Received: 9 December 2021 | Revised: 11 March 2022 | Accepted: 2 April 2022

DOI: 10.1111/jfpp.16626

Journal of Food Processing and Preservation | Institute of Food Science + Technology | ifst | WILEY

ORIGINAL ARTICLE

Use of electrochemical techniques for determining the effect of brewing techniques (espresso, Turkish and filter coffee) and roasting levels on total antioxidant capacity of coffee beverage

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Abstract

In this study, we determined how roasting levels (light, medium, and dark) of Arabica coffee seed and three brewing techniques—decoction methods (Turkish coffee), infusion method (filter coffee), and pressure methods (Espresso)—affect the total antioxidant capacity in a cup of coffee beverage by electrochemical methods such as square wave stripping voltammetry (SWSV), differential pulse stripping voltammetry (DPSV), and cyclic voltammetry (CV). The highest antioxidant capacity was found in espresso coffee prepared with light roasted coffee seeds, as equivalent of rutin and caffeic acid at 9.4 ± 0.2 g/L and 19.7 ± 0.7 g/L, respectively with SWSV on a carbon paste electrode (CPE). The antioxidant capacity of coffee beverages was influenced by the roasting degree, extraction time, and brewing methods, significantly. SWSV, DPSV, and CV voltammetric methods, fast, reliable, fully validated and without any pretreatment, are alternatives to conventional analytical methods for evaluation of antioxidant values in coffee brews.

Practical applications

This research will contribute to the literature considerably since we have established that the antioxidant capacity can be measured by electrochemical methods rapidly with high reliable results. According to the study, the brewing method and roasting temperature significantly affected the antioxidant capacity, and thus, it is important to know how brewing methods, roasting temperatures, and other conditions changes the coffee quality. The results can be used to prepare healthy coffee beverages.

1 | INTRODUCTION

Coffee is an important beverage for most people and accounts for 75% of regular soft drink (which does not contain alcohol) consumption (Ciaramelli et al., 2019), and around 500 billion cups of coffee are consumed per year in the world. The components of coffee are extremely rich in compounds that have various health benefits relate to radical scavenging capability (Cano-Marquina et al., 2013; Ciaramelli et al., 2019; Esquivel & Jiménez, 2012; Farah, 2018). Due to the high potential health benefits of coffee, it is considered as a functional beverage (Cheong et al., 2013; Farah & de Paula Lima, 2019). Rich source of bioactive compounds of coffee beverages with antioxidant capacity arising from both its natural structure and formed compounds after processing (Dybkowska et al., 2017; Hečimović et al., 2011; Kitzberger et al., 2014). The diversity of phenolic components provides potential health benefits, and phenols play an important role in the formation of coffee flavor. Of the more than 800 volatile compounds found in roasted coffee flavors, only 42 have been identified as phenols (Cordoba et al., 2019; Shahidi & Naczk, 2004).

The roasting degree is controlled by the roasting time and temperature and is categorized as light, medium, or dark roast coffee beans (Vignoli et al., 2014). During roasting various chemical reactions including Strecker degradation, Maillard reactions, oxidation,

carbohydrate caramelization, the degradation of polyphenols, and the formation of complex mixture of aroma compounds take place (Sacchetti et al., 2009; Smrke et al., 2013; Wongsa et al., 2019).

Antioxidative capacity of roasted coffee is associated with the degradation of green coffee bioactive compounds particularly polyphenols, such as phenolic acids, mainly chlorogenic acid into caffeic acid, gallic acid, quinic acid, etc., and the formation of some compounds particularly melanoidins (Król et al., 2019; Mehaya & Mohammad, 2020; Pérez-Hernández et al., 2012; Yashin et al., 2013). Time and temperature of roasting can have an influence on the phenols produced and their components (Shahidi & Naczk, 2004; Wongsa et al., 2019). Studies showed that the antioxidant activity of coffee decreases as the roasting degree increases due to the degradation of CGA (Mehaya & Mohammad, 2020; Vignoli et al., 2014).

