

TITLE: Effect of sugar addition (torrefacto) during roasting process on antioxidant capacity and phenolics of coffee

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ABSTRACT

The addition of sugar during roasting (torrefacto) has been proposed as a technique to increase the antioxidant capacity. However, other factors such as roasting degree and coffee origin also play a key role. Two batches of Colombian green coffee were roasted adding increased amounts of sucrose (0-15 g per 100 g of coffee) to reach the same roasting degree than a commercial Colombian coffee. Moreover, seven conventional roasted coffees from different origins (Colombia, Brazil, Kenya, Guatemala and Vietnam) and roasting degrees (Dark, Medium and Light), and one 100% Torrefacto roasted coffee were analyzed. Although the addition of sugar during roasting increased the DPPH quenching activity, phenolic compounds (5-caffeoylquinic, caffeic and ferulic acids, and 4-vinylguaiacol) were hardly affected by torrefacto roasting process, showing that Maillard and other roasting reactions products, such as browned-colored compounds including melanoidins (Abs 420nm), have an important role as antioxidants. Principal Component Analysis (PCA) showed that roasting degree also plays a key role on overall antioxidant activity. Moreover, the Absorbance at 420nm has been proposed as a good marker of torrefacto roasting process, whereas the roasting degree might be better characterized by L* values.

KEYWORDS: Coffee, roasting, antioxidant, phenolic compounds, Maillard Reaction Products

1. INTRODUCTION

During last few years, roasted coffee has been proposed as one of the main source of antioxidants in the diet (Svilaas et al., 2004; Pulido, Hernandez-Garcia, & Saura-Calixto, 2003). The roasting of coffee is a complex process where the loss of antioxidant activity due to natural antioxidants – mainly represented by polyphenols – by progressive thermal degradation has been found to be minimized by the formation of Maillard reaction products (MRPs) (Nicoli, Anese, Manzocco, & Lerici, 1997).

Torrefacto is a roasting process in which sugar is added to coffee, normally Robusta. This roasting technique is used in several countries of Southern Europe and South America where some segments of the population prefer coffees with a dark brown, intense aroma and a strong taste with a tendency to bitterness. This kind of roasting process was initially used to mask negative sensorial attributes in Robusta coffees. Nowadays, Torrefacto roasted coffee is usually blended with conventional roasted coffee (Arabica or Robusta) to be commercialized. The addition of sugar at the end of the torrefacto roasting process might intensify the development of Maillard reactions and, consequently, increase the antioxidant capacity of coffee (Lopez-Galilea, Andueza, di Leonardo, de Peña, & Cid, 2006; Lopez-Galilea, de Peña, & Cid, 2008; Andueza, Cid, & Nicoli, 2004). However, the analyzed samples in these works were commercial coffees in which Arabica and Robusta coffees from different unknown origins, percentages and roasting degrees were blended.

Nicoli et al. (1997) reported that dark-medium roasted coffee had the highest antioxidant capacity showing that roasting degree is a key factor. But, the origin and the variety of coffee (Arabica and Robusta) with different amounts of phenolics in green coffee also can play an important role. Consequently, the different antioxidant capacity of commercial Torrefacto roasted coffee blends previously studied by our research

group (Lopez-Galilea et al., 2006; Lopez-Galilea et al., 2008) can not be attributed only to Torrefacto roasting process. Thus, the influence of the sugar addition during torrefacto roasting process on the antioxidant capacity of coffee should be deeper studied controlling the other parameters. So that, the aim of this work was to know whether the addition of increased amounts of sugar to coffee during roasting process (torrefacto) could be a key factor to increase the antioxidant capacity, and to know its influence on the most relevant coffee antioxidant compounds (phenolic compounds and melanoidins). And secondly, whether the addition of sugar during roasting had higher or lower influence than the roasting degree and the origin of coffee.

2. MATERIALS AND METHODS

2.1 Chemicals and reagents. The methanol (spectrophotometric grade), Folin-Ciocalteu reagent and sodium carbonate were obtained from Panreac (Barcelona, Spain). Gallic acid, 2,2-Diphenyl-1-picrylhydrazyl (DPPH^{*}), Trolox (6-hydroxy-2,5,7,8-tetramethyl-chroman-2-carboxylic acid) 5-caffeoylquinic acid (5-CQA), caffeic acid, ferulic acid, 4-vinylguaiacol, were obtained from Sigma-Aldrich (Steinheim, Germany). Acetonitrile, HPLC, grade was provided by Scharlau (Barcelona, Spain).

2.2 Coffee samples. Seven conventional roasted vacuum-packed coffee samples from different origins (5 Arabica coffees from Colombia, Brazil, Kenya and Guatemala and 2 Robusta coffees from Vietnam), and one commercial 100% Torrefacto roasted coffee (T100 Light) were selected. Roasted coffee samples were classified into 3 roasting degrees according to the L* color parameter results: Dark (L* < 23), Medium (L* 23-26) and Light (L* > 26) following similar criteria of other authors (Nicoli et al., 1997; Vignoli, Bassoli, & Benassi, 2011). Colombia Dark and T100 Light coffee samples of the same brand were purchased in a local market. Colombia Medium, Brazil Medium,

Kenya Medium, Guatemala Medium, Vietnam Medium and Vietnam Light roasted coffee samples and green coffee beans (variety *Coffea arabica*, from Colombia) were supplied by two roasting companies.

