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A microwave-based extraction method for the determination of sugar and polyols: Application to the characterization of regular and peaberry coffees



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KEYWORDS

Carbohydrates; Green bean; Roasted coffee; Arabica coffee; Robusta coffee **Abstract** The brewing properties of coffee products are defined by the chemical composition in the bean, including sugars and polyols. Some factors, such as coffee species and roasting, may affect the level of these compounds in the bean. A new analytical microwave-assisted extraction (MAE) method has been developed to extract sugars and polyols from the coffee bean. The studied extraction conditions for the MAE were temperature (30-80 °C), solvent composition (0-50% ethanol in water), and solvent-to-sample ratio (10:1-30:1 mL solvent per g sample). A Box-Behnken design was applied to study the effect of extraction variables, and subsequently, the influential variables were optimized by response surface methodology (RSM). In addition to the main effect of the

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1878-5352 © 2021 The Authors. Published by Elsevier B.V. on behalf of King Saud University. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/). solvent-to-sample ratio, all quadratic effects significantly influenced (p < 0.05) the recovery of sugars and polyols from the coffee beans. RSM suggested the optimized MAE conditions: temperature 52 °C, ethanol concentration in water 18.5%, and solvent-to-sample ratio 17:1. Under the optimum condition, a kinetics study confirmed that 15 min showed high precision and accuracy of the developed method. Ultimately, a real sample application of the developed MAE revealed that the new method successfully described the composition of sugars and polyols in regular and peaberry coffee beans. Additionally, the method also effectively characterized the green and roasted Arabica and Robusta coffee beans.

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1. Introduction

Coffee is the most globally traded agricultural commodity (ICO, 2020). It is currently triggered by the availability of various downstream products, such as roasted coffee beans, ground decaffeinated coffee, and instant coffee powder. The brewing properties of these products are defined by the chemical composition in the coffee bean that may be influenced by species and the roasting degree (Farah, 2012; Illy and Rinantonio, 2005). The roasting effect on carbohydrates is inevitable as it represents a major compound of roasted coffee (39-52%). Furthermore, carbohydrates contribute to the physical properties of final coffee products, including viscosity enhancers that guarantee foam stability (Coelho et al., 2014; Nunes et al., 1997; Nunes and Coimbra, 1998). Some carbohydrates identified in coffee consist of sugars (mannose, glucose, and fructose) and polyols (xylose and inositol) (Lopes et al., 2020; Oosterveld et al., 2003). These compounds affect the sensory and health benefit of brewed coffee (Gloess et al., 2013; Simões et al., 2009).

In order to support further studies on the role of sugars and polyols in coffee beans and the derived products, a reliable analytical method for the determination of these carbohydrates is required. As a foremost step in the determination procedure, an accurate and precise extraction method should be developed. The presently available methods to extract sugars and other soluble compounds from coffee samples include sequential solid-liquid extraction using a basic solvent (Fischer et al., 2001), hydrochloric acid hydrolysis (Domingues et al., 2014), hot water extraction (Moroney et al., 2015), and the combined enzyme-steam assisted extraction (Pimpley and Murthy, 2021). However, the aforementioned methods contain some drawbacks, such as using a hazardous solvent like hydrochloric acid and time-consuming processes that result in high operational costs.

Several attempts to develop more efficient extraction methods were pursued. Previous research reports that the application of pressurized liquid extraction resulted in higher oil recovery from the spent coffee ground while performing a shorter extraction time than the conventional method (Toda et al., 2021). An improved extraction recovery of bioactive compounds from green coffee beans was also achieved due to some advances in green extraction methods, including ultrasound and microwave-assisted extraction (Pimpley and Murthy, 2021). The latter method has been proven to provide complete recovery of carbohydrates from foods in short times with a significant reduction of solvent volumes compared with conventional extraction methods (Zuluaga et al., 2020). The use of microwave-assisted extraction (MAE) as an advanced technique appears as a promising option to overcome the drawback of the existing extraction method (Khajeh, 2009). MAE could achieve a rapid increase in temperature due to a unique heating mechanism. The heating process occurs from the core of the solvent-matrix mixture, allowing the acceleration of heating simultaneously (Destandau et al., 2013). This particular property is the key to the efficiency enhancement of MAE as it results in the reduction of reaction time and solvent usage. Therefore, MAE exerts some advantages such as fast, eco-friendly, and low-cost operation (Ruiz-Aceituno et al., 2016). Hence, MAE is worth developing as an appropriate extraction method for coffee beans extraction.

