Food Chemistry 314 (2020) 126220

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Optimization of espresso coffee extraction through variation of particle sizes, perforated disk height and filter basket aimed at lowering the amount of ground coffee used

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ARTICLE INFO

Chemical compounds studied in this article:
Caffeine (PubChem: CID 2519)
Trigonelline (PubChem: CID 5570)
5-O-caffeoylquinic acid (PubChem: CID 5280633)
3-O-caffeoylquinic acid (PubChem: CID 1794427)
3,5-di-O-caffeoylquinic acid (PubChem: CID 6474310)

Keywords:
Particle size distribution (PSD)
Espresso coffee extraction
Filter baskets
HPLC-VWD
HS-SPME/GC–MS

ABSTRACT

Espresso machines maintain constant the extraction process of espresso coffee (EC), however, it is difficult to grind roasted coffee in homogeneous way. This research aims to investigate grinded beans at specific particle sizes in three variously designed filter baskets and to compare the concentration of bioactive compounds while decreasing the amount of ground coffee. Analyses on caffeine, trigonelline and chlorogenic acids are carried out with HPLC-VWD, while volatiles with HS-SPME/GC–MS. Extracting with smaller particles escalates the quantity of bioactive compounds. The amount of caffeine/cup increased moving from 500–1000 μm to 200–300 μm particle size, both in Arabica and Robusta for all filter baskets. Keeping constant the volume of EC at various heights of perforated disc, the amount of bioactive compounds at 12 g were only around 9% lower than at 14 g. The outcomes will support further studies on different extraction processes, to develop more sustainable and economically affordable coffee.

1. Introduction

Espresso coffee (EC) is the most consumed beverage in Italy (after water), more than 50 million cups being drunk daily (Severini, Ricci, Marone, Derossi, & De Pilli, 2015). The world coffee consumption is increasing every year, from 2014–15 to 2017–18 has incremented, so that the coffee industry needs to cope to a higher demand in a sustainable way. The sustainability of the coffee sectors particularly contemplates the protection of farming communities and their habitats, the reduction of the vulnerability, the conservation of natural resources (e.g. forest, soil, water) and the mitigation and adaptation to climate change. In this specific case, the environmental sustainability is mainly linked to a significant reduction in the waste production (e.g. packaging, coffee waste, plastic, energy consumption). Extracting EC with tantalizing aroma and rich mouthfeel requires certain optimal conditions as concerns the roasted coffee, temperature and water pressure, amount of grinded coffee, tamping pressure and impetus skills of barista (Kuhn, Lang, Bezold, Minceva, & Briesen, 2017). EC extraction has in fact great influence on trigeminal sensation (e.g., cooling, hot and tingling). Many different techniques for EC extraction and optimal parameters of brewing has been studied in depth (Caprioli et al., 2012, 2014; Folmer et al., 2017; Labbe, Sudre, Dugas, & Folmer, 2016; Parenti et al., 2014). As an instance, the infusion temperature has an important influence on the extraction of EC, because it generally impacts on

Abbreviations: DVB-CAR-PDMS, divinulbenzen-carboxen-polydimethylsiloxane; EC, espresso coffee; GC–MS, gas chromatography-mass spectrometry; HPLC, high performance liquid chromatography; HS-SPME head space, solid microextraction; LOD, limit of detection; LOQ, limit of quantification; PCA, principal component analysis; PD, perforated disc; PTFE, polytetrafluoroethylene; RSD, relative standard deviations; % RPA, percentage of relative peak area; VWD, variable wavelength detection

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https://doi.org/10.1016/j.foodchem.2020.126220
Received 12 September 2019; Received in revised form 3 January 2020; Accepted 13 January 2020
Available online 16 January 2020
0308-8146/

certain variables (specifically total solids, extraction percentage and concentration of bioactive compounds) (Andueza et al., 2003). Another important parameter is the pressure generated by the espresso machine, which can modify the cream of EC. To get more stable, dense and smooth cream, studies showed that optimal pressure for extraction of EC is 9 bar (Caprioli et al., 2014, 2015). In this respect, some researches inquired the consumers' preference for EC with or without cream, and they proved that those people liking EC without cream are in fact significantly fewer than those preferring EC with cream. These results were achieved through visual and in-mouth evaluation, confirming that the taste intensity is considerably higher in EC with cream (Labbe et al., 2016). One more important characteristic of EC is the flavour associated to acidity (or sourness). This acidity not only depends on the pH of EC, but also on the acidic perception of the consumer (perceived acidity). The measured pH values of EC range between 4.5 and 5.5, while the perceived acidity is defined by the total acidity, which in turn is determined by caffeine, trigonelline and chlorogenic acids. These bioactive compounds cause, in sensory analysis of EC, notes of bitterness and acidity (Parenti et al., 2014). Despite all the procedures and protocols, a sweet, clean, and intense taste and aroma in the cup is also dependent to a great extent on the grinding process, which can generate the best out of the bean, producing very different types of ground coffee. Some studies on coffee extraction show that different brewing methods (e.g. Turkish, French, or filtered coffee extraction) demand certain particle size distribution (PSD), preferably with similar average of the particle sizes, while this is not true for EC extraction. At any rate, due to the short extraction time of EC (Severini et al., 2015), beans grinded for espresso should contain at least a certain percentage of fines to achieve enough pressure in the coffee cake, as well as to produce a body and delicate cream (Folmer et al., 2017). Particle size of ground coffee plays in fact a crucial role in EC extraction. In general, particle size of grinded coffee is averagely called: fine, medium and course. According to this grinding classification, grinding machines normally can produce coffee particles from fine to course. Previous researches had demonstrated that the size of particles greatly influence on the extraction kinetics (Kuhn et al., 2017). In fact, some studies on comminution of particles had highlighted that bigger particles can ease the percolation during the brewing process. However, the fine particles generate intensity of taste, the only issue of fines being the possible clogging of the filter baskets (Blittersdorff & Klatt, 2017). Besides many independent variables, such as the ratio solid/water, and besides intrinsic factors associated with the quality of the coffee bean, such as coffee cultivation and roasting, even to customers it is evident that particle size of ground coffee can make significant alteration on the coffee extraction process and influence therefore the quality of EC (Petracco, 2001). Based on the size of particles, a certain extraction accessory (e.g. filter baskets, distributor and tamping), should be used and tuned in a specific way, in order to extract a good EC (Spiro & Selwood, 1984). However, researchers have not studied yet in depth how different tools can be adjusted complementarily in coffee extraction, as how different filter baskets could be chosen according to the grinding burr that produces different particle size of ground coffee. The objective of the present study is to investigate grinded beans at specific particle sizes (from 200 μm to 1000 μm) in three variously designed filter baskets, and to compare the concentration of bioactive compounds while decreasing the amount of ground coffee of a double EC extraction (from 14 g to 12 g). A second objective is to use various heights of perforated disc under the shower (4–7 mm), and to extract coffee with 14 g and 12 g of ground coffee. By combining these two operations, it is possible therefore to optimize the extraction of EC.