2.3 Coffee roasting process. Two batches (I and II) of Colombian green coffee beans were roasted adding increased amounts of sucrose (0, 5, 10 and 15 g per 100 g of coffee) to reach the same roasting degree (L^* 19-23, Dark) than the selected commercial Colombian coffee sample (Colombia Dark). The amount of added sugar must not exceed 15g/100g coffee beans as regulated by law in Spain (Real Decreto 1231/1988). Roasting process was developed following the time and temperature conditions presented in Figure 1. Sucrose was dissolved in the minimum volume of water and homogeneously spread out to the coffee beans at 21 min of roasting. During the roasting process, pan surface and air temperatures were controlled. Each batch of coffee was roasted in duplicate. At the end of the process, coffee samples were controlled by the L^* value (19-23, Dark) and weight loss (18-19 g per 100 g). Weight loss was calculated by the difference between green and roasted coffee weights and expressed as g per 100 g. After 4 hours of degassing, 60 g of roasted coffee were packed in plastic bags (type 160*300 PA/PE 90 μ m, Vaessen-Schoemaker Industrial S.A.U., Barcelona, Spain) and sealed under vacuum (Ramon Serie VP Mod.450, Barcelona, Spain). Samples were named with the amount of added sugar followed by the roasting degree and the batch number (0 Dark I, 5 Dark I, 10 Dark I, 15 Dark I, 0 Dark II, 5 Dark II, 10 Dark II and 15 Dark II). All coffee samples were stored in darkness and at 4 °C up to the coffee analysis (<1 month after roasting or purchasing).

2.4 Sample preparation. Coffee packages were opened immediately before the preparation of the coffee extracts in order to avoid oxidative damage. Sixty g of roasted coffee beans were ground in a Moulinex coffee grinder (model Super Junior “s”, Paris,

France) for 30 seconds. Coffee extracts were obtained by solid-liquid extraction, using deionized water at 100 °C. The ratio between coffee and water was 10/100 (g/mL). The extraction time was 10 min. The extracts were immediately cooled with cold running water and filtered through Whatman No. 1 filter paper.

2.5 Color analysis. Color analysis was carried out on ground roasted coffees by means of a tristimulus colorimeter (Chromameter-2 CR-200, Minolta, Osaka, Japan) using the D65 illuminant. The instrument was standardized against a white tile before sample measurements. Ground roasted coffee was spread out in an 1 cm Petri plate, and the color measured was expressed in L*, a* and b* CIELab scale parameters.

2.6 Browning compounds (Abs 420 nm). Fifty microliters of coffee extract were diluted up to 2 mL with deionized water. Browning compounds were quantified by measuring the absorbance of the sample at 420 nm after exactly 1 min, in a 3 mL capacity cuvette (1 cm length) with a spectrophotometer Lambda 25 UV-VIS (Perkin-Elmer Instruments, Madrid, Spain) connected to a thermostatically controlled chamber (25 °C) and equipped with UV Win- Lab software (Perkin Elmer).

2.7 Antioxidant capacity by DPPH assay. The antioxidant capacity was measured using the 2,2-diphenyl-1-picrylhydrazyl (DPPH[•]) decolorization assay (Brand-Williams, Cuvelier, & Berset, 1995) A 6.1×10^{-5} mol/L DPPH[•] methanolic solution was prepared immediately before use. The DPPH[•] solution was adjusted with methanol to an absorbance of 0.7 (± 0.02) at 515 nm in a 3 mL capacity cuvette (1 cm length) at 25 °C (Lambda 25 UV, VIS spectrophotometer, Perkin Elmer Instruments). Coffee extracts (50 μ L) were added to DPPH[•] solution (1.95 mL). After mixing, the absorbance was measured at 515 nm after exactly 1 min, and then every minute for 18 min at 25 °C. Calibration was performed with Trolox solution (a water-soluble vitamin E analogue) and Total antioxidant capacity was expressed as μ mol Trolox per g of ground coffee.

2.8 Folin-Ciocalteu (FC) assay. The Folin-Ciocalteu reducing capacity of coffee was performed according to the Singleton's method (Singleton, Orthofer, & Lamuela-Raventos, 1999). For every coffee sample extract, 1:10 dilutions with demineralized water were prepared and 500 μ L of Folin-Ciocalteu reagent were added to 100 μ L of the coffee sample solution. After 2 min 1.5 mL a 7.5 g/100g sodium carbonate solution was added. Next, the sample was incubated in darkness at room temperature for 90 min. The absorbance of the sample was measured at 765 nm in a spectrophotometer (Lambda 25 UV, VIS spectrophotometer, Perkin Elmer Instruments). Gallic acid (GA) was used as reference, and the results were expressed as g GA per 100 g of ground coffee.