In developing a reliable MAE method, factors affecting the analysis result must be evaluated and optimized to ensure correct identification and quantification of the target compounds. Most extraction kinetics were enhanced due to the increase of extraction temperature. However, applying temperature higher than 80 °C for a prolonged time can result in poor recovery rates due to the degradation of thermolabile compounds (Ameer et al., 2017; Fathimah et al., 2021). Apart from the operating temperature, the extraction solvent may also affect the extraction recovery. The dielectric constant of extracting solvent defines the absorbance of the microwave energy thus can be optimized to provide the right energy for the extraction system yet protect the target compounds from thermal degradation (Mandal et al., 2007). Additionally, MAE facilitates keeping the solvent usage as minimum as possible, therefore the solvent-to-sample ratio should be included in the optimization to meet the extraction procedure environmental friendly.

Because some factors are involved in the efficiency of MAE, Box-Behnken design (BBD) in conjunction with response surface methodology (RSM) can be helpful in the simultaneous evaluation and optimization of these factors. BBD is a suitable second-order multivariate design of experiments for the threelevel incomplete factorial designs. The design is more efficient for three factors evaluation than central composite design and three-level full factorial design in the aspect of the total experimental runs (Ferreira et al., 2007; Khajeh, 2009). In Addition, BBD is compatible with RSM to estimate the optimum level of the studied factors. RSM derives a model equation that fits the BBD data and calculates the optimal response. This statistical approach has been used for the optimization of carbohydrates recovery in different matrices (Fathimah et al., 2021; Lopes et al., 2020; Yang et al., 2017).

Therefore, based on the aforementioned challenge, this work aimed to develop an analytical MAE method for the

determination of sugars and polyols in coffee beans. Ultimately, the new developed MAE method was applied to determine sugars and polyols in different coffee samples covering regular and peaberry beans, Arabica and Robusta species, also green and roasted coffee beans.

2. Materials and methods

2.1. Chemicals and reagents

Analytical grade standard compounds (arabitol, erythritol, galactitol, glycerol, inositol, maltitol, mannitol, ribitol, sorbitol, xylitol, arabinose, fructose, galactose, glucose, lactose, maltose, mannose, rhamnose, saccharose, xylose, and cellobiose) were purchased from Sigma Aldrich Chemical Co. (St. Louis, MO, USA). Ultrapure water used in this research was produced using the Milli-Q system (Millipore Corp., Bedford, MA). Analytical grade reagents, including anhydrous sodium acetate, sodium hydroxide, and ethanol, were supplied from Sigma Aldrich Chemical Co.

2.2. Coffee samples

A roasted coffee sample (Hacendado, Ucc Coffee Spain S.L. U., Spain) was purchased from a local market in Cadiz, Spain, for the method development. The sample was ground in a coffee grinder (ML 130 Type SP-7406, Jata, Tudela, Spain) for 4 min with an on-off interval every 1 min. To check the applicability of the proposed MAE method, green and mediumroasted beans of the two most commercialized coffee species, *Coffea arabica* (Arabica) and *Coffea canephora* (Robusta), were collected from a local farmer (Kulon Progo, Yogyakarta, Indonesia) in the form of regular and peaberry beans.

