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Production of instant coffee from cold brewed coffee; process characteristics and optimization

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Abstract

This study aimed to develop a process for producing instant coffee from cold brewed Arabica and Robusta roasted coffee beans. The process starts by selecting an appropriate mixture of Arabica and Robusta coffee beans, degree of roasting (light, medium, dark) and degree of grinding (coarse, fine). Sensory evaluation by a trained panel showed that a mixture of 92.5% Arabica and 7.5% Robusta medium roasted, coarsely ground coffee beans produces instant cold brewed coffee equally or better accepted from other instant coffees available to consumers. The selected coffee beans were subjected to vacuum assisted cold extraction and the extract was spray dried. Spray drying conditions (inlet temperature, air volumetric flow and air aspiration rate) were optimized by a 3^3 full factorial design. Response variables were solids yield, process thermal efficiency and anti-oxidant capacity of the produced powder measured as %DPPH scavenging ability, FRAP and total phenolic content. The produced powder was analysed for its physicochemical characteristics (caffeine content, phenolics, moisture content, hygroscopicity, bulk density, solubility, particle size distribution, acrylamide content and shelf life determination).

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Introduction

Coffee is a major commodity worldwide and one of the most popular beverages. It can be consumed for many reasons, including its stimulatory effects due to caffeine, anti-oxidant contents, health benefits, and primarily excellent taste and aroma (Butt and Sultan 2011). The major commercial varieties of the plant Coffea worldwide are Coffea Arabica (Arabica), Coffea Canefora (Robusta) and Coffea Liberia, which is only 1% of the global production (Chu 2012). Coffee has been the target of numerous studies in order to improve quality characteristics and sensory profile, to optimize processing methods and palatability. The most important step in coffee processing is probably the roasting step. There are three levels of roasting: light, medium and dark. Depending on the combination of time and temperature, roasting results in unique flavour and aroma development, but also affects the final concentration of various components such as HMF, acrylamide, phenolic acids and other antioxidants (Aguiar et al. 2016; Fuller and Rao 2017; Rao and Fuller 2018). Widely known type of coffee is the brewed coffee (aka French or American coffee), which is produced by extracting roasted ground coffee with hot water and using a filter to separate the extract from the ground coffee. Besides roasting conditions, other factors play an important role in the sensory characteristics of the final coffee beverage, such as varieties used, handling of the unprocessed green beans, storage conditions, degree of grinding and extraction conditions (Bertrand et al. 2008; Grembecka et al. 2007). A new coffee product has emerged the last eight years and is called "cold brew". This type of coffee brew is prepared using only cold water to extract the ground coffee, at a maximum temperature of 20ºC. Due to the low extraction temperature, this coffee has unique taste, flavour and differs in caffeine and acidity compared to hot extracted coffee. Hot brew tends to extract more non-deprotonated acids, while cold brew has been found to contain more caffeine than hot brew. Chlorogenic acids and pH were found to be similar (Fuller and Rao 2017; Rao and Fuller 2018).

The last few decades the production of instant coffee has been widely spread. Instant coffee is freeze or spray dried hot coffee brew extract. The processes involved in the production of instant coffee have been extensively studied and improved over the last few years, while other techniques have also emerged, such as vacuum belt drying or spray freezing (Burmester et al. 2012; MacLeod et al. 2006; Ratti 2001).

This study aimed to develop a process for producing instant coffee from cold brewed Arabica and Robusta roasted coffee beans. Employing sensory evaluation, an appropriate mixture of Arabica and Robusta coffee beans, degree of roasting (light, medium, dark) and degree of grinding (coarse, fine) were selected. The selected coffee beans were subjected to vacuum assisted cold extraction and the extract was spray dried. Spray drying conditions (inlet temperature, spraying air volumetric rate and drying air aspiration volumetric rate) were optimized by a 3^3 full factorial design. Response variables were solids yield, process thermal efficiency and anti-oxidant capacity of the produced powder measured as %DPPH scavenging ability, FRAP and total phenolic content. The produced powder was analysed for its physicochemical characteristics.

Materials and Methods

Raw materials and reagents. Roasted coffee beans (light and dark) of Arabica and Robusta variety were purchased from a local vendor. All chemicals and reagents were of analytical grade and obtained from Merck and LGC standards.

Preparation and sensory evaluation of instant coffee. Coffee beans (Arabica and Robusta at different ratios) were coarsely or finely ground with a commercial coffee grinder (De'Longhi KG89). Water was added (1:6, w/v) and vacuum assisted extraction at room temperature took place (1KPa, 300sec). The extract was filtered through a Whatman no1 filter paper under vacuum, concentrated to 14% total dissolved solids in a rotary evaporator (40ºC). The concentrated solution was pumped to a Buchi-B190 spray drier (inlet temperature 210ºC, 1.9×10^{-4} m³/s air flow (spray air), 50% aspirator or 0.004m³/s drying air). The produced powder was stored at a vacuum sealed plastic bag and stored at 4ºC till usage.