2. Materials and methods

2.1. Chemicals

Standards of caffeine, trigonelline, 5-O-caffeoylquinic acid (5-CQA), 3-O-caffeoylquinic acid (3-CQA) and 3,5-di-O-caffeoylquinic acid (3,5-diCQA) were purchased from Sigma-Aldrich (Milano, Italy).

Divinylbenzene-carboxen-polydimethylsiloxane (DVB-CAR-PDMS) Stable Flex fiber of 50/30 μm was acquired (Supelco, USA) and used for the HS-SPME. Formic acid (HCOOH – 99%), cyclohexane (C_6H_{12} – 99%), methanol (CH_4OH – HPLC gradient) were from Sigma-Aldrich (Milano, Italy).

2.2. Coffee samples and espresso machine

Roasted coffee samples (Arabica 100% – "Le Piantagioni Del Caffe", and Robusta 100% – Bali) of certified geographical origins from two production areas (America and Asia) were suggested by certified roasters for EC preparation, while the fully automatic espresso machine (Vittoria Arduino, VA388 Black Eagle) and the grinding machine (Mythos 1) were provided by the espresso machine manufacturing company Simonelli Group SpA. (Belforte del Chienti, Italy). From now on, the two different coffee cultivars used for the EC samples, are simply referred to as *Arabica* and *Robusta*.

2.3. Grinding and sieving

The Arabica and Robusta medium roasted coffee samples were brought under sealed packages. Packages were opened just before grinding. All coffee beans were milled between fine and medium sizes and were separated by sieve plates (vibrational sieve machine, Retch, AS 200 Control, Germany). The grinded coffee was separated into sieve plates with 200–300 μm, 300–400 μm, 400–500 μm and 500–1000 μm. An analytical scale (Gibertini, Crystal Series analytical scale, Italy) was used to weigh 12 and 14 g of separated ground coffee.

2.4. Particle size analysis

Coffee beans, after passing comminution process, were analysed by Mastersizer 3000 Aero Series dry dispersion units (Malvern PANalytical Ltd., UK), which uses a laser diffraction to measure the size of particles (from 0.01 to 3500 μm). The instrument operates with continuous air flow, generated by industrial compressor at 6.5 bar, that penetrates into Aero dry dispersion units, which transfers the particles at 2–3 bar to laser diffraction. In this way, the particles move in laminar flow and the vacuum extraction unit (KARCHER Professional NT 45/1 Tact, Germany) removes samples from Aero dry. The grinded and separated coffee powder with various particle sizes were collected: one fifth for Mastersizer 3000 and the rest for extraction of espresso coffee. The size of particles for each sample were examined in fivefold and the mean value was used for comparison.

2.5. EC sample extraction

Separated microns (200–1000 μm) and weights (12–14 g) of ground coffee were transferred in three different filter baskets (***A*** – standard filter basket for Italian espresso coffee, ***B*** – small sized filter basket, ***C*** – particular design of filter basket) and were used to test concentration of bioactive compounds. The extraction conditions of EC at 12 and 14 g were maintained constant, especially with regard to the distance between the coffee cake and the shower, by modifying the height of the perforated disc (4 mm-7 mm). The espresso machine parameters were 93 ± 2 (°C) and 9 bars. In accordance to research studies about water influence on EC quality (Navarini & Rivetti, 2010), the utilized water was bought always from the same commercial brand and was a minimally mineralized water. This water is commercially available and its parameters are: total mineralization 22.0 mg/L: HCO_3^- – 9.5 mg/L; Ca^{2+} – 2.8 mg/L; Mg^{2+} – 0.45 mg/L; SiO_2 – 7.3 mg/L; NO_3^- – 1.0 mg/L; Na^+ – 1.8 mg/L; SO_4^{2-} – 3.6 mg/L; Cl^- – 0.21 mg/L; K^+ – 0.20 mg/L; F^- – < 0.10 mg/L. The extraction of EC for each filter basket was performed in triplicate (200/1000 μm with 12/14 g) and

was implemented in two ways: first, the extraction time was kept constant for all different particles and amount of ground coffee filter baskets; and second, the extracted volume was maintained constant. These parameters were automatically adjusted by the program of the espresso machine (25 ± 2 s and 50 ml of for double espresso). In both cases, the brew ratio between g of coffee cake and ml of EC was kept 1:2 ± 0.2. All extracted EC samples were immediately collected from the portafilter of the espresso machine in a ceramic espresso cup, and the weight of the extracted EC samples was measured by Hario and Acaia balance.