2.9 5-Caffeoylquinic acid (5-CQA). A 500 μ L amount of the coffee brew was diluted up to 50 mL with milliQ water. HPLC analysis was carried out with an analytical HPLC unit model 1100 (Agilent Technologies, Palo Alto, CA), equipped with a binary pump and an automated sample injector. A Hypersil-ODS column (5 μ m particle size, 250 mm \times 4.6 mm) was used. The chromatographic separation was achieved at 25 $^{\circ}$ C by using a gradient solvent system with acetonitrile/water adjusted to pH 3.0 with a phosphoric acid solution according to the method described by Perez-Martinez, Sopelana, De Peña, & Cid (2008). The wavelength of detection was 325 nm.

2.10 Hydroxycinnamic acids (caffeic acid and ferulic acid) and 4-vinylguaiacol. The extraction, clean-up and HPLC analysis of these three compounds were performed simultaneously, according to the method developed in our department (Alvarez-Vidaurre, Perez-Martinez, De Peña, & Cid, 2005). The HPLC analysis was carried out with the same equipment and column described above. The chromatographic separation was achieved at 25 $^{\circ}$ C by using a gradient solvent system with acetonitrile/water adjusted to pH 2.5 with a phosphoric acid solution according to the procedure published

by Perez-Martinez et al. (2008). The wavelengths of detection were 314 nm for caffeic acid, 325 nm for ferulic acid and 210 nm for 4-vinylguaiacol.

2.11 Statistical analysis. Each parameter was analyzed in triplicate. Results are shown as means \pm standard deviations. Analysis of variance (ANOVA) was applied to the parameters. Tukey test was applied as test *a posteriori* with a level of significance of 95%. Correlations among variables were assessed by means of the Pearson Correlation test. Principal component analysis (PCA) was applied to the analytical data (based on the Pearson correlation matrix) to observe differences among coffees samples. Principal components (PC) with eigenvalues greater than 1 were selected. All statistical analyses were performed using the SPSS v.15.0 software package.

3. RESULTS AND DISCUSSION

3.1 Influence of torrefacto roasting process on coffee color

Brown color development is one of the most visual changes in heat-treated foods, such as coffee, cereal, cookies, etc. during processing. In the present work, the color of ground roasted coffees was measured by means of the CIELab parameters (L^* or lightness, and a^* and b^* as the chromaticity parameters) and the Absorbance at 420nm (Table 1). Although Torrefacto coffee samples were roasted to reach the same roasting degree that Colombia Dark, a slight tendency to increase L^* value with the addition of sugar can be observed in the lab roasted coffee samples, except in 15 Dark II. However, this increase was not significant and, in fact, no significant correlation ($p > 0.05$) between L^* value and the amount of added sugar has been found. These results are in agreement with those reported previously by Lopez-Galilea et al. (2006) who observed a similar L^* increase with the amount of torrefacto roasted coffee in commercial blends, but only

in two of the three analyzed brands. Consequently, L^* value is clearly related with roasting degree, but not with the torrefacto roasting process.

In Table 1, it can also be observed that the Light roasted coffees showed significantly higher a^* (+red) and b^* (+yellow) values than Medium and Dark roasted coffees. In fact, significant correlations (0.786 and 0.912, $p < 0.05$) between chromaticity parameters (a^* and b^* , respectively) and lightness (L^*) have been found. Other authors also obtained similar results and correlations in conventional roasted coffees and in conventional/torrefacto coffee blends (Lopez-Galilea et al., 2006; Summa, de la Calle, Brohee, Stadler, & Anklam, 2007). However, no significant correlations have been found between any of the chromaticity parameters (a^* or b^*) and the amount of added sugar during roasting. Thus, the CIELab parameters seem to be independent of the type of roasting process (conventional or torrefacto), maybe because torrefacto roasting process only induces the formation of an external caramel coating and hardly affects the interior of the coffee beans.

The absorbance at 420nm has been commonly used to characterize melanoidins, which are mainly originated by Maillard Reactions during roasting process of coffee and other heat-treated foods (Morales, 2005; Nunes & Coimbra, 2007). However, it has been reported that melanoidins accounted for only 65 % of color potency of the high molecular weight fraction obtained from light roasted coffee, and for only 39 % from dark roasted coffee (Nunes & Coimbra, 2007). Many other brown-colored products appear to be sugar (retro)aldolization/dehydration and carbohydrate condensation products, which may or may not be attached to proteins or other structures of amino nitrogen in a similar way to the Maillard Reactions (Rizzi, 1997; Hofmann, 1999). So that, hypothetically, the addition of sugar to coffee during Torrefacto roasting process might induce a higher formation of brown-colored Maillard Reactions and

caramelization products that are water soluble and can be measured by the Absorbance at 420nm. In Table 1, it can be observed that those coffees with sugar added during roasting (torrefacto) showed significantly ($p < 0.05$) higher Absorbance at 420nm than those roasted conventionally with the same roasting degree (Torrefacto lab roasted coffees versus 0 Dark and Colombia Dark, or T100 Light versus Vietnam Light). Moreover, commercial torrefacto coffee (T100 Light) showed similar results ($p > 0.05$) to those coffees roasted with 15g sugar per 100g coffee. In fact, highly significant ($p < 0.001$) and excellent correlation (0.876) between the Absorbance at 420nm and the amount of sugar added during roasting process has been found showing that this parameter might be proposed as a marker of torrefacto roasting process. The highest Absorbances at 420nm in Torrefacto coffees also explain that caramelization products in torrefacto roasted coffee are mainly water soluble.