CGA is an ester formed between caffeic acid and quinic acid (Cano-Marquina et al., 2013; Król et al., 2019) and is hydrolyzed by the intestinal microflora into various aromatic acid metabolites including caffeic acid and salicylic acid (Farah & Donangelo, 2006; Flament, 2001; Itagaki et al., 2011; Król et al., 2019; Olthof et al., 2001). CGA takes part in the formation of color, flavor, and aroma during roasting. Its amount in the coffee beverage depends on the degree of roasting, the grinding, the ratio of coffee to water, the brewing method, the water temperature, and the contact time of coffee with water (Farah & Donangelo, 2006; Kitzberger et al., 2014). CGAs are responsible for the astringent taste of coffee (Flament, 2001).

In an in vitro study of Itagaki et al. (2011), the antioxidant activity of caffeic acid was found to bbe stronger than that of CGA, and the uptake of the CGA by Caco-2 cells was much less than that of caffeic acid (Itagaki et al., 2011). Caffeic acid (3,4-dihydroxycinnamic) is one of the hydroxycinnamate and phenylpropanoid metabolites in plant foods such as coffee drinks, blueberries, apples, and cider (Itagaki et al., 2011; Magnani et al., 2014).

Rutin, vitamin P, is one of the bioactive flavonoid compounds present in plants. Chemically, it is a glycoside comprising of flavonolic aglycone quercetin along with disaccharide rutinose (Atanassova & Christova-Bagdassarian, 2009; Ganeshpurkar & Saluja, 2017; Ghorbani, 2017; Siti et al., 2020). Rutin and its glycoses were found in various fruits, vegetables, tea leaves, coffee grains, etc, and are considered to have high antioxidant capacity because of their ability to bind free radicals and metal ions. Living organisms are incapable of synthesizing it and can be obtained only from plants (Chua, 2013; Koval'skii et al., 2014). Rutin has a high potential for cardioprotective benefits (Siti et al., 2020).

Health benefits of coffee are mainly attributed to its antioxidant capacity of biological compounds such as phenolics, which exhibit a wide range of physiological properties, such as antiallergenic, anti-inflammatory, antiartherogenic, antioxidant, antimicrobial, antithrombotic, vasodilatory effects, cardioprotective, antiaging, and antiproliferative properties (Ciaramelli et al., 2019; Coso et al., 2020; Farah & de Paula Lima, 2019; Lin et al., 2016; Shahidi & Ambigaipalan, 2015; Yashin et al., 2013). Flavonoids modulate a number of biological functions such as anti-inflammatory and anti-microbial activities with their ability to terminate free radicals, chelate metal ions, and scavenge free oxygen (Lin et al., 2016). Coffee may prevent cardiovascular diseases (CVD) and Parkinson's disease, reduce the risk of developing type 2 diabetes and stroke, and prevent the formation of gallstones, Alzheimer's disease, gallbladder diseases, and gout by reducing the level of uric acid in blood, (Al Doghaither et al., 2017; Ding et al., 2014; Lim et al., 2020). Liver diseases may be avoided by drinking two cups coffee in a day (Ergin et al., 2021). In the systematic review and meta-analysis of Ding et al. (2014), they concluded that drinking coffee 3–5 cups per day associated with the reduction in CVD risk, and heavy coffee consumption was not associated with increase in CVD risk. Coffee, which is a source of the caffeine (purine alkaloid; 1,3,7-trimethylxanthine) has very important physiological effects such as increased awaking periods, reduced fatigue (Ludwig et al., 2014), enhanced alertness, concentration, and mental and physical performance (George et al., 2008). Another potential benefit of caffeine was found to be as suppressing body weight gain by stimulating thermogenesis, extending sympathetic stimulation, suppressing food intake, and reducing adipose tissue mass (Ballis, 2019; Shahidi & Ambigaipalan, 2015).

