Phenolic and volatile compound composition influence to specialty coffee cup quality

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Abstract. With increasing specialty coffee consumption, more attention is focused not only on the cup quality (sensory quality) of the coffee beverage but also about the impact of coffee on health. The beneficial effects of coffee on human health are mainly based on a wide range of biologically active components, including phenolic compounds. The aim of the study was to evaluate the influence of phenolic and volatile compound composition to specialty coffee cup quality. Seven specialty coffees from two Latvian roasteries were selected and analysed. Total phenolic and flavanoid content and radical scavenging activity by DPPH and ABTS assay were determined spectrophotometrically. Sensory evaluation (cup quality) was performed by trained panellist team using the SCAA protocols cupping specialty coffee. Volatile compounds were extracted by SPME and analysed by Gas Chromatography-Mass Spectrometry (GC–MS). Coffee final cup quality score ranged in amplitude of 83–90.25 points. HON_2 with dry fruits and melon characteristics has shown the highest final cup quality score. Almost detected volatile compounds in KEN_1 is associated with positive specialty coffee characteristics. In ETH_1 coffee with the final cup quality score 88.25 was detected highest floral, fruity compounds and highest coffee–like roasted notes. The highest total phenolic content and DPPH, ABTS$^{\bullet+\cdot}$ value showed Roastery_1 coffee samples (HON_1; KEN_1; COL_1) and the lowest values Roastery_2 coffee samples (HON_2; ETH_1; HON_3; SAL_1).The results indicate that the roastery specific roasting process parameters could influence not only volatile compounds profile and cup quality but also the total and individual phenolic compound content.

Key words: phenolic compounds, coffee aroma, cup quality, volatile compounds.

INTRODUCTION

Product quality is the key factor for consumer choice of product purchasing today. The quality of coffee is based on its flavour and aroma characteristics, and specialty coffee is a coffee which has been standardized from the coffee harvesting until its delivery to the consumer, in compliance with all quality standards, to highlight the best flavour of the coffee beverage (Hendon et al., 2014; Kwak et al., 2017).With increasing coffee consumption, more attention is focused not only for the flavour and texture of the coffee brew but also about the impact of coffee on health. The beneficial effects of coffee on human health are mainly based on a wide range of biologically active compounds. From the medical point of view, the positive effects of coffee on human health are even more prominent (Carman et al., 2014). Moderate and regular coffee consumption has been more positively associated with the wide range of bioactive compounds in it.

Research studies have shown that both caffeinated and non–caffeinated coffee consumption is proven to gain health benefits. This statement set, that the phenolic compounds in coffee have equally beneficial characteristics as caffeine (Ludwig et al., 2012; Martínez–Lopez et al., 2018).

Therefore, it is important to evaluate the best way to balance specialty coffee as the high–quality coffee drink not only with great aroma and flavour but also with positive biologically active compound value for human health. The desired aroma profile for specialty coffees has sweet, fruity and floral notes, which are formed in light–medium roast level. For the phenolic compounds, their concentration in coffee is rapidly decreasing during roasting. With the phenolic compounds, like chlorogenic acid, degradation, there are formed compounds with strong bitterness, which is not tolerated in good cup quality (Fischer et al., 2001; Fuller & Rao, 2017). During the roasting process chlorogenic acid converts to chlorogenic acid lactones and at this point having pleasant bitterness. But further roasting process from light to medium transforms the chlorogenic acid lactones into phenylindanes. Phenylindanes shows harsh bitterness which is not related to coffee–like bitterness (Frank et al., 2007).

To avoid in the roasting process formed phenolic compounds with the harsh bitter taste, it is important to know phenolic and volatile compound profile composition in coffee and their influence to cup quality. The aim of the study was to evaluate the influence of phenolic and volatile compound composition to specialty coffee cup quality.

MATERIALS AND METHODS

Samples
Seven samples of coffee *(Coffea arabica L.)* beans were collected from two different coffee roasteries in Latvia roasted at light–medium roast level (roasted at a maximum temperature of 214 °C; 11 min.). All coffee bean sample packages were sealed and stored in dry, cool place till coffee beverage preparation and analysed in one-month interval from coffee bean roasting. The main characteristics of coffee samples are summarized in Table 1.