2.6. Total dissolved solids (TDS)

The total dissolved solids (expressed as mg l^{-1}) were analysed following a developed procedure (Parenti et al., 2014; Bell, Wetzel, & Grand, 1997): the analysis of TDS was performed in the oven by drying 1 ml of EC to a constant weight (24 h, 100 °C). This procedure was carried out for extracting EC with various particle sizes and for analysing EC extracted with various heights of perforated discs. The results of TDS were estimated according to the amount of dry coffee residue.

2.7. Sample extraction and HPLC-VWD analysis

1 ml of each EC sample was diluted 50 times in the mobile phase. The solution was centrifuged at 13,000 rpm for 10 min and filtered at 0.45 μm (PTFE filter) before HPLC-VWD analysis (Caprioli et al., 2014). The analytical column Gemini C18 110A (250 × 3 mm I.D., 5 μm, Phenomenex, Cheshire, U.K.) was used for separation. The mobile phases for the analysis of HPLC-VWD were 0.3% of formic acid with water (A), and methanol (B). The flow rate was 0.4 ml min^{-1} with gradient elution. The gradient program (0 min, 25% B; 0–10 min, 60% B; 10–15 min, 60% B; 15–20 min, 25% B) was held at 25% B until the end of the run at 25 min. The injection volume was 10 μl and HPLC-VWD experiments were carried out using a Hewlett Packard (Palo Alto, CA, USA) HP-1090 Series II, made of an autosampler and a binary solvent pump, equipped with a variable wavelength detector (VWD). HPLC-VWD analyses were performed at two different wavelengths in the same run: 265 nm for trigonelline and 270 nm for caffeine. For chlorogenic acid (CGA) derivatives analysis, an analytical column Polar-RP 80 Å (150 × 4.6 mm I.D., 4 μm) from Phenomenex (Chesire, U.K.) was used for separation. The mobile phases for analyses of HPLC-VWD were: 0.1% of formic acid in water (A), and 0.1% of formic acid in methanol (B). The flow rate was 1 ml min^{-1} with gradient elution. The gradient program (0 min, 25% B; 0–10 min, 60% B; 10–15 min, 60% B; 15–20 min, 25% B) was held at 25% B until the end of the run at 25 min. The injection volume was 5 μl and HPLC-VWD analyses were carried out at 345 nm wavelength in the run for 3- Caffeoylquinic acid (3-CQA), 5-Caffeoylquinic acid (5-CQA) and 3,5- di-Caffeoylquinic acid (3,5-diCQA) (Caprioli et al., 2013).

2.8. Head space solid-phase microextraction (HS-SPME)

Sample injection techniques with HS-SPME was implemented through PAL3 auto sampler system. Polydimethylsiloxane (PDMS) fiber (Sigma Aldrich, Milan, Italy; 10 μm thickness) was in HS-SPME. For the analysis, 2 ml of EC sample was placed in a screw top vial. The vial was tightly screwed on magnetic cap with septum and the system was set for automatic functioning mode. The sample was placed into a stirrer, incubated at 60 °C, and stirred at 250 rpm for 20 min. Then, HS-SPME automatically was inserted in the sample and remained 20 min for adsorption. After adsorption, HS-SPME automatically injected the analytes into the gas-chromatographic system. A desorption time of 10 min was sufficient to desorb analytes from the fiber. Cleaning was automatically performed with PAL system by inserting the fiber in the conditioning port at 230 °C for 20 min after each process.

2.9. GC–MS analysis

A gas chromatograph/mass selective detector (GC/MSD – Agilent, Santa Clara, CA, USA, Agilent 7890B GC Hardware with Agilent 5977 Series MSD and Mass Hunter GC/MSD Data Acquisition) was used. The column used for separation was DB-WAX (0.25 mm × 60 m × 0.25 μm – Agilent 122–7062, CA, USA). The workstation in the GC–MS system was an Agilent Chem. The flow rate (He) was 1.2 ml min^{-1} under splitless mode. The temperature of the injector was 260 °C. The temperature for the column was programmed: from 35 °C (4 min) to 120 °C (2.5 °C per min), and from 120 °C to 250 °C (15 °C per min); then, 250 °C for 3.33 min remained plateau and the total run time was 50 min. Data were acquired through the electron impact (EI) mode and the total ion chromatogram monitoring (TIC) scan mode.

2.10. Statistical analysis

Data on selected volatile compounds were examined by principal component analysis (PCA) using Statistica v.7.1 (Stat Soft Italia, Vigonza, Italy). PCA was applied in order to visualize information at various particle sizes that were used for EC extraction in three filter baskets.