Principal Component Analysis (PCA) has been applied to evaluate at a glance the influence of the roasting type (conventional or torrefacto) and roasting degree (Dark, Medium and Light) on the antioxidant activity and color of coffee samples. Figure 2 shows the bidimensional representation of all the variables and coffee samples according to the two selected Principal Components (PC). PC1 (65.2% of the total variance) was mainly characterized by the CIELab color parameters (L^* , a^* and b^*) and the Folin-Ciocalteu reducing capacity. It could be observed that PC1 distributed all the coffee samples according to the roasting degree, being the dark samples on the left half-part of the graphic, but independently of the origin, variety and type of roasting process (conventional or torrefacto). PC2 (19.2% of the total variance) was mainly and positively characterized by the Absorbance at 420nm. So that, those coffees roasted with sugar addition (torrefacto) were mapped in the top half-part of the graphic. Roasting degree also exerts influence on brown compounds formation because dark

roasted coffees showed significantly ($p < 0.05$) higher Absorbances at 420nm than medium and light conventional coffees (Table 1). However, this influence was much lower than that induced by torrefacto roast because (1) there were no significant differences between medium and light conventional coffees in agreement with other authors (del Castillo, Ames, & Gordon, 2002), and (2) PC2 can not discriminate among different roasting degree coffees. In conclusion, the Absorbance at 420nm might be proposed as a good marker of torrefacto roasting process, whereas the roasting degree might be better characterized by L^* values.

3.2 Influence of torrefacto roasting process on phenolic compounds and antioxidant capacity. The antioxidant capacity of coffee was evaluated by two colorimetric assays, the chain-breaking activity by DPPH· radical quenching assay and the Folin-Ciocalteu assay.

Figure 3 shows the antioxidant capacity, measured by the DPPH quenching assay, of the Torrefacto roasted coffees in comparison with conventional roasted coffees of different origins (Colombia, Brazil, Kenya, Guatemala, and Vietnam) and different roasting degrees (Dark, Medium and Light). Commercial conventional roasted coffees showed lower DPPH results than lab-roasted torrefacto roasted coffees. This might be explained by a longer storage (from roasting to purchase) under less controlled conditions (room temperature) in commercial samples, because during storage the antioxidant capacity decreases due to the presence of residual oxygen, and other radicals or pro-oxidant compounds formed during the roasting process (Manzocco, Calligaris, & Nicoli, 2002). According to the DPPH quenching activity, conventional roasted coffees of different origins and the same roasting degree (Medium) can be ranked in increasing order as Brazil < Vietnam < Colombia < Guatemala < Kenya. However, roasting degree seems to influence conventional roasted coffees in different way depending on the origin or

variety because DPPH increased with a higher roasting degree (Dark vs Medium) for Colombia coffee (Arabica), but decreased for Vietnam one (Robusta) (Medium vs Light). This could be due to the fact that although Robusta coffee has higher amounts of phenolic compounds than Arabica ones, roasting induces a higher loss of these antioxidant compounds in Robusta coffees (Clifford, 1997; Perrone, Donangelo, Donangelo, & Farah, 2010). In fact, only moderate correlation between DPPH and L* values (-0.483, $p < 0.001$) has been found.

Focusing into the influence of the sugar addition during roasting process, correlation results show a clear and significant (0.701, $p < 0.001$) tendency to increase the DPPH antioxidant capacity with the amount of sugar added during roasting process. Also T100L coffee exhibited higher DPPH results than the commercial conventional roasted coffees. A significant and good correlation between DPPH and Absorbance at 420nm, proposed as a good marker of torrefacto roasting process, has been found (0.721, $p < 0.001$). Moreover, DPPH quenching activity contributed partially to the PC2 in Principal Component Analysis (Figure 2) that mapped those coffees roasted with sugar addition (torrefacto) in the top half-part of the graphic. These findings are in agreement with those obtained in commercial torrefacto roasted coffee blends (Lopez-Galilea et al., 2006; Lopez-Galilea, de Peña & Cid, 2007). The higher DPPH quenching activity can be attributed mainly to the formation of Maillard Reactions and caramelization antioxidant products ((Manzocco, Calligaris, Mastrocola, Nicoli, & Lerici, 2001), but the influence of torrefacto roasting on phenolic compounds should be deeper studied in those coffees roasted with increasing amounts of sugar addition.