2.3. Microwave-assisted extraction (MAE)

MARS 6 240/50 (CEM, Matthew, NC, USA) system was used to perform the extraction. The ground coffee sample was accurately weighed to match the solvent-to-sample ratio established for the optimization (10:1-30:1 mL solvent per g sample) and was placed in 100 mL Green Chem vessels (PTFE-TFM). Extraction solvent based on the design of experiments (0-50% ethanol in water) was added into the PTFE-TFM vessel. The samples were submitted to the MAE system at 800 W of microwave power for 15 min, while the extraction temperature was set according to experimental design conditions. After the extraction, the extract was separated from the solid material using a vacuum filter with Whatman filter paper No. 1. The remaining solid material on the vessel was rinsed out using a fresh solvent. The final volume of the combined extracts was adjusted to 50 mL with the rinsing liquid. The extract was subjected to filtration using a 0.45 µm nylon filter before the injection into the chromatography system.

2.4. HPAE-PAD conditions

Chromatographic analyses were performed with a highperformance anion-exchange chromatography (HPAE) system (Metrohm 930 Compact IC Flex, Gallen, Switzerland). The detection system was equipped with a pulsed amperometric detector (PAD) using a gold electrode as a working electrode. Elution was carried out using aqueous mobil phase containing 300 mM sodium hydroxide and 1 mM sodium acetate at a flow rate of 0.5 mL min⁻¹. The compounds were separated in a Metrosep Carb 2–150/4.0 column (Metrohm AG). Chromatographic analysis was recorded using MagIC net®, version 3.1 software (Metrohm AG). All samples were diluted 1:25 using the mobile phase before the chromatographic analysis ensuring the levels of the compounds fit the linear ranges of the calibration curves. The injection volume was set at 20 µL. Peaks were identified by comparing the retention time with those of standard compounds.

2.5. Design of experiment

Box-Behnken design (BBD) with three independent factors, each in 3 levels, including temperature (X_1 , 30–80 °C), solvent composition (X_2 , 0–50% ethanol in water), and solvent-tosample ratio (X_3 , 10:1–30:1 mL solvent per g sample) and three center points was established. Levels of the independent factors are listed in Table 1 and the design with a total of 15 experiments in randomized order is presented in Table 2. The response was the relative values concerning the maximum response (%) of the total concentration of the studied compounds in the samples.

Once the responses from the BBD had been obtained, a second-order polynomial function was built based on a response surface methodology (RSM):

$$y = \beta_o + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^k \sum_{j=1}^k \beta_{ij} X_i X_j + \varepsilon$$
(1)

where y is the dependent factor, i.e. the response (relative values for total sugars and polyols recovered from coffee beans); x is independent factors (temperature, x_i ; solvent composition, x_2 ; and solvent-to-sample ratio, x_3); β_o is the intercept; β_i are the first-order coefficients; β_{ii} the quadratic coefficients for *i*th factors; β_{ij} the coefficients for the interaction of factors *i* and *j*; and ε is the error. The regression coefficients were calculated based on a least-squared method.

2.6. Statistical analysis

The BBD construction and RSM analysis were performed using STATGRAPHICS Centurion XVI (Statpoint Technologies, Inc., USA) to achieve the optimum MAE condition providing the highest recovery. The experimental results from a single factor experiment in kinetics study were analyzed using an Analysis ToolPak added in Excel by Microsoft Office. The analysis of variance (ANOVA, p = 0.05) was used to determine the effect of the extraction time on the recovery. Subsequently, a least significant difference (LSD, p = 0.05) was tested to check the differences among the means.

2.7. Method validation

The analytical MAE method was validated following the ISO 17025 and ICH Guidelines (R1) (ICH, 2005; ISO, 2005). The linearity of calibration curves for inositol, glycerol, arabitol,

Table 1 The level of independent factors				
Factors	-1	0	+1	Unit
Temperature (X_I)	30	50	80	°C
Solvent composition (X_2)	0	25	50	% ethanol in water
Solvent-to-sample ratio (X_3)	10:1	20:1	30:1	mL solvent g sample ⁻¹

sorbitol, mannitol, glucose, xylose, fructose, lactose, cellobiose, and saccharose was validated with a high coefficient of determination (R^2 , higher than 99.77%). The limit of detection (LOD) ranged from 0.027 (sorbitol) to 1.51 (fructose) mg L⁻¹, while the limit of quantification (LOQ) ranged from 0.09 (sorbitol) to 5.21 (fructose) mg L⁻¹.