3. Results and discussion

3.1. Particle size distribution, and instrumental analysis of grinded coffee

The analysis of particle size distribution was implemented with Mastersizer 3000 instrument. The process of analysis was rapid due to the laser diffraction. Measurements were repeated fivefold for single sample and the mean value was used to create the Gaussian graph. The graphs (Fig. 1S and Fig. 2S – supplementary material) deliver information of particles through percentage of volume density. The percentage of volume density comes through scattered light, because large particles scatter light at small angles with the laser beam, whereas small particles scatter light at large angles. This angular scattering intensity data are used to calculate the size of particles. Data analysis is based on the Mie theory (Do, Hargreaves, Wolf, Hort, & Mitchell, 2007). Fig. 1S and Fig. 2S highlights that size of particles separated through vibrational sieve are distinguishable. These results show differences of particles (from 200 μm up to 1000 μm) used to extract EC with 14 g and 12 g. The various percentages of volume density in both figures in similar micron range are superior and inferior due to the prior extraction of EC during calibration, where mixed particles are used for brewing at 25 s with 25 ml of EC. Afterwards, the grinding condition was kept constant. Eventually, the grinded coffee was filtered through vibrational sieve and the separated particles of different microns were utilized for extraction of EC. The average size of particles between 200 and 300 μm was 256 μm, 300–400 μm was 325 μm, 400–500 μm was 440 μm and 500–1000 μm was 580 μm.

3.2. Results of total dissolved solids (TDS)

In general, consumers description of the “strength” term in coffee can be characterized as its overall concentration of beverage, which in chemical terms broadly associate with the total solids content of coffee (Petracco, 2001). Three filter baskets and perforated discs were applied to analyse total solids in 14 g and 12 g of grinded coffee for extraction of EC.

Table 1 presents the results of TDS in EC extracted with various particle sizes of ground coffee (between 200 μm and 1000 μm). Before separating different particle sizes, mixed particles of ground coffee were put in each filter basket directly from the grinding machine, as a calibration of the EC extraction. The resulting EC samples were used as a reference for the rest of samples extracted with different size of particles. TDS in Arabica coffee, where 14 g was used to extract EC with

Table 1
Total dissolved solids (mg per l of coffee) in Arabica (n = 3, RSD% < 3.18) and Robusta (n = 3, RSD% < 4.14) EC samples, by using 12 and 14 g of ground coffee with various particle size distribution in three different filter baskets.

	Particle Sizes (μm)	Total Dissolved Solids (TDS, 10^3 mg/l)					
		Ground cofee (14 g)			Ground cofee (12 g)		
		Filter A	Filter B	Filter C	Filter A	Filter B	Filter C
Arabica	Mixed particles	44.6	52.2	35.96	46.5	44.3	43
	500–1000	29.15	69.8	29.2	15.5	13	24.35
	400–500	35.15	66.1	25.1	16.8	17.85	20.75
	300–400	32.1	40.1	23.7	22.75	23.35	22.1
	200–300	53.7	35.16	34.15	29.05	29.85	24.35
Robusta	Mixed particles	77.8	63.2	57.55	54.55	59.35	37.55
	500–1000	35.4	17.15	27.6	19.8	17.05	19.8
	400–500	20.85	19.85	24.9	21.9	20.25	11.1
	300–400	14.95	18.15	19.65	14.25	13.05	14.9
	200–300	135.55	120.95	33.15	130.8	46.9	46.35

filter baskets ***A*** and ***C,*** had stable growth from above 500 μm to 200–300 μm particle size. This trend performs vice versa in the case of filter basket ***B***. The microns between 500 and 1000 μm and 400–500 μm have higher TDS content: 69.8*10^3 mg l^{-1} and 66.1*10^3 mg l^{-1}, respectively, and when the size of particles became fine the TDS result was nearly halved. However, in the range from above 500 μm to 200–300 μm, when the amount of ground coffee was 12 g, TDS has a stable growth in filter baskets ***A*** and ***B,*** from 15.5 to 29.05 * 10^3 mg l^{-1} and from 13 to 29.85 * 10^3 mg l^{-1}, respectively. Instead, in filter basket ***C***, not a big change was observed from above 500 μm to 200–300 μm.

On the other hand, Robusta coffee that was used to extract 14 g yielded the highest TDS in Filter ***A*** and ***B*** in the range 200–300 μm. In these filter baskets, the amount of TDS found, (135.55 and 120.95 * 10^3 mg l^{-1} respectively) were considerable higher with respect to values found when the size of particles were in the range 400–300 μm, 500–400 μm and 1000–500 μm. Robusta coffee that was used to extract 12 g yielded the highest TDS in the range 200–300 μm, i.e. 130.8 * 10^3 mg l^{-1} for filter ***A***, 46.9 * 10^3 mg l^{-1} for filter ***B*** and 46.35 for filter ***C.*** For both blends, higher TDS were observed when 14 g of coffee was used for preparing EC samples.

Table 2 shows the TDS results when various heights of perforated discs were applied to extract EC, one with constant time (25 s for double EC) and the other with constant volume (50 ml for double EC). The outcomes prove equal levels of TDS at time constant condition for EC using 14 g with 4 mm of perforated disc (PD), and 12 g with 5 mm of PD (84 * 10^3 mg l^{-1} and 84.5 * 10^3 mg l^{-1}, respectively). When the volume was constant, the same results were achieved using 14 g with 5 mm of PD, and 12 g with 7 mm of PD (83.5 * 10^3 mg l^{-1} and 83.8 * 10^3 mg l^{-1}, respectively). By comparing constant time and volume cases, the equal TDS data, obtained when the amount of coffee in the basket was 12 g, were at PD 4 mm and 7 mm with almost 74 * 10^3 mg l^{-1} (time constant), and at PD 4 mm and 6 mm with around 74 * 10^3 mg l^{-1} (volume constant).

It is evident that decreasing the amount of coffee allow to obtain similar amount of TDS when increasing the heights of the perforated disk.

Table 2
Total dissolved solids (mg per l of coffee) in EC samples with *Time constant* (n = 3, RSD% < 2.80) and *Volume constant* (n = 3, RSD% < 3.76) by using 12 and 14 g of ground coffee for filter basket ***A*** with various perforated disc heights.