Figure 4 shows the FC results of the Torrefacto roasted coffees in comparison with conventional roasted coffees of different origins (Colombia, Brazil, Kenya, Guatemala, and Vietnam) and different roasting degrees (Dark, Medium and Light). The Folin

Ciocalteau method is traditionally used to measure phenolic compounds, but several authors have reported that this method also evaluates other reducing nonphenolic compounds, such as melanoidins, proteins and thiols, and thus should be seen as a measure of total antioxidant capacity rather than phenolic content (Perez-Martinez, Caemmerer, De Peña, Cid, & Kroh, 2010; Caemmerer & Kroh, 2006; Everette, Bryant, Green, Abbey, Wangila, & Walker, 2010). A decrease of the antioxidant capacity measured by Folin-Ciocalteau technique with the increase of roasting degree can be observed. A highly significant ($p < 0.001$) and good correlation (0.785) with L^* values has been found. Moreover, FC values also contributed to the PC1 in Principal Component Analysis (Figure 2) together with the CIELab parameters and, then, to the distribution of coffees according to the roasting degree. Similar patterns were reported by other authors (Bekedam, Loots, Schols, Van Boekel, & Smit, 2008; Sacchetti, Di Mattia, Pittia, & Mastrocola, 2009) in conventional roasted coffees, mainly due to a higher degradation of chlorogenic acids, the most abundant phenolic compounds in coffee. Loss of phenolic compounds during roasting is very well known and losses of 8-10% for every 1% loss of dry matter (Clifford, 1997; Clifford, 1999; Clifford, 2000) up to 95% of the chlorogenic acid content in green coffee with drastic roasting conditions (Trugo & Macrae, 1984) were reported.

Higher FC reducing capacities were found in those coffees roasted with sugar (torrefacto) in comparison with their respective conventional roasted ones (0 Dark I and 0 Dark II) (Figure 4), but only were statistically significant in the batch II. For that reason, 5-caffeoylquinic, caffeic and ferulic acids, and 4-vinylguaiacol were quantified in lab-roasted coffees by HPLC analyses (Table 2). Little differences among lab-roasted coffees in the four phenolic compounds (3.48-6.34 mg 5-CQA, 4.03-5.16 μ g caffeic acid, 37.22-49.15 μ g ferulic acid, and 6.60-7.87 μ g 4-vinylguaiacol per g of coffee)

were observed. These differences, most of them statistically non-significant ($p < 0.05$), seem to be due to the normal variations during roasting process, but not to the addition of sugar during torrefacto roasting process. The most abundant chlorogenic acid in coffee, 5-caffeoylquinic acid, and caffeic acid that is a hydroxycinnamic acid partially originated by hydrolysis of caffeoylquinic and dicaffeoylquinic acids during roasting process, were found in similar amounts than in commercial Colombian coffees (4.3 mg/g for 5-CQA and 5.5 $\mu\text{g/g}$ for caffeic acid) (Lopez-Galilea et al., 2008). However, in commercial torrefacto coffee blends in the latter study (Lopez-Galilea et al., 2008) ferulic acid was found in lower amounts (12.8-19.3 $\mu\text{g/g}$), whereas 4-vinylguaiacol was higher (30.6-57.6 $\mu\text{g/g}$). Ferulic acid is a hydroxycinnamic acid mainly derived from the roasting degradation of feruloylquinic acids (FQAs) that only account for a 5-13% of total chlorogenic acids in green coffee (Farah, Monteiro, Calado, Franca, & Trugo, 2006). And 4-vinylguaiacol is a degradation product of ferulic acid. So that, both compounds might be present in different amounts depending on the initial content of feruloylquinic acids in green coffee and roasting process conditions.

According to the results of Table 2, the addition of sugar during torrefacto roasting process did not reduce the decrease of the main phenolic compounds caused by heat treatment as it was suggested by the Folin-Ciocalteu technique if it is used as a measurement of the total phenolic compounds. This discrepancy in the results might be explained by the formation of other reducing nonphenolic compounds that react with the Folin Ciocalteu reagent during torrefacto roasting process, but not by a protective effect of torrefacto roasting against the degradation of phenolic compounds. This agrees with the results of Lopez-Galilea et al. (2008) who do not find any correlations between phenolic compounds and torrefacto roast in commercial coffee blends. Thus, it could be said that torrefacto roasting process hardly affects the final content of phenolic

compounds that seems to be more influenced by other factors such as roasting degree, the variety of coffee, etc. (Farah et al., 2006).

In conclusion, although the addition of sugar during roasting increases the antioxidant properties of coffee measured as radical quenching capacity, roasting degree and other factors, such as coffee variety, origin or also storage conditions, influence overall antioxidant activity as well. Moreover, in this study, the Absorbance at 420 nm has been proposed as a good marker of torrefacto roasting process, whereas the roasting degree might be better characterized by L* values.

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Table 1: CIELab color parameters (L*, a* and b*) and browned compounds (Abs_{420nm}) of ground roasted coffee.