Table 1. Main characteristics of the samples

Coffee sample	Roastery(roaster)	Origin	Roasting level
HON_1	Roastery 1 (Besca BSC-01, Turkey)	Honduras	Light–Medium (max. temperature of 193 °C; 11 min.)
KEN_1	Roastery 1 (Besca BSC-01, Turkey)	Kenya	Light–Medium (max. temperature of 193 °C; 11 min.)
COL_1	Roastery 1 (Besca BSC-01, Turkey)	Columbia	Light–Medium (max. temperature of 193 °C; 11 min.)
HON_2	Roastery 2 (Loring Smart Roast Kestrel35, USA)	Honduras	Light–Medium (max. temperature of 214 °C; 11 min.)
ETH_1	Roastery 2 (Loring Smart Roast Kestrel35, USA)	Ethiopia	Light–Medium (max. temperature of 214 °C; 11 min.)
HON_3	Roastery 2 (Loring Smart Roast Kestrel35, USA)	Honduras	Light–Medium (max. temperature of 214 °C; 11 min.)
SAL_1	Roastery 2 (Loring Smart Roast Kestrel35, USA)	El Salvador	Light–Medium (max. temperature of 214 °C; 11 min.)

Brewing method

After coffee bean sample package opening the coffee beans were (16 g per sample) and immediately were grind (coarse particle size 1.00–2.0 mm) (DeLonghi KG79 Coffee grinder/ Italy). The grind coffee was prepared by French Press brewing technique (SCAA Best Practice, 2016): 16 g of coarse grind coffee to 150 mL of 93°C water (Neptunas/ Lithuania) and the extraction time was 4 min. All brewed coffee samples were prepared by triplicates and immediately was analysed.

Determination of total phenolic content

Total phenolic content (TPC) was determined by spectrophotometric method using Folin–Ciocalteu reagent by Singleton et al. (1999) using a gallic acid as standard. A 2.5 mL of Folin–Ciocalteu reagent (Sigma–Aldrich Chemie, Steinheim, Germany) (diluted in proportion 1:10 with distilled water) was added to 0.5 mL diluted coffee extract (diluted in proportion 1:33 with distilled water). After 5 minutes, 2.0 mL of 7.5% Na_2CO_3 solution was added. After 30 minutes incubation at room temperature the absorbance of samples was measured at 765 nm using a Spectophotometer (Jenway 6300). The total phenolic content was expressed as gallic acid equivalent (GAE)100 g^{-1} using standard curve of gallic acid (y = 0.1069x–0.0107; R^2 = 0.9991).

Determination of total flavanoid content

Total flavonoid content (TF) was determined by spectrophotometric method reported by Zhishen et al. (1999)with some modifications. To 2.0 mL of distilled water and 0.5 mL diluted coffee extract (diluted in proportion 1:33 with distilled water) was added 0.15 mL 5% $NaNO_2$ solution. After 5 minutes, 0.15 mL of 10% $AlCl_3*6H_2O$ solution was added. After 5 minutes, 1.0 mL of 1M NaOH solution was added. Each coffee sample flask was mixed and after 15 min incubation at room temperature the absorbance of samples was measured at 415 nm with a Spectophotometer (Jenway 6300). Total flavonoid content was expressed as catechin equivalent (CE) 100 g^{-1} using standard curve of catechin (y = 2.7592x+0.0244; R^2 = 0.9982).

Determination of ABTS radical scavenging activity

ABTS radical scavenging activity was determined by Re et al. (1999) method with some modifications. To prepare ABTS radical, 2,2–azinobis(3–ethylbenzothiazoline–6–sulfonic acid (Sigma–Aldrich Chemie, Steinheim, Germany) was dissolved in phosphate buffer (PBS) solution and oxidized with potassium persulfate. The solution was kept in the dark at room temperature for 16h before further use. The ABTS˙+solution was diluted with PBS solution to an absorbance of0.70ABS (± 0.02) at 734 nm. To 0.05 mL of diluted coffee extract (diluted in proportion 1:33 with distilled water) 5 mL of diluted ABTS˙+solution was added. After 30 minutes incubation in dark, the absorbance of samples was measured at 734 nm using a Spectophotometer (Jenway 6300). Trolox (6–hydroxy–2,5,7,8–tetramethylchromane–2–carboxylic acid) (Sigma–Aldrich Chemie, Steinheim, Germany) was used as standard and the ABTS radical scavenging activity was expressed as μmol Trolox equivalent 100 g^{-1}using standard curve of Trolox (y = –0.9755x + 0.7604; R^2 = 0.9948).