Perforated Disc (mm)	Total Dissolved Solids (TDS, 10^3 mg/l)			
	Ground coffee (14 g)		Ground coffee (12 g)	
	Time constant (25 *sec*)	Volume constant (50 ml)	Time constant (25 *sec*)	Volume constant (50 ml)
4	84.0	86.3	74.0	73.8
5	97.0	83.5	84.5	77.0
6	79.0	74.0	86.3	74.5
7	88.0	78.5	73.5	83.8

3.3. Method validation for HPLC-VWD

The validation of the method has been carried out in relation to linearity, repeatability and with-in reproducibility, limits of detection (LODs) and limits of quantification (LOQs) (Caprioli et al., 2014). Prior to analysis, calibration curves of identified compounds at five different concentrations, i.e. 10, 20, 50, 100 and 250 mg l^{-1}, were prepared by injecting 10 μl from standard solutions. Relative standard deviations (RSDs) of each concentration ranges from 0.33 to 3.17%. A correlation curve for each analysed compound indicates a value greater than 0.9996. Analysis of EC in HPLC-VWD was carried out by obtaining standard mixture of caffeine, trigonelline and chlorogenic acids in the following concentrations: 10 and 50 mg l^{-1}. Within these standard concentrations, method repeatability and calculations (n = 8) demonstrated accurate and moderate data of RSD %, with a range of 3.52–11.01% and 0.74–4.44%. The LODs and LOQs of three compounds (estimated in matrix and indicated in mg l^{-1}) are calculated in signal to noise ratio of 3:1 and 10:1 S/Ns. For nitrogenous compounds LODs and LOQs are in the range of 0.03–0.06 and 0.1–0.2 mg l^{-1}. The specificity of the method is verified by the retention time stability.

3.4. Caffeine, trigonelline and chlorogenic acids in EC

The effect of particle sizes on EC extraction in different filters has been studied. EC samples (thirty samples of Arabica and thirty samples of Robusta for different particle size with three different filters, and eight samples of Arabica with various heights of the perforated disc) were analysed for detecting caffeine, trigonelline and derivatives of chlorogenic acids (3-Caffeoylquinic acid, 5-Caffeoylquinic acid and 3,5-di- Caffeoylquinic acid). The extraction of EC was performed in triplicate in the same conditions for each filter and each particle size (Tables 3 and 4).

Preliminarily, for each filter basket, the grinding machine has been tuned and calibrated so as to obtain optimal EC without separating ground coffee by sieves. To extract EC with 14 and 12 g of ground coffee, the variables of the espresso machine were set at the following conditions: 25 s at 93 ± 2 °C and 9 bars. After calibration, grinded coffee was separated by a vibrational sieve. From each different sieve, 14 and 12 g of ground coffee were used for extraction. According to previous research studies (Severini et al., 2015; Gloess et al., 2013; Labbe et al., 2016) particle size influences on the amount of the extracted bioactive components. In fact, the average caffeine and trigonelline quantities in milligrams in the cup are higher in the range 200–300 μm particle size, because milling coffee in smaller particles allows for a more efficient extraction of biocomponents. On the other hand, reducing the mass of grinded coffee from 14 g to 12 g decreases the number of bioactive components with all the three filter baskets and differently grinded particles. Results show therefore that the content of bioactive components is dependent on the relative particles size, as well as on the amount of the ground-coffee mass in the filter baskets.

In Table 3, when the size of particles (200 μm–500 μm) and the amount of ground coffee (14 g–12 g) were kept constant, a significant alteration on bioactive compounds amount was highlighted by using different filter baskets. Filter basket ***A,*** compared to baskets ***B*** and ***C***, allowed a gradual stable rise of all biocomponents in most cases and samples, with the exception of 400–500 μm in Arabica (14 g) and 500–1000 μm in Robusta (14 g). For instance, caffeine in Arabica (14 g)

Table 3
Milligrams of caffeine and trigonelline obtained in EC samples of Arabica (n = 3, RSD% < 7.22) and Robusta (n = 3, RSD% < 8.26) and milligrams of chlorogenic acids (3-CQA, 5-CQA and 3,5 di-CQAs) obtained in EC samples of Arabica (n = 3, RSD% < 6.86) and Robusta (n = 3, RSD% < 7.33), by using 12 and 14 g of ground coffee with various particle size distribution for each of the three filter baskets.

