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# Optimization of Roasting Conditions for High-Quality Robusta Coffee

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### Abstract

Central Composite Design (CCD) was used to optimise temperature and time of Robusta coffee beans roasting. Current method of roasting was able to give good quality beans in term of flavour but the formation of acrylamide was not studied. In this study, optimization was based on high quantity of flavour compounds (pyrazines) with low level of acrylamide resulted in roasting temperature and time of 180°C and 26 minutes, respectively. The coffee beans produced using the optimized conditions have the following characteristics: colour; L\* 10.47-15.45, a\* 2.78-4.69and b\* 6.11-10.95; flavour compounds: 2,3,5 trimethyl pyrazine (0.832mg/100g), 2,3 dimethyl pyrazine (1.69mg/100g), 2 methyl pyrazine (0.49mg/100g) and 2,5 dimethyl pyrazine (0.62mg/100g) and low concentration of acrylamide (0.23mg/100g). This proposed roasting condition will be very useful for coffee manufacturers in order to produce high quality coffee beans.

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Keywords: Robusta coffee; pyrazines; acrylamide; colour; roasting.

#### 1. Main text

Commercially available coffee beans are derived from two genotypes i.e. *coffea Arabica* and *coffea canephora* var. *robusta* that are cultivated in many tropical countries [4]. Roasting is an important step in the production of coffee because it enables the development of flavour, aroma and colour. The temperature and time of roasting will influence the development of flavour compounds such as pyrazines. Coffee aromatic

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compounds are formed by the reactions that occur during roasting such as Maillard reaction, Strecker degradation, degradation of sugar and breakdown of amino acids. However, the formation of undesirable compounds such as acrylamide may also resulted from this process. Acrylamide is a well known carcinogenic compound which is formed mostly during food processing at very high temperature such as cooking, baking, roasting, frying and sterilization. Acrylamide has been classified by the International Agency for Research on Cancer (IARC) as probably carcinogenic to human (IARC, 1994). Acrolein could be one of the possible precursor of acrylamide beside from asparagine. Oxidation of acrolein to acrylic acid and subsequent reaction of acrylic acid with ammonia generated from the pyrolysis of nitrogen-containing compounds present in food, results in the formation of acrylamide [9]. The use of RSM in the process optimization leads to the need for an experimental design, which can generate a lot of samples for consumer evaluation in short period of time, and thus laboratory level tests are more efficient [10]. Central composite design (CCD) is the most useful design for estimating multifactor response surface which keeps the numbers of experiments to a minimum while allowing simultaneous assessments of variations of all the experimented factors studied and distinguishing the interaction among them [10]. The objective of this study is to optimise Robusta coffee beans roasting conditions that were able to produce superior quality coffee beans with low acrylamide formation.

#### 2. Materials and methods

#### 2.1. Sample preparation.

Dried wet process (WP) Robusta coffee beans samples were obtained from Malangsari Plantation, East Java, Indonesia.

#### 2.2. Roasting of coffee beans

Coffee beans were roasted using a roaster (PROBAT, Germany) with roasting conditions as suggested by Central Composite Design as shown in Table 1.

# 2.3. Grinding

Roasted coffee beans were finely ground in a coffee grinder.

#### 2.4. Analysis of acrylamide

Ground samples (5 g) were dissolved in 50 mL hot water and filtered using 0.45 mm Whatman filter paper. Solid Phase Extraction (SPE) C18 column was conditioned using 3 mL acetone and 3 mL formic acid. Filtered sample was applied to SPE tube at which the sample solution is allowed to pass through tube with gravity flow. SPE tube was washed using 2 mL distilled water and vacuum was used for 2 minutes to dry the excess water. Sample was eluted using 3 mL acetone. The eluted sample was filtered using 0.45  $\mu$ L syringe filter and kept at -20°C until further GC-FID analysis. Analysis of acrylamide was carried out using Gas Chromatography (Shimadzu 2010) equipped with Flame Ionization Detector with a temperature 260°C. RTX-5 column (30m x 0.25mm I.D. x 0.25  $\mu$ m film thickness) was used with injector temperature of 260°C, helium gas at constant pressure as carrier gas, and with oven temperature of 100 °C (hold 0.5 min) to 200°C at 15°C/minute [1].