The antioxidant capacity of coffee can be influenced by several factors, such as the variety and origin of coffee, the type and degree of roasting, brewing techniques, and properties of machines used in brewing (Figure 1) (Duarte et al., 2005; Gok, 2021; López et al., 2016; Odžaković et al., 2016; Pérez-Martínez et al., 2010). There are several parameters during the brewing process that affect the extraction mechanism of coffee soluble solids. Volatile and nonvolatile flavor compounds are extracted from ground coffee and form the final quality of coffee beverage. The size reduction of the roasted beans and the contact of water with roasted coffee grounds is essential for the antioxidant composition and health properties of a coffee brew, and it is a crucial step for the extraction of coffee compounds (Figure 1) (Caprioli et al., 2015; Cordoba et al., 2019, 2020; López et al., 2016; Ludwig et al., 2012).

Coffee beverages can be prepared with different methods (Caprioli et al., 2015). Widely, three methods were used: decoction methods (boiled coffee, Turkish coffee, percolator coffee, and vacuum coffee), infusion methods (filter coffee and Napoletana), and pressure methods (Plugger, Moka, and espresso) (Cordoba et al., 2020). The filter, espresso, and Turkish coffee are the widely known coffee brewing methods, and the differences between their extraction techniques with the other popular brewing methods are illustrated in Figure 2. Although Turkish coffee is the oldest type, there are few scientific papers about chemical attributes and flavor profiles compared to the filtered coffee and espresso brewing techniques (Cordoba et al., 2020; Elmacı & Gok, 2021; Gok, 2021).

The preparation techniques of Turkish coffee are different than espresso and filter coffee beverages, where the sediment is present in its beverage, the particle size of ground coffee seed is very small like flour, and water at room temperature is used (Elmacı & Gok, 2021; Gok, 2021). To prepare Turkish coffee, finely ground roasted coffee seeds are mixed with water at room temperature and