Determination of DPPH radical scavenging activity

DPPH radical scavenging activity was assessed by Brand–Williams et al. (1995) with some modifications. The 2,2–diphenyl–1–picrylhydrazyl (DPPH) was dissolved in ethanol and the DPPH solution was diluted to an absorbance of 1.00 ABS (± 0.02) at 517 nm. To 0.5 mL of diluted coffee extract (diluted in proportion 1:33 with distilled water) 3.5 mL of diluted DPPH$^{\bullet+}$solution was added. After 30 minutes incubation in dark, the absorbance of samples was measured at 517 nm using a Spectophotometer (Jenway 6300). Trolox (6–hydroxy–2,5,7,8–tetramethylchromane–2–carboxylic acid) (Sigma–Aldrich Chemie, Steinheim, Germany) was used as standard and the DPPH radical scavenging activity was expressed as μmol Trolox equivalent 100 g^{-1} using standard curve of Trolox (y = –11,579x + 0.8931; R^2 = 0.9985).

Volatile compounds profile analysis

For volatile compounds extraction by solid phase micro–extraction (SPME) thedivinylbenzene/carboxen/poly (dimethylsiloxane) (DVB/CAR/PDMS) fibre was used. Akiyama et al. (2007) and Mestdagh et al. (2014) have also reported the DVB/CAR/PDMS fibre as suitable and efficient for coffee brew volatile compound detection. Coffee extract (5 mL) was placed in 20 mL glass container. The SPME parameters were chosen according to Gloess et al. (2013) with some modifications: extraction temperature: +50 ± 2 °C; incubation time: 4 minutes; extraction time: 7 minutes. Injection parameters: desorption time: 15 minutes; temperature: +250 °C.

For gas chromatography method 'Perkin Elmer Clarus 500' chromatograph with mass spectrometer and 'Elite–Waw ETR'(60 mx 0.25 mminternal diameter; DF 0.25 column were used. The column initial flow rate of 1 mL min^{-1} was held using helium as carrier gas. The outlet split 1:2 and between 40 and 300 mass–to–charge ratios were scanned. Oven temperature start – 40 °C, hold 7 min, programmed from 40 to 160 °C at 6 °C min^{-1}, and from 160 to 210 °C at 10 °C min^{-1}, hold 5 min; carrier gas (He) – 1 mL min^{-1}; split ratio – 2:1; ionization – EI+ mode; acquisition parameters in full scan mode – scanned m/z 40–400.

The compounds were tentatively identified using mass spectral database 'Nist98' (Gloess et al., 2013; Steen et al., 2017).

Sensory analysis

Sensory analysis was performed by six trained panellists according to the SCAA protocols cupping specialty coffee (SCAA, 2015). Each coffee sample was made with five replicates. The coffee evaluation process is stated in three steps: 1) evaluating the aroma of dry ground samples (15 minutes after the coffee sample was ground); 2) evaluating aroma of coffee brew after 3 minutes from extraction (coffee brew temperature ±93 °C); 3) evaluation of coffee brew flavour after 8–10 minutes from extraction (flavour, aftertaste at ± 71 °C; acidity, body and balance at 71–60 °C). Coffees were measured by ten specialty cup quality attributes: cup cleanness, acidity, body, flavour, aroma, after taste, uniformity, sweetness, balance and overall cup preference. The panellists also gave a description of specific flavour, aroma perceived according to The World Coffee Research Sensory Lexicon (2017) statements. Each attribute was

evaluated in scale from 1 to 10, with the final cup quality score of 100 points. The specialty coffee grade only applies if the total specialty cup quality score is 80 points or above (Figueiredo et al., 2013; Tolessa et al., 2016; Bressanello et al., 2017).

Statistical Analysis

One–way ANOVA analysis was used to statistically evaluate the differences between total phenolic, flavanoid content, antiradical activity and final cup quality score. The sensory analysis measures were carried out in five replicates and all chemical analysis measures were carried out in triplicate. The data were express as means. The significant differences were stated if $p \leq 0.05$. The data was analysed with Microsoft Office Excel 2013. A linear correlation analysis was performed in order to determine relationship between TPC, TF, antioxidant activity such as DPPH˙ and ABTS˙+, volatile compounds and final cup quality score.