			Ground coffee (14 g)						Ground coffee (12 g)					
			Trigonelline (mg)	Caffeine (mg)	3-CQA (mg)	5-CQA(mg)	3,5-di-CQA (mg)	Total CQA (mg)	Trigonelline (mg)	Caffeine (mg)	3-CQA (mg)	5-CQA (mg)	3,5-di-CQA (mg)	Total CQA (mg)
Filter A	Arabica	Mixed Particles	37.33	68.82	11.34	20.06	1.74	*33.14*	43.63	79.60	13.18	23.74	2.00	*38.92*
		500–1000	39.87	73.20	12.24	21.21	1.75	*35.2*	22.75	37.58	6.29	10.74	0.75	*17.78*
		400–500	47.01	88.23	14.95	25.72	2.16	*42.83*	28.62	49.64	8.39	14.54	1.11	*24.04*
		300–400	42.74	82.77	14.01	24.37	1.89	*40.27*	27.07	47.76	8.19	14.14	1.11	*23.44*
		200–300	44.39	85.66	14.59	26.29	2.08	*42.96*	31.46	64.03	10.33	18.85	1.80	*30.98*
	Robusta	Mixed Particles	43.45	174.03	20.59	24.40	5.93	*50.92*	25.21	105.43	14.37	23.78	4.17	*42.32*
		500–1000	40.38	158.03	19.64	31.66	4.81	*56.11*	15.30	78.26	10.07	16.19	2.60	*28.86*
		400–500	17.86	82.56	11.47	18.17	2.48	*32.12*	18.22	92.88	8.73	13.88	2.21	*24.82*
		300–400	18.66	90.04	9.48	14.99	2.31	*26.78*	13.51	60.78	7.37	11.67	1.67	*20.71*
		200–300	47.33	165.63	24.68	40.54	5.66	*70.88*	42.36	146.49	21.56	36.38	5.55	*63.49*
Filter B	Arabica	Mixed Particles	40.34	74.51	12.32	21.86	1.64	*35.82*	40.71	73.40	12.12	21.76	1.84	*35.72*
		500–1000	35.85	61.89	10.98	19.59	1.43	*32.00*	19.80	33.02	5.30	8.99	0.66	*14.95*
		400–500	39.11	72.57	12.49	21.85	1.55	*35.89*	29.15	51.64	8.59	15.16	1.25	*25.00*
		300–400	41.86	85.77	13.99	23.60	1.92	*39.51*	36.80	64.45	11.26	19.33	1.65	*32.24*
		200–300	45.62	95.12	15.69	27.12	2.23	*45.04*	39.00	73.02	12.74	22.91	2.08	*37.73*
	Robusta	Mixed Particles	32.67	143.28	16.48	27.45	4.01	*47.94*	27.81	121.79	14.02	23.08	3.41	*40.51*
		500–1000	23.59	118.49	13.35	21.32	3.12	*37.79*	16.37	87.37	10.22	16.05	2.39	*28.66*
		400–500	19.39	103.64	10.54	16.86	2.66	*30.06*	17.24	75.03	9.82	15.48	2.31	*27.61*
		300–400	21.33	93.60	10.52	16.94	2.56	*30.02*	11.64	55.04	6.48	9.99	1.61	*18.08*
		200–300	46.42	168.56	24.52	40.94	6.08	*71.54*	24.92	108.85	12.84	20.43	3.16	*36.43*
Filter C	Arabica	Mixed Particles	26.50	49.63	8.31	14.54	1.06	*23.91*	36.23	61.72	10.88	19.58	1.58	*32.04*
		500–1000	34.88	64.40	10.63	18.48	1.30	*30.41*	21.48	32.09	5.73	9.59	0.65	*15.97*
		400–500	39.82	76.51	12.59	21.82	1.57	*35.98*	39.04	65.03	11.43	19.77	1.55	*32.75*
		300–400	37.46	69.27	12.07	20.52	1.43	*34.02*	37.40	64.18	11.25	19.63	1.53	*32.41*
		200–300	40.89	76.52	13.72	23.99	1.79	*39.5*	41.69	74.68	13.33	23.60	1.95	*38.88*
	Robusta	Mixed Particles	28.47	126.83	14.70	23.89	3.25	*41.84*	21.49	99.59	11.61	19.28	2.96	*33.85*
		500–1000	27.25	136.46	14.92	24.73	3.56	*43.21*	18.33	89.24	10.37	17.06	2.24	*29.67*
		400–500	26.19	100.93	13.25	22.32	3.18	*38.75*	16.13	79.32	8.48	13.67	1.72	*23.87*
		300–400	22.33	105.42	11.09	18.17	2.87	*32.13*	19.33	83.66	9.68	15.78	2.16	*27.62*
		200–300	24.16	111.25	11.84	20.01	3.00	*34.85*	17.98	81.01	9.17	15.44	2.26	*26.87*

was 73.20 mg/cup at 500–1000 μm and 85.66 mg at 200–300 μm, but 88.23 mg at 400–500 μm. In the case of filter basket ***B***, the content of bioactive compounds was moderately increased when the amount of ground coffee was 12 g. This tendency was not fully evident when the mass of ground coffee was 14 g at 300–400 μm for both cultivars. The content of caffeine in Robusta was 118.49 mg at 500–1000 μm and

Table 4
Milligrams of caffeine, trigonelline and chlorogenic acids (3-CQA, 5-CQA and 3,5-CQAs) obtained in Arabica EC samples (n = 3, RSD% < 5.86) by using 12 and 14 g of ground coffee with various heights of perforated disc used for filter basket ***A*** at constant time (25sec) and volume (50 ml for double EC).

		Perforated Disc (mm)	Trigonelline (mg)	Caffeine (mg)	3-CQA (mg)	5-CQA (mg)	3,5-di-CQA (mg)	Total CQA (mg)
Time constant (25 *sec*)	Ground coffee (14 g)	4	91.08	150.69	24.23	41.27	2.91	68.41
		5	86.27	141.64	22.81	41.22	3.22	67.25
		6	84.14	145.40	22.34	39.30	2.74	64.38
		7	83.96	151.47	21.88	39.30	3.33	64.51
	Ground coffee (12 g)	4	78.89	134.53	21.81	38.49	3.28	63.58
		5	69.77	120.10	18.56	33.29	2.65	54.5
		6	77.02	136.84	21.41	37.50	3.14	62.05
		7	68.06	124.94	17.95	32.38	3.01	53.34
Volume constant (50 ml)	Ground coffee (14 g)	4	79.08	127.47	20.87	36.13	2.46	59.46
		5	87.84	151.25	23.32	41.72	3.51	68.55
		6	79.05	138.34	21.10	37.22	2.60	60.92
		7	82.89	154.72	22.20	39.73	3.48	65.41
	Ground coffee (12 g)	4	76.49	135.61	20.90	37.09	2.75	60.74
		5	74.64	126.87	19.70	34.94	2.73	57.37
		6	75.84	132.06	20.71	36.39	2.92	60.02
		7	70.66	125.59	19.05	34.22	3.08	56.35

168.56 mg at 200–300 μm, but 93.60 mg at 300–400 μm. In the case of filter basket ***C***, the increase of bioactive compounds was not significant from 500 to 1000 μm and 200–300 μm, while the most remarkable results appeared by lowering the amount of ground coffee from 14 g to 12 g. Almost all bioactive compounds at 12 g and 500–1000 μm were in fact nearly 50% lower than at 14 g, while those obtained at 200–300 μm were lower only 20%.