The precision assessments on the MAE method, including repeatability and intermediate precision, were performed. The intra-day repeatability was determined by analyzing nine extractions on the same day (n = 9). In contrast, the interday intermediate precision was based on three extractions in a day performed over three consecutive days (n = 3×3). The precision was expressed as the coefficient of variation (%CV) of the analyses. The extraction recovery (%R) was measured by comparing the concentration of each compound in the spiked sample before and after the extraction procedure with the concentration of the corresponding standard compound added in the sample.

3. Results and discussion

3.1. Sugars and polyols identification in coffee bean

Before developing a new method, a preliminary qualitative study was conducted to identify the compositions of sugars and polyols in a homogenous sample of roasted coffee beans. Extraction of a 2.5 g sample with 20 mL solvent of ethanol:water (1:1) by microwave-assisted extraction (MAE) at 50 °C in 10 min was performed. The collected extracts were analyzed using HPAE-PAD for sugars and polyols identification. The compounds were identified based on the comparison of the retention times of the peaks that appeared in the sample chromatogram to the peaks of twenty-one standards compounds (Table S1) that were used as references (arabitol, erythritol, galactitol, glycerol, inositol, maltitol, mannitol, ribitol, sorbitol, xylitol, arabinose, fructose, galactose, glucose, lactose, maltose, mannose, rhamnose, saccharose, xylose, and cellobiose).

Eleven carbohydrates were detected in the ground coffee bean sample, namely inositol, glycerol, arabitol, sorbitol, mannitol, glucose, xylose, lactose, cellobiose, fructose, and saccharose. These identified compounds were in common to those that have been reported to be presented in coffee samples of Arabica and Robusta during grain maturation (glucose, fructose, saccharose, and mannitol) (Rogers et al., 1999), throughout the roasting process (xylose, glucose, and inositol) (Flament and Bessière-Thomas, 2002; Oosterveld et al., 2003), and after the extraction processes (saccharose, glucose, fructose, inositol, sorbitol, and mannitol) (Clinton, 1986; Constantino et al., 2020). The other ten compounds of sugars and polyols were not detected in the ground coffee sample as the concentration was lower than the detection limits of the validated HPAE-PAD method.

Table 2	Design of experimental with t	heir observed responses.		
Run	Temperature (°C)	Solvent (% ethanol in water)	Ratio solvent-to-sample (mL solvent g sample ⁻¹)	Response* (%)
1	50	50	10:1	68.56
2	50	0	30:1	88.95
3	80	0	20:1	77.50
4	30	25	30:1	70.74
5	50	0	10:1	67.10
6	50	25	20:1	92.18
7	50	25	20:1	95.31
8	30	0	20:1	73.63
9	30	25	10:1	65.31
10	50	25	20:1	100.00
11	80	25	30:1	84.02
12	80	25	10:1	67.47
13	30	50	20:1	67.69
14	80	50	20:1	60.63
15	50	50	30:1	77.53

The relative value to maximum response (%) of the total concentration of sugars and polyols in the samples.

3.2. Effect of extraction factors

The effect of the experimental factors on the recovery of sugars and polyols from coffee bean samples was studied. MAE factors, including extraction temperature (X_1) , solvent composition (X_2) , and solvent-to-sample ratio (X_3) , were considered to be the most important parameters influencing the extraction process. Therefore, the individual contribution of these factors and the interaction effects between the factors on extraction recovery were studied. An efficient experimental design with 15 runs based on the Box-Behnken design (BBD) was performed (Table 2) to evaluate the MAE factors. The responses in varying MAE factors through BBD were recoveries indicated by relative values to the maximum response (100%) of the total concentration of sugars and polyols in the samples.