RESULTS AND DISCUSSION

Sensory analysis

The final cup quality score and panellists sensory flavour and aroma description is shown in Table 2. All coffees scored in range 83–90.25. The lowest score (83 points) had SAL_1 coffee sample, it can be associated with the high acidity, which can disbalance the overall cup preference. From other point of view HON_2 with highest score (90.25 points) shown balance between the acidity and sweetness of the coffee brew.

Table 2. Final cup quality scores with sensory description

Roastery	Coffee sample	Final cup quality score	Sensory description
Roastery_1	HON_1	86.50	dark plum, grapes, red pepper, toffee (caramel)
	KEN_1	88.75	blackberry, red pepper, roses, dark chocolate
	COL_1	89.00	pineapple, dried apricot, elderflower
Roastery_2	HON_2	90.25	dried fruits, passion fruit, melon, kombucha
	ETH_1	88.25	lime, jasmine, chocolate cream, cherry brandy
	HON_3	85.00	cacao, red apples, dried fruits
	SAL_1	83.00	nutty and creamy notes

The balance between acidity and sweetness is correlated with higher final cup quality score as was showed in study Alex et al. (2016) that analysed relationship between different coffee plantation regions in Brazil and cup quality attributes, and in results the fruit, caramel flavour notes and pleasant acidity positively correlated with coffees whose quality standards was the most in accordance with the SCAA standards (SCAA, 2105). Final cup quality score has also shown strong correlation with sucrose and pleasant acidity in Borém et al. (2016) research. Pleasant acidity mostly is associated with dry fruit taste (Poltronieri & Rossi, 2016)and this could be the reason why HON_2 with dry fruits and melon characteristics shown the highest final cup quality score.

The major volatile compounds in coffee brew samples were furans, pyrazines, aldehydes and ketones (Table 3).Furans set the highest concentration, but the pyrazines, ketones and aldehydes contain some of the most important volatile compounds which are associated with a pleasant aroma and flavour notes in specialty coffee (Moon & Shibamoto, 2009; Parenti et al., 2014; Piccino et al., 2014; Steen et al., 2017; Yang et al., 2016). In Caporaso et al. (2018) study grouping volatile compounds by their chemical classes positive correlation was detected between aldehydes and ketones, but negative correlation between aldehydes and pyrazines. Ketones and aldehydes are also associated with floral, fruity aroma and pleasant acidity in coffee. More studies are approving the positive correlation between coffee cup quality and volatile compound concentration with floral, fruity aroma notes (Piccino et al., 2014; Poltronieri & Rossi, 2016). Ribeiro et al. (2009) study reports that higher concentration of 5–methyl–2–furancarboxaldehyde and furfural increased the overall quality of Brazilian *Coffea arabica L.* coffee samples.

If the fermentation process is not controlled at coffee bean harvesting moment, and also in roasting process, then the desired aldehyde and ketone compounds can easily transform in spirits, which can imbalance the coffee volatile compound composition (Preedy, 2015). Isoamyl acetate which was detected in HON_2 coffee have specific fermented aroma and flavour, with potential brandy, over ripe fruit notes (Toledo et al., 2016), and in sensory analyses panellists detected kambucha (non–alcoholic fermented fruit beverage) notes. In this situation fermented flavour notes are associated with positive cup quality characteristics and the high final cup quality score is in the line with trend in specialty market – exploring the fermented and specific aroma notes (Sepúlveda et al., 2016).

In previous studies about volatile compound composition and final cup score, coffee furanone (dihydro–2–methyl–3–furanone) concentration is positively associated with higher final cup quality scores (Toledo et al., 2016). Only dihydro–2–methyl–3–furanone was only detected in KEN_1 coffee samples. All detected volatile compounds in KEN_1 is associated with positive specialty coffee characteristics (Steen et al., 2017).In ETH_1 coffee with the final cup quality score 88.25 was detected highest floral, fruity compounds, like furfuryl acetate, 2–furanmethanol, and highest coffee–like roasted notes (2–methyl butyraldehyde,2–methyl–propanal).

None of the coffee samples were detected compounds with strong association with defected coffee quality. This approves that the specialty coffee high standards for green coffee beans limits the risk of damaged or unripe beans. By limiting coffee defects it also excludes possible defective/unpleasant volatile phenolic compound presents in coffee brew, for an example, 4-ethyl-2-methoxyphenol, 2-methylphenol (Giacalone et al., 2019; Steen et al., 2017).