In Table 4, when maintaining the time and volume as constant (25 s and 50 ml for double cup, respectively) by changing the heights of PDs (4 mm, 5 mm, 6 mm and 7 mm) notable results were found. The mean content of caffeine at time constant was above 141 mg by using 14 g at various heights of PDs, whereas the mean content of caffeine in 12 g was just above 120 mg. This inclination was not similar when the volume was constant. When the height of PD was 4 mm and at 14 g of ground coffee, the content of caffeine was 127.47 mg, the same condition for 12 g showed 135.61 mg of caffeine. All bioactive compounds at 12 g in time constant with various heights of PDs were nearly 15% lower than at 14 g, while keeping constant volume with various heights of PD at 12 g were almost 9% lower than at 14 g.

3.5. Volatile compounds determination in EC

Volatile compounds in EC were analysed on DB-WAX column, which suits for coffee analysis. The brewed samples were inserted into GC–MS using HS-SPME and the analyses of volatiles acquired in full scan mode. The scanned ions of each sample were calculated through their percentage of relative peak area (RPA). Table 1S (Supplementary material) provides information about description of volatiles.

All volatiles above sixty percent were collected and divided into chemical classes (Thammarat, Kulsing, Wongravee, Leepipatpiboon, & Nhujak, 2018). The peak identification was performed through the NIST17.L Mass Spectral Search Program Library (Version 2.3/ build May 4, 2017, USA). A further division in chemical classes was then pursed according to their base of chemical groups.

Between 3 and 40 key odorants are typically composing a specific odour code of a food, which could feature in fact more than 10,000 volatiles (Dunkel et al., 2014). Descriptive aroma notes of each single volatile compound are given in Table 1S. In GC–MS results, the combination of the chemical groups was highlighted through their main representors that picked at retention time and estimated their mean relative peak area percentage. Signals at retention time 22.315 and 33.144 min are identified as member of Pyridine group, the mean RPA of single volatiles were found in this group corresponds to 7.55% of pyridine and 0.46% of 3-ethylpyridine. A key odorant of this group presents bitter, astringent, roasted and burnt aroma notes. In fact, coffee roasting defects can be associated to the presence of compounds of the pyridine family (Yang et al., 2016).

The members of pyrazine group are found in extracted EC at the following mean RPA: 5.62% methylpyrazine, 4.69% 2,5-dimethylpyrazine, 3.61% ethyl pyrazine, 4.41% 2-ethyl-6-methylpyrazine, 3.95% 2-ethyl-5-methylpyrazine, 3.71% 2-ethyl-3-methylpyrazine, 0.41% 2-(*n*-propyl)-pyrazine, 4.13% 3-Ethyl-2,5-dimethylpyrazine, 1.49% 2-Methyl-5-propylpyrazine and 0.46% 3,5-Diethyl-2-methylpyrazine, the retention time of these compounds were within 26.816–37.555 min. They generate nutty, roasted, grassy, corn and hazelnut-like aroma notes. It is evident therefore that the same volatile compounds are much faster to be released and easier to be detected in dry conditions (roasted ground coffee), than when they are processed and to be found in the liquid (EC).

The components of furan group in EC were obtained at different mean RPA %: 0.78% 2-(methoxymethyl)-furan, 0.98% dihydro-2-methyl-3 (2H)-furanone, 0.99% 2-furanmethanol, 0.96% 5-ethylmethyl-tetrahydro-2-furanmethanol, 3.55% 1-(2-furanyl)-ethanone, 1.44% 2-*N*-Butylfuran, 4.56% furfuryl acetate, 3.84% 5-methylfurfural, 1.13% 2-furanmethanol propanoate, 2.77% 2-propionylfuran, 2.05% furan, 2,2′-methylenebis, these compounds detected at the range of 25.165 and 45.252 min. The general key odorants of furan groups are caramel, ethereal, rum, cocoa note, and nutty. The fluctuation in detecting the furan groups shows that EC aroma is quite dense and complex. Notably, the furan groups quantities are higher in Arabica than in Robusta (Cordeiro, Valente, Santos, & Rodrigues, 2018). A derivative group of furans is furfural, found with 5.66% in EC aromas, and its detected retention time is 37.261 min. The generated aroma notes are almond, sweet, toasted odour and burnt.

Chemicals of phenol group were 1.60% 4-ethyl-2-methoxyphenol and 1.91% 2-methoxyphenol detected at retention time 45.587 and 47.139 min. Key-odorants that come with this group are mostly spicy and smoky.

Members of ketone and aldehyde groups were in moderate mean RPA in EC: 4.36% 1-hydroxy-2-propanone, 0.92% 3-hydroxybutanone and 1.89% benzaldehyde. Normally, the aroma of these groups is close to buttery, almond and caramelly (Aroma Chemicals, 2015). Terpene alcohol groups, as linalool with 0.48%, mostly present instead floral notes.