| Coffee samples ¹ | L* ² | a* ² | b* ² | Abs ₄₂₀ ² |
|-----------------------------|----------------------------|----------------------------|-----------------------------|---------------------------------|
| Lab roasted coffees | | | | |
| <i>Arabica</i> | | | | |
| 0 Dark I | 19.40 ± 0.50 ^{ab} | 11.80 ± 0.29 ^c | 13.16 ± 0.35 ^{cd} | 0.457 ± 0.012 ^c |
| 5 Dark I | 20.23 ± 0.11 ^{ab} | 11.95 ± 0.04 ^c | 13.90 ± 0.16 ^e | 0.499 ± 0.023 ^{de} |
| 10 Dark I | 20.56 ± 0.11 ^b | 11.88 ± 0.14 ^c | 13.30 ± 0.20 ^{cde} | 0.535 ± 0.021 ^f |
| 15 Dark I | 22.45 ± 0.34 ^c | 11.80 ± 0.28 ^c | 15.51 ± 0.31 ^{fg} | 0.574 ± 0.020 ^g |
| 0 Dark II | 19.24 ± 0.81 ^a | 11.73 ± 0.14 ^{bc} | 13.77 ± 0.44 ^{de} | 0.458 ± 0.017 ^c |
| 5 Dark II | 20.10 ± 0.19 ^{ab} | 11.84 ± 0.27 ^c | 13.04 ± 0.30 ^{bc} | 0.483 ± 0.014 ^{cd} |
| 10 Dark II | 20.57 ± 0.26 ^b | 11.90 ± 0.14 ^c | 13.48 ± 0.19 ^{cde} | 0.518 ± 0.015 ^{ef} |
| 15 Dark II | 19.94 ± 0.05 ^{ab} | 11.37 ± 0.20 ^b | 12.45 ± 0.14 ^b | 0.575 ± 0.021 ^g |
| Commercial coffees | | | | |
| <i>Arabica</i> | | | | |
| Colombia Dark | 19.38 ± 0.42 ^{ab} | 9.86 ± 0.02 ^a | 10.12 ± 0.08 ^a | 0.464 ± 0.017 ^{cd} |
| Colombia Medium | 24.82 ± 0.81 ^d | 12.70 ± 0.13 ^d | 15.11 ± 0.40 ^f | 0.352 ± 0.002 ^a |
| Brasil Medium | 25.70 ± 0.13 ^d | 12.88 ± 0.08 ^d | 16.51 ± 0.07 ^h | 0.356 ± 0.018 ^a |
| Kenya Medium | 25.20 ± 0.69 ^d | 12.62 ± 0.18 ^d | 15.83 ± 0.34 ^g | 0.352 ± 0.024 ^a |
| Guatemala Medium | 24.92 ± 0.01 ^d | 12.69 ± 0.02 ^d | 15.73 ± 0.03 ^{fg} | 0.421 ± 0.022 ^b |
| <i>Robusta</i> | | | | |
| Vietnam Medium | 25.40 ± 0.71 ^d | 11.84 ± 0.36 ^c | 15.65 ± 0.56 ^{fg} | 0.379 ± 0.015 ^a |
| Vietnam Light | 30.93 ± 0.64 ^f | 13.49 ± 0.18 ^e | 21.53 ± 0.39 ⁱ | 0.354 ± 0.006 ^a |
| T100 Light | 28.33 ± 0.27 ^e | 13.34 ± 0.24 ^e | 21.61 ± 0.48 ⁱ | 0.599 ± 0.010 ^g |

¹ 0, 5, 10 and 15 is the amount of sugar added (g per 100 g of coffee) during roasting in lab-roasted coffees. Dark, Medium and Light are the roasting degrees. I and II are the batch number in lab-roasted coffees. The origin of coffee is indicated with the name of the country. T100 is commercial 100% Torrefacto roasted coffee.

² All values are shown as means ± standard deviations (n=6). Different letters in the same column indicate significant differences (p < 0.05) among different roasted coffees.

Table 2. 5-CQA, Caffeic acid, Ferulic acid and 4-Vinylguaicol (4VG) amounts of ground roasted coffees.

| Coffee samples ¹ | 5-CQA (mg/g) ² | Caffeic acid (μ g/g) ² | Ferulic acid (μ g/g) ² | 4VG (μ g/g) ² |
|-----------------------------|-------------------------------|---|---|----------------------------------|
| Lab roasted coffees | | | | |
| <i>Arabica</i> | | | | |
| 0 Dark I | 4.44 \pm 0.06 ^{cd} | 4.03 \pm 0.12 ^a | 37.22 \pm 1.18 ^a | 7.46 \pm 0.15 ^{cd} |
| 5 Dark I | 4.74 \pm 0.03 ^d | 4.91 \pm 0.19 ^{bc} | 46.30 \pm 0.46 ^{cd} | 7.67 \pm 0.32 ^d |
| 10 Dark I | 3.96 \pm 0.04 ^b | 4.46 \pm 0.25 ^b | 42.43 \pm 1.91 ^b | 7.37 \pm 0.31 ^{bcd} |
| 15 Dark I | 4.23 \pm 0.13 ^{bc} | 4.90 \pm 0.21 ^{bc} | 44.44 \pm 3.11 ^{bc} | 7.83 \pm 0.44 ^d |
| 0 Dark II | 6.34 \pm 0.07 ^f | 5.16 \pm 0.23 ^c | 49.15 \pm 0.96 ^d | 7.87 \pm 0.14 ^d |
| 5 Dark II | 5.83 \pm 0.22 ^e | 4.86 \pm 0.17 ^{bc} | 45.33 \pm 0.53 ^{bc} | 6.94 \pm 0.14 ^{abc} |
| 10 Dark II | 4.54 \pm 0.03 ^{cd} | 4.81 \pm 0.06 ^{bc} | 44.25 \pm 2.36 ^{bc} | 6.90 \pm 0.09 ^a |
| 15 Dark II | 3.48 \pm 0.08 ^a | 4.55 \pm 0.18 ^b | 38.59 \pm 1.09 ^a | 6.60 \pm 0.13 ^{ab} |

¹ 0, 5, 10 and 15 is the amount of sugar added (g per 100 g of coffee) during roasting in lab-roasted coffees. Dark, Medium and Light are the roasting degrees. I and II are the batch number in lab-roasted coffees.