Samples for sensory evaluation were prepared as follows: 6 different combinations of Arabica and Robusta were used to prepare the instant coffee

powder (Arabica 70 to 100%, light or dark). 0.5 gr of each instant coffee were solubilized in 20mL water and served at room temperature to 6 trained panelists. As a reference sample, a known instant coffee was used. Each preparation was given a random 3 letter code, and panelists were asked to grade 25 attributes of the 7 different samples in a 0-10 scale according to Kreuml et al. (2013). The attributes (in aroma, taste and mouthfeel) and the reference material used to train the panelists are listed in Table 1.

Spray Drying Optimization and physicochemical characterization. Optimization of the spray drying conditions was performed by full factorial design (3^3 design) using the statistical software SPSS. Optimization variables were inlet temperature of air (130,180, 210ºC), spray air flow (1.4, 1.6 and $1.9x10^{-4}$ m^3/s) and aspirator power (50%, 70% and 90% corresponding to 0.004, 0.006 and 0.0075m^3/s of drying air respectively). Response variables were yield, thermal efficiency, phenolic (measured as total phenolics by Folin-Ciocalteu method) and caffeine content (HPLC), antioxidant activity measured as %DPPH scavenging ability and ferric reducing power (FRAP). Other physicochemical characteristics measured for all samples were bulk density, solubility, hygroscopicity, acrylamide and moisture content. Determination of the above properties was conducted according to the literature (Chandrasekar and Viswanathan 1999; Andrzejewski et al. 2004; Pérez-Hernández et al. 2012). Optimum spray drying conditions for maximum yield and phenolic content were able to be identified using the software's response optimizer. Physicochemical characteristics and shelf life determination of the instant coffee produced under optimum conditions were determined (Manzocco and Nicoli 2007; Nicoli et al. 2009). Mean size distribution of the instant coffee powder was determined by the Mastersizer (Malvern Instruments Ltd, UK).

For determination of the statistical important differences between the analyzed samples, the statistical software SPSS 15.0 (SPSS Inc., Chicago IL) was used.

Table 1. Sensory attributes and reference materials for training panelists

Attribute	Reference
Fruity	Apple juice
Hay like	Grass
Woody	Saw dust
Burnt	Caramelized sugar
Sour	Lemon peal
Nutty	Raw almonds
Roasty	Roasted almonds
Brew-Like	Freshly brewed Arabica
Sweet	5% sugar solution
Bitter	Bitter nut
Astringent	Quince

Table 2. Full factorial design for optimizing spray drying conditions.

| Inlet (ºC)	Aspirator (%)	Flow Control (10⁻⁴ m³/s)	Yield (%)	Phenolics (x10⁻³ mg equivalent gallic acid/g coffee)
130	50	1.4	31.6	75.3
130	50	1.6	40.8	70.1
130	50	1.9	55.0	70.7
130	70	1.4	25.3	74.9
130	70	1.6	32.8	72.1
130	70	1.9	56.0	79.8
130	90	1.4	35.6	70.1
130	90	1.6	44.5	70.1
130	90	1.9	56.9	73.9
180	50	1.4	34.4	60.1
180	50	1.6	43.2	76.3
180	50	1.9	59.7	73.2
180	70	1.4	37.4	79.4
180	70	1.6	43.6	81.5
180	70	1.9	52.3	83.7
180	90	1.4	35.3	73.4
180	90	1.6	39.8	75.3
180	90	1.9	55.9	69.6
210	50	1.4	36.8	80.6
210	50	1.6	40.3	87.4
210	50	1.9	54.6	82.2
210	70	1.4	31.9	87.3
210	70	1.6	40.7	84.9
210	70	1.9	53.8	84.5
210	90	1.4	34.9	88.6
210	90	1.6	42.5	83.8
210	90	1.9	50.7	80.6

Results and Discussion

Figure 1 is the arachnoid scoring plot comparing a beverage prepared from a known commercial brand and a beverage prepared from cold brewed instant coffee that was the most accepted by the panellists. Total likeness is similar for both samples (score 7.3 for both), while small only differences appear among the rest of the sensory attributes. The only statistical important difference was found in the "Burnt aroma" attribute, where the cold brew instant coffee had a lower score than the commercial brand. These sensory evaluation results are a strong indication that this new type of instant coffee has the potential to be widely accepted by the consumers. This particular sample (fgh) was prepared by blending dark roasted Arabica and dark roasted Robusta (92.5% and 7.5% respectively). This blend of coffee was used throughout the rest of the experiments. Experiments showed (data not shown) that degree of grinding did not play a role in the sensory evaluation of the final product. Other researchers (Cordoba et al. 2019) suggest that the degree of grinding in combination with extraction time might lead to different flavor profiles. In this paper, the extraction conditions were constant and ground coffee was extracted to completion, limiting therefore flavor variations. Furthermore, extracted coffee was spray dried, which can lead to some volatile loss. Optimization of the spraying drying conditions was performed by a 3^3 full factorial design. Detailed results of the design are listed in