Table 3. In the headspace of coffee brew samples identified volatile compounds

Compounds	GC–MS peak area (x10^6)							Compound sensory description
	HON_1	KEN_1	COL_1	HON_2	ETH_1	HON_3	SAL_1	
1-(2-Furanylmethyl)-1H-pyrrole				7.025a	7.847b	8.539c	7.597b	savory, vegetables**
2.3 Pentanedione	17.453b	20.989c	23.374d	12.765a	26.013e	30.189f	26.266e	buttery***
2.6–Dimethyl-4–thiopyrone				5.006				caramel**
5–Methyl–2–furancarboxaldehyde	48.053b	40.476a	76.429e	51.623c	63.474d	97.233e	76.848f	red pepper *
2–Furanmethanol	39.029c	37.860b	55.160e	32.837a	49.475d	72.414e	70.254f	floral***
Furfuryl acetate	38.760b	31.600a	93.105e		78.378c	98.544f	83.259d	floral, fruity****
Dihydro–2–methyl–3(2H)–furanone		17.567						coffee–like, caramel*****
2–Methyl–butanal	14.937a	21.138b	36.067c	93.308e	36.939c	93.311e	39.428d	cacao, coffee–like*****
1–(2–Furanyl), ethanone			22.425					nutty ***
2–(Methoxymethyl)furan					4.591			roasted almond, coffee***
2–Methyl–furan					24.119	18.563		caramel***
Furfural		10.800a	102.107e	64.532b	95.721c	133.029f	99.558d	roasted sweet almond**
2–Methyl–propanal				19.239	9.889	11.145		chocolate******
Ethyl–pyrazine				8.465	7.456			chocolate–peanut, nutty **
4–Methyl–pyrimidine	14.156		26.268	1.433	24.662	32.687	2.707	popcorn*
2–Methoxy–4–vinylphenol		1,923						spicy, floral*****

* – Toledo et al., 2016; ** – Bressanello et al., 2017; *** – Lee et al., 2017; **** – Steen et al., 2017; ***** – Piccino et al., 2014; Different letters indicate statistically significant ($p < 0.05$) differences between samples.

Total phenolic and flavonoid content

Significant differences were detected between coffee roasteries and total phenolic content in coffee brews. It can be associated with different technical conditions in the roasting process and with the specific characteristics of each raw materials have.
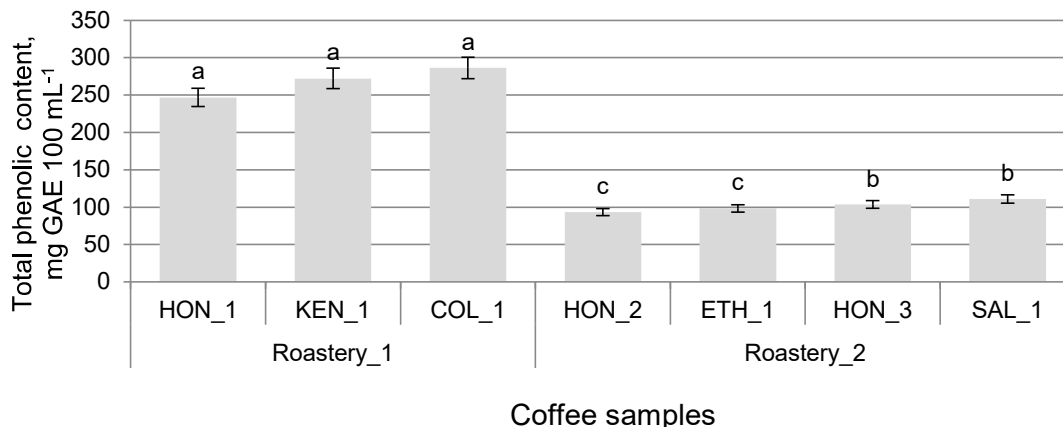

Figure 1. Total phenolic content in coffee brews.