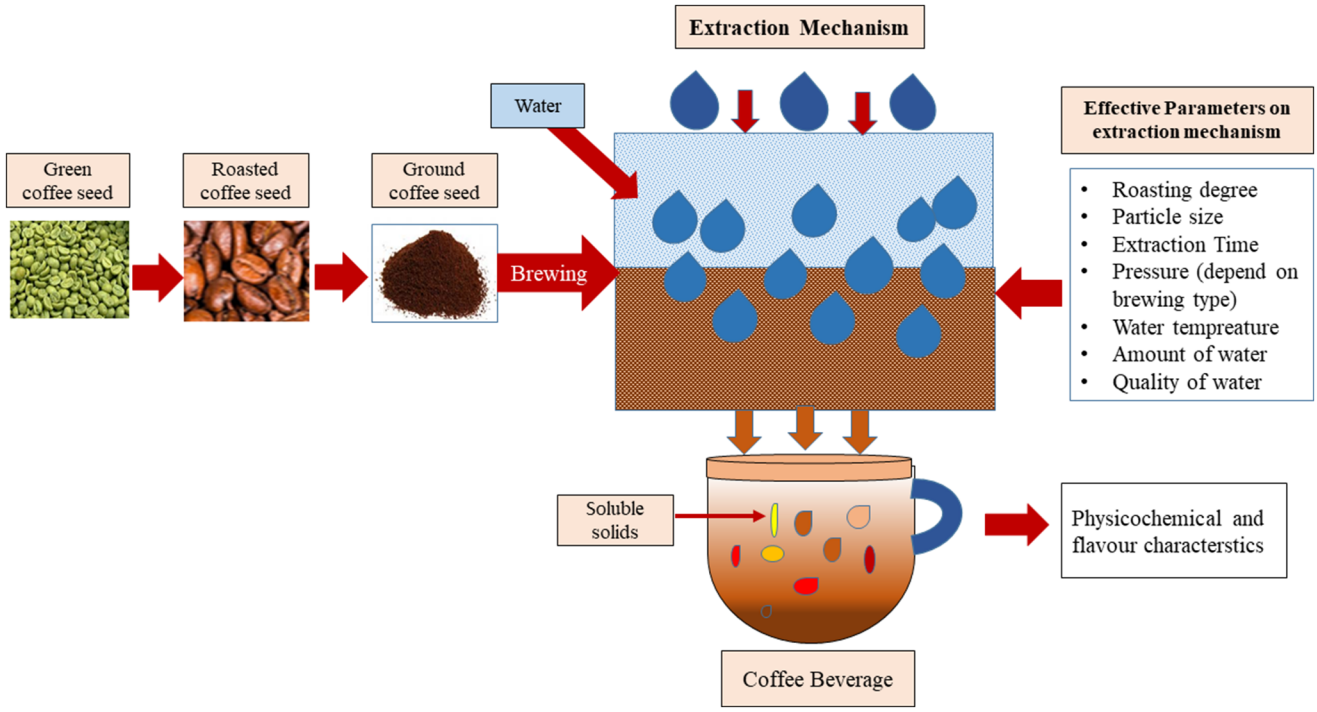

FIGURE 1 The main effective parameters on extraction method for quality of coffee brews (Cordoba et al., 2019, 2020; Gok, 2021; Ludwig et al., 2012)

