Three main factors were selected as reported in the literature [16], i.e. temperature ( $x_1$ , 50–100 °C), pressure ( $x_2$ , 1000–2000 psi) and static extraction time ( $x_3$ , 5–15 min) (Table 1). The 20 runs were in triplicate in order to calculate the averages and standard deviations (Table 1).

The generalised response surface model to describe the variations in response variables is given as follows [12]:

$$y = \beta_0 + \sum_{j=1}^q \beta_j x_j + \sum_{i=1}^q \beta_{ij} x_j^2 + \sum \sum_{i < j} \beta_{ij} x_i x_j$$

where  $y$  is the predicted response;  $\beta_0$  is a constant;  $\beta_j$  is the linear regression coefficient;  $\beta_{ij}$  is the quadratic coefficient,  $\beta_{ij}$  is the interaction coefficient; and  $x_i$  and  $x_j$  are independent parameters. The adequacy of the model was determined by evaluating the coefficient of determination ( $R^2$ ) and lack-of-fit tests obtained from the analysis of variance (ANOVA). Statistical significance of the model and model terms were determined at 95% confidence level [12]. The terms found to be non-significant ( $p > 0.05$ ) were dropped from the initial model and refitted with the significant ( $p < 0.05$ ) independent parameters in order to obtain the final reduced model. However, some insignificant linear terms were retained in the model if a quadratic or interaction term containing this parameter was significant [30].

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