Similar results showed also total flavonoid content in coffee brews (Fig. 2). The highest phenolic and flavonoid content were detected in COL_1 and the lowest in HON_2. From volatile compounds profile COL_1showed more balanced composition in comparison with HON_2. COL_1 volatile compounds profile has fruity and floral aroma, flavour notes from organic acids and also phenolic acids. But HON_2 dominated in higher nutty, chocolate flavour notes from furans.
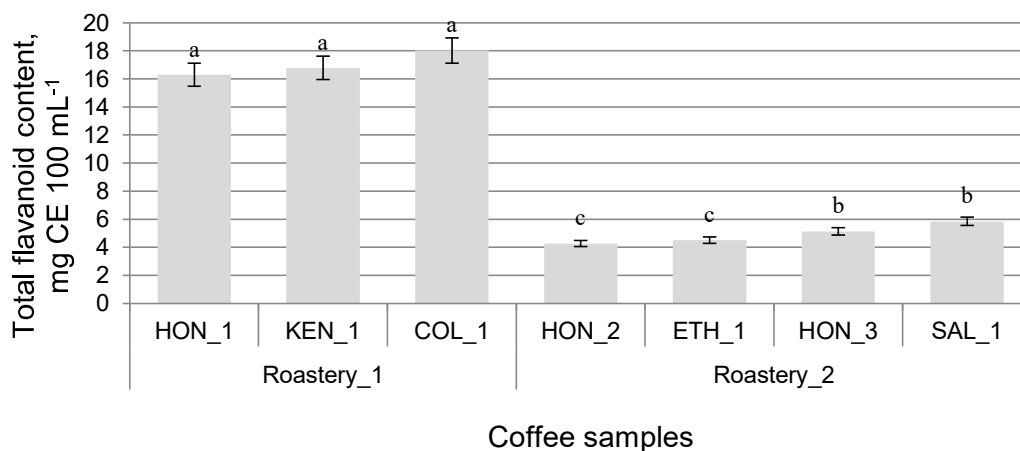

Figure 2. Total flavanoid content in coffee brews.

It has been proven that between green coffee samples the compositions of phenolic compounds are similar, but between roasted coffee samples phenolic compound composition can change significantly for various reasons, for example, roasting temperature, time, storage etc. (Somporn et al., 2011; Cheong et al., 2013), this can be

one of the main reasons why results for high quality coffees have significant differences between roasteries.

ABTS and DPPH radical scavenging activity

Similar to total phenolic and flavonoid content COL_1 showed the highest DPPH value (832.441 µmol trolox equivalent g^{-1}) and ABTS+˙value (34.127 µmol trolox equivalent g^{-1}) and HON_2 showed the lowest DPPH value (505.20 µmol trolox equivalent g^{-1}) and ABTS+˙value (34.127 µmol trolox equivalent g^{-1}).

There was high correlation between DPPH and ABTS+˙ assay with total phenolic content in coffee brews. DPPH had higher correlation ($r = 0.996$) than ABTS+˙($r = 0.9345$) but both assays showed similar results with other research studies (Somporn et al., 2011; Daniel & Workneh, 2017). Also, the radical scavenging activity showed significant differences between coffee roasteries.

Correlation between total phenolic content and cup quality

There was no correlation detected between final cup quality scores and total phenolic content in coffee brews overall. But by grouping coffee brews by roasteries, there was positive correlation ($r = 0.971$) between final cup quality scores and total phenolic content for Roastery_1 coffee brews (Fig. 4, B.) and negative correlation ($r = -0.957$) between final cup quality scores and total phenolic content for Roastery_2 coffee brews (Fig. 4, A).
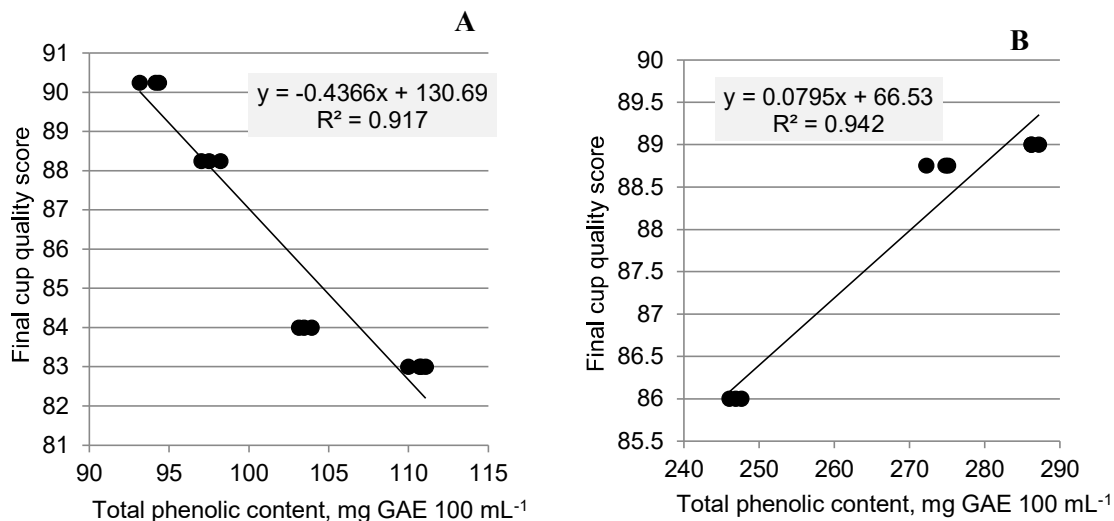