Analysis of variance (ANOVA) was used to calculate the significances of the temperature (X_1) , solvent composition (X_2) , and solvent-to-sample ratio (X_3) effects on the extraction recovery. The statistical significance for each extraction factor was evaluated by comparing the mean square against an estimate of the experimental error. The standardized values of main, interaction, and quadratic effects were plotted in a Pareto chart (p < 0.05) (Fig. 1). The corresponding bar crossing the vertical line indicated factors that significantly influenced the extraction recovery (p < 0.05).

The sole main effect that significantly influenced the extraction recoveries was the solvent-to-sample ratio (X_3) . As this factor contributed a positive effect, the increased ratio of the solvent-to-sample exerted a higher extraction recovery. A higher ratio of solvent-to-sample facilitates better the mass transfer of the analytes in the sample matrix into the solvent. However, the quadratic effect of solvent-to-sample ratio (X_3X_3) contributed negatively to the extraction recovery. A large increase in the solvent-to-sample ratio for the MAE could provide an excessive amount of polar solvent absorbing the microwave energy, thus reducing the energy reaching the sample then lowering the extraction yield.

Despite the fact that the main effect of temperature (X_1) and solvent composition (X_2) did not significantly influence the MAE recoveries, the quadratic effects of temperature (X_1X_1) and solvent composition (X_2X_2) provided negative influences on the sugar and polyol extraction. Due to a mild extraction temperature with 80 °C as the maximum level (Lopes et al., 2020) was applied to avoid compounds degradation, the studied range for temperature did not significantly influence the extraction recovery. However, an extreme increase (quadratic effect) of temperature (X_1X_1) led to a lower extraction recovery. Similarly, varying the solvent composition of 0 to 50% ethanol in water did not significantly alter the extraction recovery but the quadratic effect (X_2X_2) . A vast increase in ethanol concentration might not be favorable to the solubility of the very polar compounds. The estimated regression coefficients of the aforementioned effects were subsequently calculated and included in constructing the model for MAE factors optimization.

3.3. Prediction capability of the regression model

A mathematical model was constructed based on the significant effects suggested by the ANOVA to predict the optimum



 X_1 , extraction temperature; X_2 , solvent composition; X_3 ; solvent-to-sample ratio; X_1X_2 , interaction between extraction temperature and solvent composition; X_1X_3 , interaction of extraction temperature and solvent to sample ratio; X_1X_1 , interaction between solvent composition and solvent to sample ratio; X_1X_1 , quadratic of extraction temperature; X_2 X_2 , quadratic of solvent composition; X_3 X_3 ; quadratic of solvent-to-sample ratio.

Fig. 1 Standardized values of main, interaction, and quadratic effects of MAE factors on the extraction recoveries.

MAE experimental conditions for sugars and polyols extraction from coffee beans. The model fitted the responses after following the experimental combination generated by BBD to the polynomial equation. The resulting model for the proposed MAE technique was as follows:

$$Y = 89.09 + 6.13X_3 - 13.77X_1X_1 - 2.54X_2X_2 - 8.49X_3X_3$$
(1)

where y was the total concentration of sugars and polyols in coffee beans (% relative value to maximum response), X_i were the studied factors (X_i , temperature; X_2 , solvent composition; X_3 , solvent to sample ratio).

In order to ensure that the established model was capable of describing the observed data or whether a more complex model was required, a lack-of-it test was performed by comparing the variability of the current model residuals to the variability between observations at replicate settings for the factors. A higher p-value than 0.05 (0.41) was obtained for the lack-of-fit by ANOVA, indicating the model was suitable for the observed data at the 95.0% confidence level. The proposed model was also validated with a high value of the coefficient of determination (R^2 , 0.9499), explaining the reliability of the model in predicting the level of extracted sugars and polyols. Furthermore, the residual standard deviation was 3.65, and the sum of residuals was below 10^{-13} . As the prediction capability of the model was confirmed to be satisfactory to estimate the response, the model could be used to predict the optimum MAE conditions for the extraction of sugars and polyols from coffee beans.