#### 2.5. Analysis of flavour

Ground samples (5 g) were heated to 30 °C and SPME fiber Polydimethylsyloxane-Divinyllbenzene (PDMS-DVB) was introduced to the headspace of sample for 30 minutes. The fibre was reconditioned for 15 minutes into GC injection port. Flavour compounds (methyl pyrazine, dimethyl pyrazine, trimethyl pyrazine and tetramethyl pyrazine) were separated using GC-FID equipped with Rtx-5(*dimethylpolisiloxane crossbone*) capillary column, helium with 30 ml/min constant flow as carrier gas and injector SPL-1 operating in splitless mode. GC temperature programmed from 60°C (3 min) to 180°C at 5°C/min for 3 minute. Identification of the component of the standard was carried out by comparing the retention time of standards and quantification using the internal standard, 4-picoline [7]

#### 2.6. Determination of color

Measurement of colour after roasting was determined by using Chromameter (CR 400). The results were expressed in L\*a\*b colorimetric system according to the International Commision of Illumination (CIE), whereby L\* (luminance) represented quantity of reflected light and chromatic coordinated, a\* represented red-green axis while b\* represented yellow-blue axis.

#### 3. Results and discussion

The effect of two independent variables, A: temperature  $(210 - 150^{\circ}C)$  and B: time (15-40 minute) on seven response variables, 2-methylpyrazine, 2,3-dimethylpyrazine, 2,5-dimethylpyrazine, 2,3,5-trimethylpyrazine, 2,3,5,6-tetramethylpyrazine acrylamide and overall sensory evaluation were studied using Response Surface Methodology (RSM). CCD resulted in 14 treatments whereby the centre point was repeated six times to calculate the repeatability of the method. The arrangement of CCD for independent variables and responses is shown in Table 1. The summary of the results obtained from CCD is shown in Table 2. The adequacy of the model was determined by using model analysis, lack of fit test and coefficient of determination ( $\mathbb{R}^2$ ). The significance of the equation parameter was assessed by F value at probability (p>F) less than 0.05.

In this study, low  $R^2$  value obtained for acrylamide 0.77. According to Zaibunnisa *et al.* [10], for a good fit of a model,  $R^2$  should be at least 0.80. This result indicates that the presence of acrylamide in roasted cocoa beans was not influenced by roasting temperature and time probably due to the unstable characteristics of this compound. Acrylamide will form at high temperature but will be destroyed at extreme temperature. In green coffee, the amount of acrylamide measured increased rapidly at the onset of heating, reaching an apparent maximum, and then decreasing exponentially as the rate of degradation exceeds the rate of formation at 200 and 225°C [8].

All responses were based on quadratic model and non transformation. From this analysis, there are four marker compounds, 2-methylpyrazine, 2,3-dimethylpyrazine, 2,5-dimethylpyrazine and 2,3,5-trimethylpyrazine were found influenced by roasting temperature and time as their models are significant with not significant lack of fit and high R-squared (>0.90) (Table 2). As reported by Alessandre *et al.* [2], these compounds represent important classes of coffee aroma compounds and present in high concentration in roasted coffee aromatic oil analysed using Supercritical Fluid Extraction (SFE).