Figure 4. Correlation between final cup quality scores and total phenolic content in coffee brews. (A) Roastery_2 coffee brews; (B) Roastery_1 coffee brews.

The opposite correlations between two roasteries could suggest that roasting process parameter influence important chemical compound content in coffee brews differently. Roastery_1 coffees volatile compounds profile is more balanced and focus to fruity and floral compounds, like furfuryl acetate, 2–furanmethanol, while Roastery_2 coffee volatile compounds profiles have a higher number of volatile compounds (2–methyl–propanal – chocolate notes; 1–(2–Furanylmethyl)–1H–pyrrole – savory notes; 4–methyl–pyrimidine– popcorn, roasted bread notes). The sensory results for

Roastery_1 coffee samples showed minimal final cup quality score differences, while Roastery_2 had wider amplitude from 83 point to 90.25 points. These two results could suggest that it is possible to maintain high phenolic compound content in coffee brew if the volatile compounds composition is focused on specific aroma attributes like sweet and pleasant acidity of fruity, floral aroma, flavour notes.

Analysing other studies about phenolic content correlation with sensory results in coffee, it also shows opposite results about cup quality and phenolic compound composition. In some research studies phenolic compounds like 5–caffeoylquinic acid (5–CQA) and feruloylquinic acid (5–FQA) is associated with lower cup quality, because of the bitterness(Fujioka & Shibamoto, 2008). Fank et al. (2007) analysing the bitter–tasting compounds in roasted coffee, states that in sensory analysis 5–CQA is associated with coffee–like bitterness, caffeic acid with strong roasted coffee bitterness and only ferulic acid and trigonelline has association with harshly strong bitterness. Phenolic compound as2–methoxy–4–vinylphenol brings pleasant spicy, floral notes to coffee brew in low concentrations (Piccino et al., 2014). In low concentrations 2–methoxy–4–vinylphenol was detected in Roastery _1 Kenya_1 (II) coffee brew and its volatile compounds sensory descriptions match with the panellists compound sensory description. Moon & Shibamoto (2009) research states that phenolic compounds with pleasant and fresh aroma, flavour notes as 2–methoxyphenol, chlorogenic acids and 2–methoxy–4–vinylphenol rapidly decreases after light roasting level but caffeic acid, catechol increases with the roasting level bringing harsh bitterness to coffee brew. Zanin et al. (2016) in his research proved that it is possible to contain good cup quality without losing the valuable chlorogenic acid content. These studies suggest that individual phenolic compounds could affect differently the overall sensory characteristics of the coffee. This is one of the reasons why it is important to determine individual phenolic content and analyse its correlation with the sensory analysis results.

CONCLUSIONS

Final cup quality score in sensory analysis varied in the range of 83–90.25 points with the highest score had HON_2 and the lowest score had SAL_1. Roastery_1 coffees volatile compounds profile was more balanced with fruity and floral compounds, like furfuryl acetate, 2–furan methanol, while Roastery_2 coffee volatile compounds profile had chocolate, nutty, roasted aroma notes. The difference between the roastery coffee sample total phenol, flavonoid content and antiradical scavenging activity also showed significant differences. A positive correlation was found between final cup quality scores and total phenolic content for Roastery_1 coffee brews and a negative correlation between final cup quality scores and total phenolic content for Roastery_2 coffee brews. The different correlations could be associated with specific phenolic compound presents in the coffee brew. These results indicate that the roastery specific roasting process parameters could influence not only volatile compounds profile but also the total and individual phenolic compound content. To better predict roasting process influence to phenolic compound composition it is important to further analyse the specific roasting parameters and individual phenolic compounds with volatile compounds profile correlation.

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