² All values are shown as means \pm standard deviations (n=6). Different letters in the same column indicate significant differences ($p < 0.05$) among different roasted coffees.

FIGURE CAPTION

Figure 1. Time and temperature conditions of coffee roasting process and control of pan surface (bold line) and air (dotted line) temperatures.

Figure 2. Principal Component Analysis of ground roasted coffee. 0, 5, 10 and 15 is the amount of sugar added (g per 100 g of coffee) during roasting in lab-roasted coffees. Dark, Medium and Light are the roasting degrees. I and II are the batch number in lab-roasted coffees. The origin of coffee is indicated with the name of the country. T100 is commercial 100% Torrefacto roasted coffee.

Figure 3. DPPH antioxidant capacity ($\mu\text{mol Trolox/g}$) of ground roasted coffee. 0, 5, 10 and 15 is the amount of sugar added (g per 100 g of coffee) during roasting in lab-roasted coffees. Dark, Medium and Light are the roasting degrees. I and II are the batch number in lab-roasted coffees. The origin of coffee is indicated with the name of the country. T100 is commercial 100% Torrefacto roasted coffee. All values are shown as means \pm standard deviations ($n = 6$). Different letters indicate significant differences ($p < 0.05$) among different roasted coffees.

Figure 4. Folin-Ciocalteu reducing capacity (g Gallic acid/100g) of ground roasted coffee. 0, 5, 10 and 15 is the amount of sugar added (g per 100 g of coffee) during roasting in lab-roasted coffees. Dark, Medium and Light are the roasting degrees. I and II are the batch number in lab-roasted coffees. The origin of coffee is indicated with the name of the country. T100 is commercial 100% Torrefacto roasted coffee. All values are shown as means \pm standard deviations ($n = 6$). Different letters indicate significant differences ($p < 0.05$) among different roasted coffees.

Figure 1. Time and temperature conditions of coffee roasting process and control of pan surface (bold line) and air (dotted line) temperatures.