Table 1. Central Composite Design arrangement for independent variables A (Temperature, °C) and B (Time, minute) and their responses; 2-Methylpyrazine, 2,3-Dimethylpyrazine, 2,5-Dimethylpyrazine, 2,3,5-Trimethylpyrazine, 2,3,5,6-Tetramethylpyrazine, acrylamide and overall sensory evaluation.

| Temp<br>(°C) | Time (min) | 2-Methyl<br>pyrazine<br>(mg/100g) | 2,3-<br>dimethyl<br>pyrazine<br>(mg/100g) | 2,5-<br>dimethyl<br>pyrazine<br>(mg/100g) | 2,3,5-<br>trimethyl<br>pyrazine<br>(mg/100g) | 2,3,5,6-<br>tetramethyl<br>pyrazine<br>(mg/100g) | Acrylamide<br>(mg/100g) | Overall<br>sensory<br>acceptability |
|--------------|------------|-----------------------------------|---|---|--|--|-------------------------|-------------------------------------|
| 201.25       | 18.67      | 0.0986                            | 0.7816                                    | 0.2248                                    | 0.6140                                       | 0.7710   | 0.4640                  | 7.2                                 |
| 201.25       | 36.37      | 0.0556                            | 0.8802                                    | 0.1684                                    | 0.1896                                       | 0.4780   | 0.5758                  | 5.8                                 |
| 158.80       | 18.67      | 0.1156                            | 0.8802                                    | 0.1676                                    | 0.3896                                       | 0.4780   | 0.5565                  | 7.0                                 |
| 180.03       | 27.52      | 0.4816                            | 1.8392                                    | 0.7154                                    | 1.1708                                       | 1.1232   | 0.2358                  | 6.1                                 |
| 158.80       | 36.37      | 0.2416                            | 0.7434                                    | 0.1592                                    | 0.2920                                       | 0.7682   | 0.6342                  | 7.8                                 |
| 180.03       | 27.52      | 0.5070                            | 1.5476                                    | 0.5942                                    | 0.9148                                       | 0.9466   | 0.3464                  | 7.0                                 |
| 180.03       | 27.52      | 0.5406                            | 1.3938                                    | 0.5616                                    | 0.8010                                       | 0.9362   | 0.2698                  | 6.3                                 |
| 180.03       | 40.04      | 0.0666                            | 0.8530                                    | 0.1000                                    | 0.1708                                       | 0.1480   | 1.1984                  | 6.0                                 |
| 210.04       | 27.52      | 0.3082                            | 0.8334                                    | 0.3412                                    | 0.4280                                       | 0.3898   | 0.1828                  | 5.0                                 |
| 180.03       | 15.00      | 0.1554                            | 0.6638                                    | 0.1192                                    | 0.1104                                       | 0.4328   | 0.1790                  | 7.2                                 |
| 180.03       | 27.52      | 0.4578                            | 1.7672                                    | 0.5692                                    | 0.7742                                       | 1.2400   | 0.2968                  | 6.4                                 |
| 150.01       | 27.52      | 0.2740                            | 0.5102                                    | 0.1208                                    | 0.0822                                       | 0.2296   | 0.1501                  | 6.4                                 |
| 180.03       | 27.52      | 0.4228                            | 1.6780                                    | 0.5122                                    | 0.7294                                       | 1.3492   | 0.1088                  | 6.4                                 |
| 180.03       | 27.52      | 0.5268                            | 1.9680                                    | 0.7762                                    | 0.5966                                       | 1.2178   | 0.2808                  | 6.5                                 |

Table 2. Summary of Central Composite Design for roasting of Robusta coffee beans