Table 2. Statistical analysis of the results revealed that the main factor that affects %yield is the spray air flow, while the total phenolic content is mainly affected by the inlet temperature (figure 2b and 2c). Optimum conditions for maximum %yield and phenolic content are shown in Figure 2a.
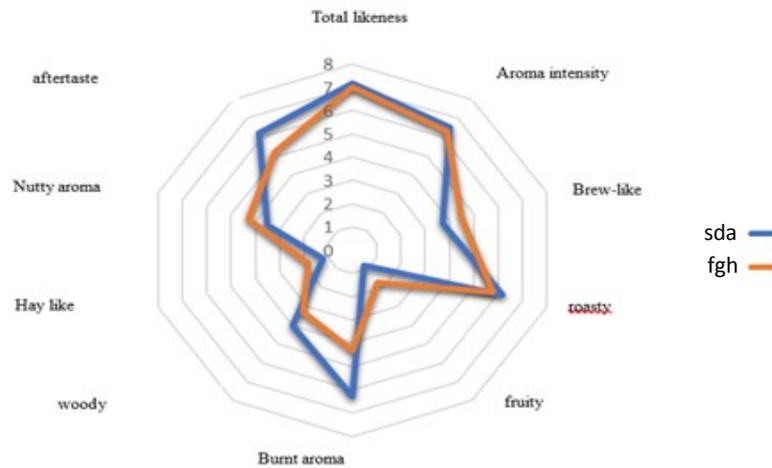
Figure 1. Sensory scoring plot of two beverage samples: a) sample "fgh" (orange line) is the coffee beverage produced from instant cold brewed coffee (92.5% Arabica and 7.5% Robusta) and b) sample "sda" (blue line) is instant coffee beverage produced from a known commercial instant coffee.
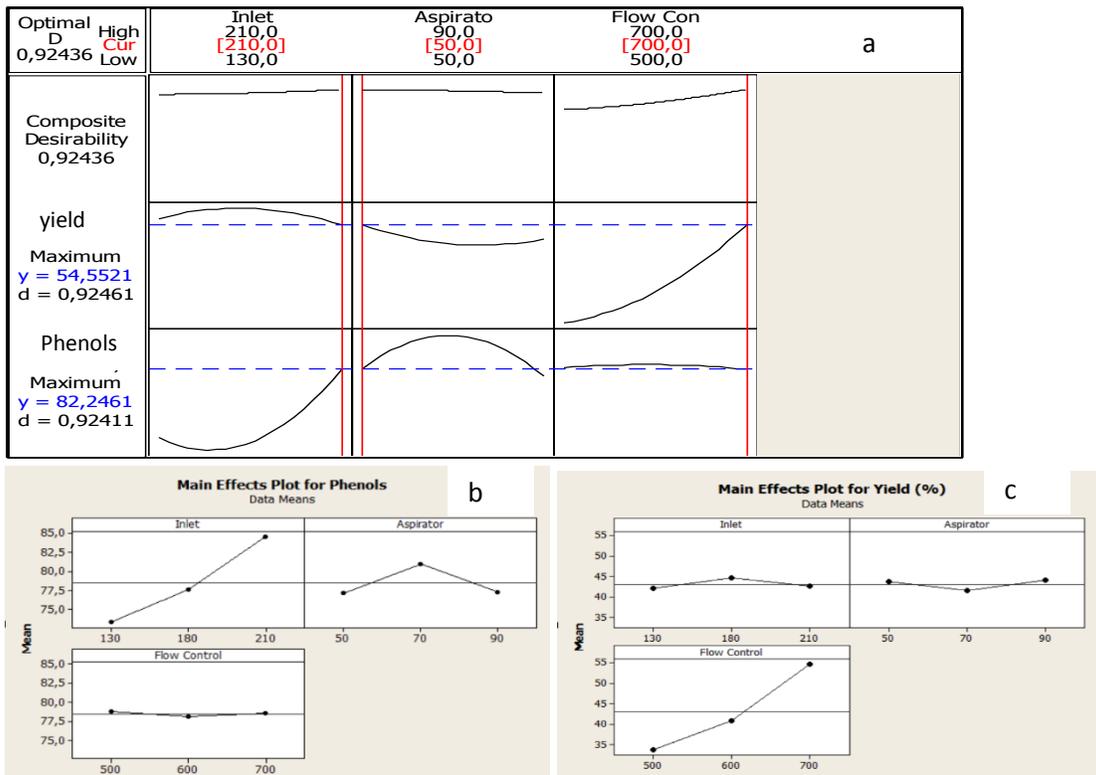
Figure 2. a) Optimization plot for yield and total phenolic content, b) Main effects plots for phenolic content and c) main effects plot for % yield.