Components of pyrrole groups, as 0.68% 2-formyl-1-pyrrole, 1.04% 2-acetylpyrrole, and 2.05% 1-furfurylpyrrole in EC aroma, release nutty, musty, hay-like, mushroom-like, and herbaceous notes (Yang et al., 2016).

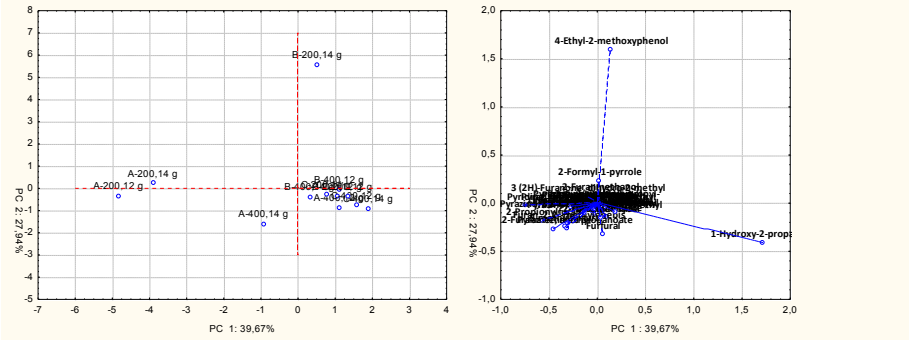
Chemical groups found in different EC aroma areas are compared by extraction conditions. As the amount of ground coffee and the size of grinded particles has varied, resulting GC–MS chromatogram highlighted that the smaller particles develop stronger aroma, because of the relative increase of the surface area in the coffee cake.

3.6. Principal component analysis (PCA)

The principal component analysis was applied to evaluate the relationship among the different particle sizes (200 μm and 400 μm) and three different filter baskets (***A, B*** and ***C***), by reducing the amount of ground coffee in the filter baskets, from 14 g to 12 g (Fig. 1a). Statistical data analysis indicates that filter baskets play a key role in brewing coffee. Three filter baskets named as ***A, B*** and ***C*** proved that lowering the amount of ground coffee at the same particle size had little impact on the release of volatile compounds. With filter basket ***A***, left side of Fig. 1a, 14 g of coffee are capable of generating nearly the same percentage of volatile compounds produced by 12 g at the same size of particles. Indeed, the two filter baskets ***B*** and ***C*** show no significant percentage variance of volatile compounds with different particle sizes at 12 and 14 g of ground coffee. Fig. 1b illustrates the percentage variance of volatiles in PCA. The variables most contributing to data variability were 1-Hydroxy-2-propanone (caramelly) on the first principal component and 4-ethyl-2-methoxyphenol (spicy) on the second principal component. They explained 67.61% of variance. Most of samples bearing baskets B and C were positively correlated with toasty caramel aroma, whereas only one sample with 200 μm mesh size and equipped with basket B was characterized by spicy aroma. Finally, the samples on left-hand side of Fig. 1b were those from two samples with 200 μm mesh size and equipped with basket A; they were correlated with several compounds such as pyrazine, furan, pyridine, carboxylic acid, aldehyde, alcohol and pyrrole groups.

4. Conclusion

The comparison between different extractions of EC with three different filters while reducing the amount of ground coffee from 14 g to 12 g provides information and results about the influence of different particle sizes of grinded coffee, and various heights of perforated discs in the extraction process. Three filter baskets were used to extract EC



a b

Fig. 1. a, b. Principal component analysis (PCA) of different filter baskets with 12 and 14 g of ground coffee used for extraction of EC.

with 14 g and 12 g of ground coffee, and it was found that, at the same particle sizes, volatiles had a similar percentages area although reducing the amount of ground coffee. Moreover, the changes in the structure of the perforated disc can help generate almost equal concentration of bioactive compounds when the mass of ground coffee in the basket is only 12 g. The implementation of different filters for smaller particle sizes, and of different heights of perforates discs for reduced amount of ground coffee, are both easy adjustments to apply in coffee houses (bars, coffee shops etc.). Simple and feasible as it is, this optimization of the coffee brewing process could lead in fact, in the long run, to a more sustainable consumption of EC, by reducing in the end the amount of the raw material and produce less amount of waste while maintaining the same quality of beverage. The environmental sustainability is mainly linked to a significant reduction in the waste production (e.g. packaging, coffee waste, plastic, energy consumption). Less discards not only result in a more sustainable waste reduction but also in a decrease in the disposal costs. Some of the main advantages presented in the study contemplate sustainability also at the socio-economic level, since the availability and use of such filters do not require *ad hoc* training and could be easily and widely adopted by a large number of selling categories at affordable costs, especially while considering the expense reductions in the coffee supply, where needed.

CRediT authorship contribution statement

Gulzhan Khamitova: Conceptualization, Methodology, Writing - original draft. **Simone Angeloni:** Methodology, Validation. **Germana Borsetta:** Conceptualization. **Jianbo Xiao:** Writing - review & editing. **Filippo Maggi:** Formal analysis, Software. **Gianni Sagratini:** Resources. **Sauro Vittori:** Supervision. **Giovanni Caprioli:** Data curation, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

G K and S A are grateful to Simonelli Group SpA (Belforte del Chienti, Macerata, Italy) for partially funding their fellowships and for providing coffee samples, espresso coffee machines, the grinder “Mythos 1” and the separation sieve “Retch AS200”.

Conflict of interests

The authors declare that no competing interests exist.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2020.126220.

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