3.4. Optimization of MAE factors

The three-dimensional response surface methodology (RSM) based on the proposed second-order polynomial model was plotted (Fig. 2) by assigning the level of sugars and polyols as the dependent factors in the *y*-axis against two independent factors in the *x* and *z*-axes, while the level of another independent factor was fixed at the centre point.

The RSM disclosed that the recovery of sugars and polyols increased (56 to 96%) while the temperature increased up to 52 °C (Fig. 2). This result confirmed that a higher temperature could enhance the solubility of sugars and polyols into the solvent, thereby increasing the extraction recovery. This finding was in agreement with a former report on the extraction of soluble sugars from green coffee beans evaluating extraction temperature ranging from 30 to 60 °C in an orbital incubator shaker. The optimum extraction temperature was 60 °C, as the higher extraction temperature was concluded to contribute to the solubilization of carbohydrates (Constantino et al., 2020). However, at the same time, further increasing temperature from the optimum point to 80 °C reduced the solvent density and consequently decreased the extracted sugars and polyols (Vatai, 2013).

An elevation of sugars and polyols recoveries also occurred as the ratio of solvent-to-sample escalated, reaching 17:1 (Fig. 2b). A higher ratio of solvent-to-sample provides the advantage of high mass transfer driving force of the analytes in the sample matrix into the solvent. A former study on MAE optimization for sugars and polyols in Spirulina revealed a similar response that applying a higher solvent-to-sample



Fig. 2 Response contour and surface plots showing effects of variables on the extraction yield of sugar and polyol from the coffee beans.

ratio provided better extraction recovery (Fathimah et al., 2021). In contrast, the recovery could not be further improved, although a higher ratio of solvent-to-sample was applied as the extraction was completed.

The RSM suggested the optimum solvent composition (Fig. 2a) and disclosed that the highest recovery for sugars and polyols was achieved by applying 18.5% ethanol in water. This result discloses that the high polarity of the solvent is favorable for the extraction of sugars and polyols from the coffee beans. The presence of adequate water proportion in the solvent composition could promote matrix swelling, facilitating the easier transfer of analytes from the matrix into the solvent (Dahmoune et al., 2021).

Henceforth, the optimum MAE condition for sugars and polyols extraction from the coffee beans can be achieved by applying the following experimental conditions: solvent-to-sample ratio 17:1, the solvent composition 18.5% ethanol in water, and extraction temperature 52 °C. Under these optimized MAE parameters, the amount of sugars and polyols $(1.49 \pm 0.07 \text{ g} 100 \text{ g}^{-1} \text{ of coffee beans})$ in the coffee bean samples could be measured.

3.5. Extraction kinetics

After the optimum extraction condition for the MAE factors was defined, a kinetics study was performed to evaluate the extraction time required to recover sugars and polyols completely from the coffee bean. Fig. 3 displays the yield of the extracted sugars and polyols from the coffee bean throughout the extraction time of 5 to 25 min.

The amount of extracted sugars and polyols was enhanced by prolonging the irradiation time. More prolonged exposure to microwave irradiation provide sufficient heat accumulation due to the microwave energy absorption by the extraction system that facilitates the dissolution of analytes into the solvent. In this case, a completed recovery with a more precise result was achieved at 15 min of extraction time. The full recovery of sugars and polyols from the coffee beans was confirmed as extending the extraction time for another 5 min showed a negligible change in the extraction yield. However, prolonging the extraction time to 25 min caused a reduction of the extracted sugar and polyol compounds. By extending the extraction time, a higher amount of interference compounds could also be extracted and later could interact with the sugars and polyols providing difficulties in recovering these analytes. Thus, 15 min was chosen to be the optimum extraction time using MAE.