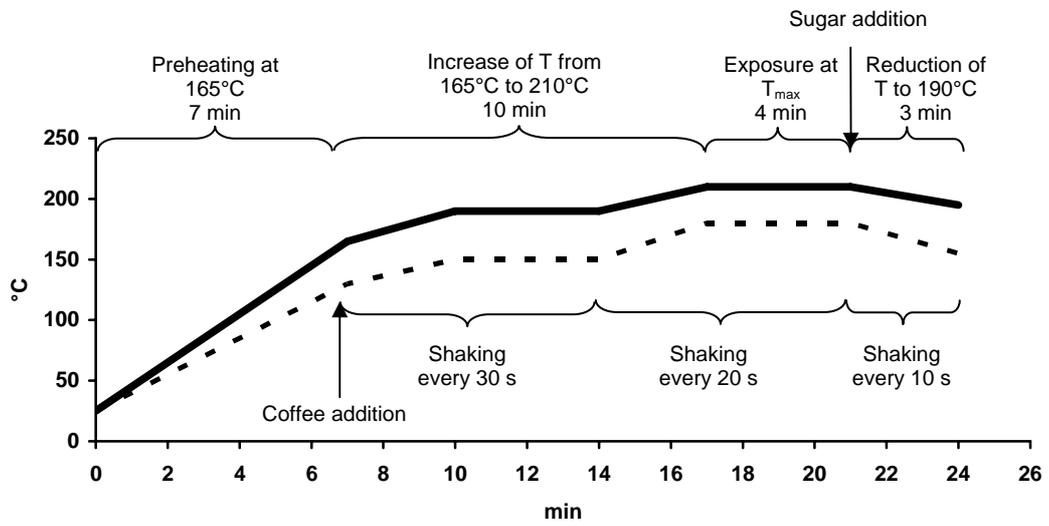


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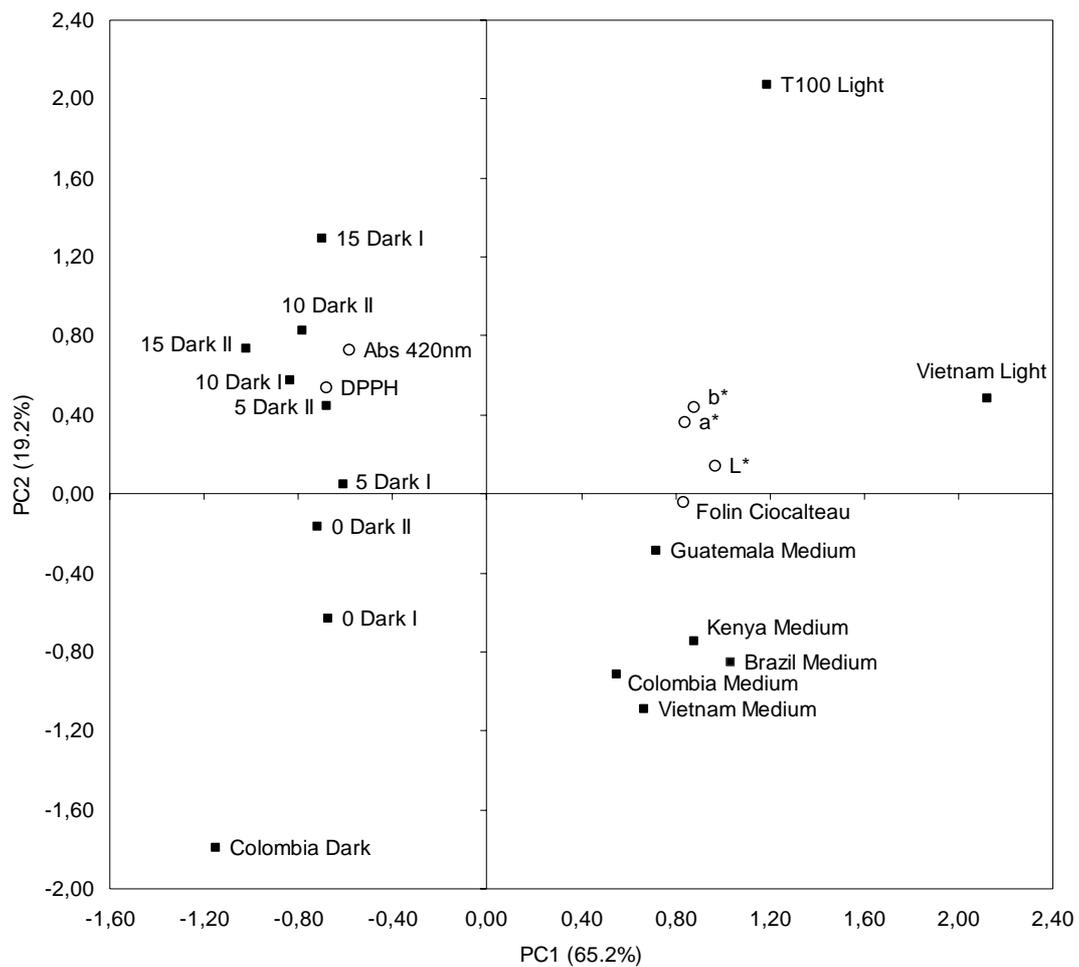


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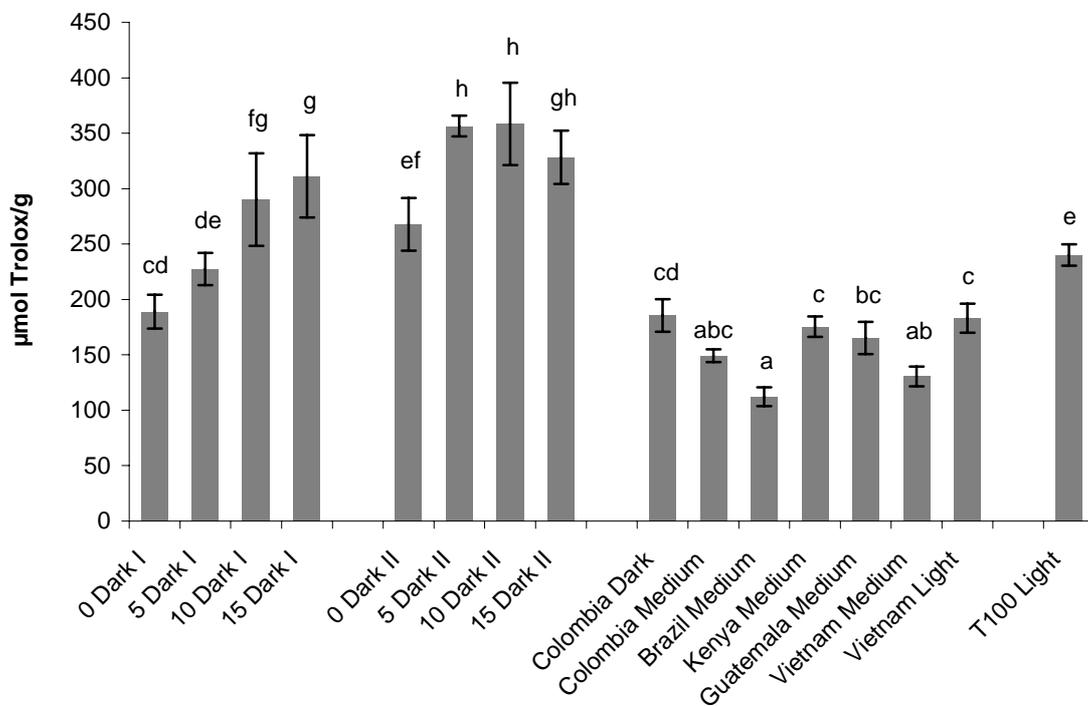


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