Table 3. Optimal spray conditions and physicochemical properties of the produced instant coffee (92.5% Arabica and 7.5% Robusta) (reported values are the means of three samples)

Attribute	Value
Inlet temp	210 ºC
Spray air flow	1.9×10^{-4} (m³/s) or 700 Lt/hr
Drying air flow	15m³/hr
Yield	54,5%
Caffeine	35 mg/g coffee
Total Phenols	82.2 ppm
DPPH	86%
HMF	117 mg/Kg
Mean size of coffee particles	11.96 μm
Acrylamide	594 ppb
Moisture	2.34%
Bulk density	0,5 g/mL
Solubility	338sec
Spray drying thermal efficiency	54%
Shelf-life	18 months

Spray air flow, drying air flow and inlet temperature of the air are three of the main parameters affecting spray drying efficiency and the overall quality of the final powder. Feed concentration and rate also play an important role (Bansal et al.,= 2014; Goula et al. 2004). However, the last two parameters were chosen as constants and their values (14% total dissolved solids and 1.75g/min respectively) were experimentally determined as the maximum safe values so that the spray nozzle would not block. When the inlet temperature is increased, the yield is usually also increased. However, special care must be taken when drying thermally liable components, such as phenolics (Murugesan and Orsat 2011). Therefore, it was crucial to carefully optimize the operating conditions in order to achieve maximum yield and total phenolics at the same time.

The physicochemical properties of the produced coffee powder are presented in Table 3. Acrylamide had a concentration of 594ppb. It was interesting to notice that as the inlet temperature of spray drying was increasing, the acrylamide concentration was decreasing (4139 ppb at 130 ºC, 2250 ppb at 180 ºC and 594 ppb at 210 ºC). Investigations have shown that acrylamide is formed in the first minutes during roasting and gradually decreases with increasing roasting time, storage time, or exposure at high temperatures (Bagdonaite and Murkovic 2004; Bagdonaite et al. 2008). Regarding the HMF content of roasted coffee, a range of values between 300 and 2900 ppm is reported in literature. Increasing the temperature of the extraction water seems to increase the HMF content of the beverage (Mortas et al. 2017; Murkovic and Pichler 2006). The low extraction temperature employed in this research probably led to lower HMF content in the final product.

Hudáková et al. (2016) determined the total phenolic content and antioxidant activity (% DPPH inhibition) of several instant coffee varieties. Phenolic content varied in the range 31-78 ppm, while %DPPH inhibition in the range 56 to 90%. The instant coffee produced in the laboratory by applying the optimal spray drying conditions had total phenols 82 ppm and 85% DPPH demonstrating its high antioxidant capacity compared to other commercial types of coffee (Table 3).

According to Nicoli et al. (2009), 50% of the consumers find the sensory attributes of coffee and its derivatives unacceptable when $[H_3O^+] = 8 * 10^{-6}$ M. The reaction rate for the formation of $[H_3O^+]$ is given by the following equation:

$$[[H_3O^+] = -k * t + a \qquad (1)$$

Where k is the reaction constant, t is the storage time and α is a constant. The temperature effect on equation 1 is described by the Arrhenius equation

$$k = k_o * \exp(-\frac{E_a}{R*T}) \qquad (2)$$

Where k_o is a constant, R the universal gas constant, T the temperature and E_a the activation energy. E_a is in turn dependent on the water activity of the sample as described by equation 3 (Manzocco and Nicoli 2007)

$$E_a = a - b * aw^2 \qquad (3)$$

In order to calculate these constants, accelerated storage experiments were conducted at two different aw (0.43, 0.75) and three temperatures (10, 25 and 35 ºC). With the above equations it was feasible to calculate the shelf life of the produced instant coffee at 25 ºC at 18 months.

Conclusions

This research led to the production for the first time of an instant coffee from cold brewed coffee. Various parameters of the whole process were optimized. The most acceptable coffee blend was Arabica 92.5% and Robusta 7.5% leading to a beverage equally or better liked than a known commercial brand. The spray drying process was optimized in respect to yield and total phenolic content. The final product was fully characterized and its self-life was determined.

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