Due to the application of microwave energy, the extraction time was enhanced from 8 h extraction by Soxhlet or 1.5 h by orbital incubator shaker (Constantino et al., 2020; Oosterveld et al., 2003). Additionally, the extraction kinetics of the new MAE was two times faster than the previously reported MAE for soluble sugars in coffee beans without an organic solvent (Constantino et al., 2020). As the utmost advantage of the MAE system is the possibility of performing multi-sample (up to 18 samples) at the same extraction time; therefore, the time needed to extract sugar and polyol compounds from each sample is less than 1 min.

3.6. MAE method validation

The method validation should be performed to evaluate the accuracy and precision of the new MAE method. The accuracy of the method was assessed through MAE recoveries (%R) which were defined by comparing the response of the coffee bean samples with and without spiked analytes. The standard solution of sugars and polyols used for spiking was also measured. The spiked and non-spiked coffee bean samples were extracted, and the targeted compounds were quantified on the resulting extracts. The recoveries ranging from 95.3% (cellobiose) to 106.2% (saccharose) were obtained. These recovery values were within the acceptable range by AOAC guidelines (80–110%), thus indicating the high accuracy of the developed method (AOAC, 2016).

Two levels of precision of the developed MAE method, namely repeatability and intermediate precision, were calculated. Both levels were assessed following the ICH guidelines (ICH, 2005). The precision, indicated as CV, of the MAE method was ranged from 0.29% (glucose) to 0.98% (lactose) for repeatability (n = 9) and 2.11% (mannitol) to 9.35% (sorbitol) for intermediate precision (n = 3×3). Since both CV values for repeatability and intermediate precision were below 10%, the developed MAE has been proved as a precise extraction method.

3.7. Real sample application

Once the proposed MAE method was validated by high precision and accuracy, the method was applied to extract a number of coffee bean samples to evaluate the applicability of the



Fig. 3 Extraction yield versus extraction time.

method. The resulting extracts were then measured for the concentration of sugar and polyol compounds in the real coffee samples covering the following parameters: type of the beans (regular and peaberry), coffee species (Robusta and Arabica), and processed bean (green and roasted). The concentration of sugars and polyols in the coffee beans sample is summarized in Table 3.

The determination of sugars and polyols in coffee beans is essential because the complex changes of these compounds during the roasting process could contribute to the organoleptic appeal of the final product. The content of saccharose, as the highest sugar compound in the green beans (6.2-17.8 g 100 g^{-1}), contributes to the sweetness level of the coffee beverage. However, the saccharose concentration was depended on the coffee species wherein Arabica was two times higher than the Robusta coffees beans. A former study and review also reported that Arabica contains a higher saccharose (6.3-11.4%) than the Robusta (0.9–4.85%) beans (Arya and Rao, 2007; Mondego et al., 2011). Interestingly, for Arabica species, the level of saccharose in peaberry was roughly 1.5 times higher than in the regular coffee beans. The different levels of saccharose in the beans apparently affect the consumption trend, where the Westerners generally prefer a richer coffee flavor of Arabica. In contrast, the Asian palate prefers the bitter Robusta coffees.

Saccharose has also been implicated as an essential precursor of coffee flavor and aroma because this compound degrades rapidly during roasting, forming various volatile compounds (Yeretzian et al., 2002). The result of the sample application revealed a decrease in saccharose content by 55-99% for both coffee species and bean types. This finding agreed to a report explaining that the hydrolysis of saccharose (by 47-59% in the case of medium roast and by 96-98% in the case of full city roast) to glucose and fructose occur during the roasting process (Diviš et al., 2019). Consequently, the level of glucose and fructose was relatively constant, most likely due to the degradation kinetics being similar to the rate of the saccharose hydrolysis. The reducing sugars, including glucose and fructose, may be reacted with amino acids (Maillard reaction) during the roasting process or further fragmented to aliphatic acids (Ginz et al., 2000).

On the contrary, the presence of the other sugars and polyols in coffee beans, both green and roasted, was found in a very low concentration. A sufficient limit of quantification of the developed method demonstrates the utility of MAE to extract the trace sugar and polyols compounds in coffee beans samples.