| Compounds                       | Transform | Model                    | Lack of fit        | $\mathbb{R}^2$ | Equation <sup>a</sup>  | Significance model term |
|---------------------------------|-----------|--------------------------|--------------------|----------------|--|-------------------------|
| 2- Methylpyrazine               | None      | Quadratic<br>Significant | Not<br>significant | 0.93           | $2MP = 0.49 - 0.019A - 5.323E - 003B - 0.12A^2 - 0.21B^2 - 0.042B$                                   | $A^2$ , $B^2$           |
| 2,3-Dimethylpyrazine            | None      | Quadratic<br>Significant | Not<br>significant | 0.92           | $\begin{array}{l} 2,3DMP = 1.70 + 0.062A + \\ 0.029B - 0.49A^2 - 0.44B^2 + \\ 0.059AB \end{array}$   | $A^2$ , $B^2$           |
| 2,5-Dimethylpyrazine            | None      | Quadratic<br>Significant | Not<br>significant | 0.92           | $2,5DMP = 0.62 + 0.017A - 0.011B - 0.19A^2 - 0.25B^2 - 0.012AB$                                      | $A^2$ , $B^2$           |
| 2,3,5-<br>Trimethylpyrazine     | None      | Quadratic<br>Significant | Not<br>significant | 0.88           | 2,3,5TMP = 0.83 + 0.076A - 0.055B - 0.24A <sup>2</sup> - 0.30B <sup>2</sup> - 0.082AB                | $A^2$ , $B^2$           |
| 2,3,5,6-<br>Tetramethylpyrazine | None      | Quadratic<br>Significant | Significant        | 0.82           | $\begin{array}{l} 2,3,5,6TP = 1.14 + 0.029A - \\ 0.051B - 0.33A^2 - 0.34B^2 - \\ 0.15AB \end{array}$ | $A^2$ , $B^2$           |
| Acrylamide                      | None      | Quadratic<br>Significant | Significant        | 0.77           | Acrylamide = $0.26 - 0.013A$<br>+ $0.20B - 0.012A^2 + 0.25B^2$<br>+ $8.511E-003AB$                   | B, B <sup>2</sup>       |
| Overall sensory<br>Evaluation   | None      | Quadratic<br>Significant | Not<br>Significant | 0.80           | Overal sensory evaluation =<br>$6.46 - 0.46A - 0.29B - 0.19A^2 + 0.27B^2 - 0.53AB$                   | A AB                    |

The central composite design was generated using Design Expert 6.0 Software  ${}^{a}A$  = Temperature (°C), B= Time (minute)

The value of coefficient of determination (R<sup>2</sup>) for 2-methylpyrazine, 2,3-dimethylpyrazine, 2,5-

dimethylpyrazine, 2,3,5-trimethylpyrazine and overall sensory evaluation were 0.93, 0.92, 0.92, 0.88 and 0.80 respectively. The statistical analysis of the data were significant (p<0.05). The ANOVA also showed that there was a non-significant (p>0.05) lack of fit which validates the model.

In optimizing the roasting conditions, the goals were set at maximum level of flavour compounds (2methylpyrazine, 2,3-dimethylpyrazine, 2,5-dimethylpyrazine and 2,3,5-trimethylpyrazine, 2,3,5,6tetramethylpyrazine and overall sensory evaluation) and minimum level of acrylamide. Roasting temperature of 180°C and roasting time of 26 minutes are found to be the optimum conditions with desirability of 0.754. The concentration of 2-methylpyrazine, 2,3-dimethylpyrazine, 2,5-dimethylpyrazine and 2,3,5trimethylpyrazine obtained from this study also increased with temperature but it started to decrease after roasting temperature reached 180°C as shown in Figure 1 and 2.



Fig. 1. 3D surface plot for (a) 2-methylpyrazine and (b) 2.3-Dimethylpyrazine from the Central Composite Design (CCD).



Fig. 2. 3D surface plot for (a) 2,5-dimethylpyrazine and (b) 2,3,5-Trimethylpyrazine from the Central Composite Design (CCD).

## 4. Conclusion

Results obtained from this study indicate that four volatile flavours compounds; 2-methylpyrazine, 2,3dimethylpyrazine, 2,5-dimethylpyrazine, 2,3,5-trimethylpyrazine and overall sensory evaluation were significantly influenced by roasting temperature and time with optimum conditions of 180°C for 26 minutes. Under the studied roasting conditions, low amount of acrylamide was detected (0.23mg/100g).

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