CRediT authorship contribution statement

Widiastuti Setyaningsih: Conceptualization, Methodology, Validation, Formal analysis, Resources, Writing – original draft, Supervision, Funding acquisition. Andika Wicaksono Putro: Investigation, Resources, Writing – original draft. Rohmah Nur Fathimah: Investigation, Writing – original draft, Project administration. Kiki Adi Kurnia: Visualization, Writing – review & editing. Noviyan Darmawan: Visualization, Writing – review & editing. Brian Yulianto: Validation, Writing – review & editing. Prastika Krisma Jiwanti: Writing – review & editing. Ceferino A. Carrera: Validation, Supervision. Miguel Palma: Conceptualization, Methodology, Validation, Formal analysis, Resources, Writing – review & editing.

Coffee 1	Jample			Concentration c	f sugars and poly	ols (g 100 g ⁻¹ \pm	SD)							
Code	Processed	Species	Type	Inositol	Glycerol	Arabitol	Sorbitol	Mannitol	Glucose	Xylose	Fructose	Lactose	Saccharose	Cellobiose
GRN	Green	Robusta	Normal	0.315 ± 0.011	0.140 ± 0.008	0.062 ± 0.006	0.001 ± 0.000	0.033 ± 0.005	0.063 ± 0.003	0.781 ± 0.023	0.063 ± 0.005	0.003 ± 0.001	7.296 ± 0.213	ND
RRN	Roasted	Robusta	Normal	0.605 ± 0.001	0.061 ± 0.001	0.014 ± 0.000	0.053 ± 0.001	Ŋ	0.044 ± 0.001	0.300 ± 0.010	0.052 ± 0.003	0.001 ± 0.000	0.969 ± 0.015	ND
GAN	Green	Arabica	Normal	0.424 ± 0.001	0.760 ± 0.006	0.020 ± 0.004	0.010 ± 0.000	0.121 ± 0.001	0.479 ± 0.005	0.870 ± 0.069	0.569 ± 0.002	0.018 ± 0.002	12.394 ± 0.016	ND
RAN	Roasted	Arabica	Normal	0.377 ± 0.019	0.819 ± 0.042	0.017 ± 0.003	0.002 ± 0.001	0.031 ± 0.001	0.312 ± 0.015	0.674 ± 0.033	0.394 ± 0.018	0.014 ± 0.002	2.645 ± 0.116	ND
GRP	Green	Robusta	Peaberry	0.373 ± 0.011	$0.132\ \pm\ 0.002$	0.019 ± 0.005	0.001 ± 0.000	0.052 ± 0.002	0.127 ± 0.003	0.579 ± 0.041	0.236 ± 0.002	0.033 ± 0.001	6.243 ± 0.253	ND
RRP	Roasted	Robusta	Peaberry	0.423 ± 0.030	0.085 ± 0.006	0.016 ± 0.002	0.003 ± 0.001	0.0174 ± 0.007	0.289 ± 0.183	1.824 ± 0.155	0.350 ± 0.207	0.009 ± 0.017	2.768 ± 0.163	ND
GAP	Green	Arabica	Peaberry	0.430 ± 0.176	0.105 ± 0.048	0.029 ± 0.011	0.004 ± 0.001	0.0462 ± 0.198	0.431 ± 0.106	1.207 ± 0.078	0.733 ± 0.124	0.013 ± 0.015	17.800 ± 0.025	ND
RAP	Roasted	Arabica	Peaberry	0.490 ± 0.022	0.073 ± 0.003	0.015 ± 0.001	0.004 ± 0.001	$0.0153\ \pm\ 0.005$	0.002 ± 0.002	1.098 ± 0.404	ND	ND	0.102 ± 0.000	0.055 ± 0.00
* ND:	not detected	, in which t	he concentration	n was lower than Lo	DD value.									

Sugars and Polyols Concentration of Coffee Beans Sample

Table 3

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.arabjc.2021.103660.

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