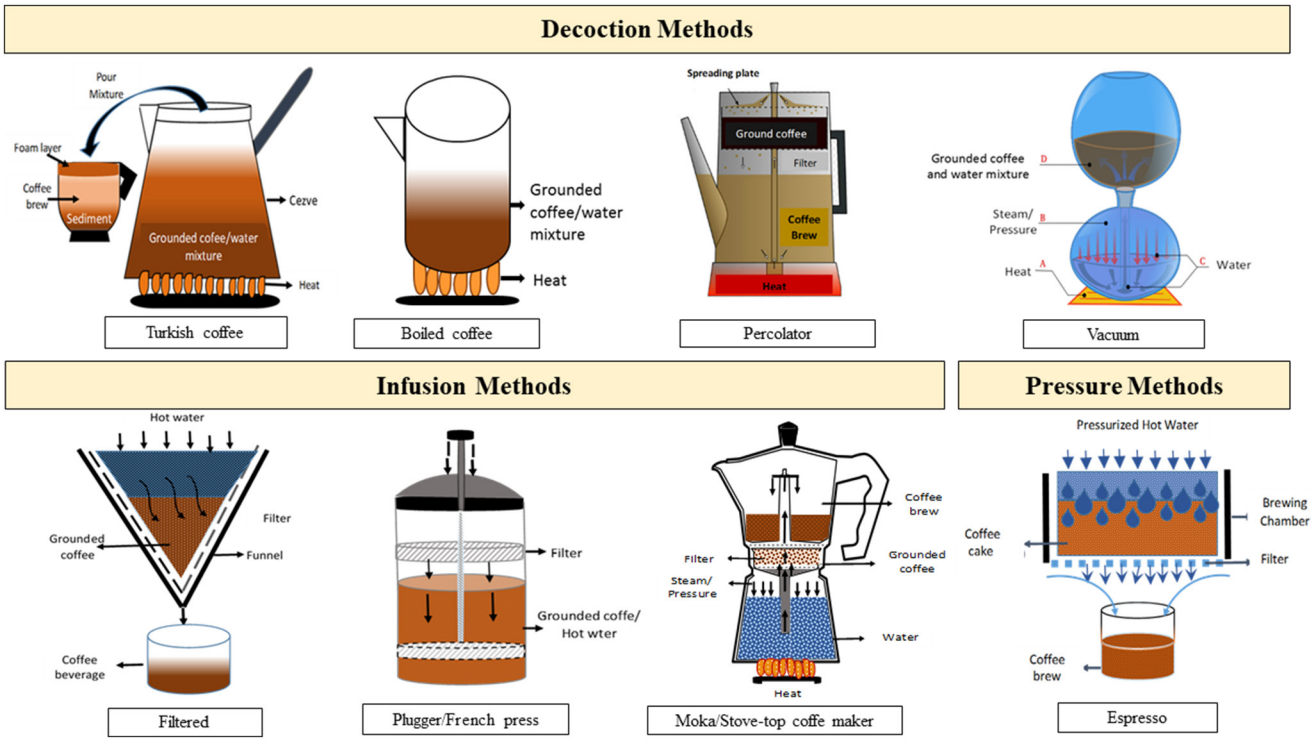

FIGURE 2 Extraction techniques of coffee solubles in brewing methods: decoction methods (Turkish coffee, boiled coffee, percolator coffee, and vacuum coffee), infusion methods (filter coffee), and pressure methods (Plugger, Moka, and espresso) (Cordoba et al., 2020; Elmacı & Gok, 2021; Gok, 2021)

heated until form foams on the surface, ans when the foam starts to increase at nearly 92–95°C, it is poured carefully in a special coffee cup called fincan in Turkish to prevent foam disruption (Figure 1). Strong coffee with a foam layer on the top and settled coffee particles as a sediment (not for drinking) at the bottom is known as Turkish coffee. It is prepared optimally with in 3 min and not boiled (Caprioli

et al., 2015; Cordoba et al., 2020; Elmacı & Gok, 2021; Gok, 2021; Özdestan, 2014).

Cyclic voltammetry (CV) has been commonly used in the fields of organic compounds, inorganic substances, foodstuffs, and biochemical agents for the investigation of electrochemical behaviors, kinetic model studies, and illumination of the mechanism of the electroactive compounds (Ozkan et al., 2015). Furthermore, CV is used not only for the qualitative analysis of electroactive compounds but also for their quantitative analysis. In the last two decades, numerous CV studies have been performed by using various working electrodes in conjunction with developing technology (Demir et al., 2018). In antioxidant assays for the food materials, electrochemical methods have attracted great attention recently because of being cheap, short analysis time, environment friendly, no pretreatment required, and portable. The square wave stripping (SWS) measurements were taken to determine the total antioxidant in different brewed coffee samples from various coffee beans. Differential pulse voltammetry (DPV) and its stripping mode as DPSV are one of the most widely used for the analysis of both organic and inorganic species such as drugs, pesticides, antioxidants, and heavy metals. Pulse voltammetry techniques were proposed by Barker and Jenkin (1952) as a more sensitive as other electroanalytical measurement method. Furthermore, the DPV techniques can be used to determine up to 10^{-8} M concentration of the target agents. In addition, it is possible to analyze substances not only quantitatively but also qualitatively with the pulse technique. The peak currents are related to the concentration of the substance, whereas the peak potential values are related to the selectivity. Thus, simultaneous determinations of the substances have been studied by using DPV methods. Also, according to comparison with others analytical methods, DPV has a fast analysis time, without any pretreatment application, uses less using organic solvent, and is highly selective, and sensitive. It is one of the most important voltametric methods for the determination of antioxidants, since it has a wide working range, has low detection limits, is easy to apply and cheap, and requires no pretreatment (Demir, 2019). The caffeic acid in beverages such as coffee, green tea, and red wine was determined by the electrochemical method by Nehru et al. (2020), and they concluded that monitoring caffeic acid in antioxidant beverages by the CV method is very sensitive and fast and can detect lower concentrations up to nanomolar limits and has a high potential for the detection of antioxidants in healthcare system safely (Nehru et al., 2020).

The aims of this study were to determine the total antioxidant capacity as equivalent caffeic acid and rutin in three different coffee brewing techniques (Turkish coffee, filter coffee, and espresso) at three roasting degrees (light, medium, and dark) by using CV, SWS, and DPV techniques, and to investigate the effect of roasting degree and brewing techniques on the antioxidants (caffeic acid and rutin content) and the total antioxidant capacity, and also the compare results with other methods used before. This paper is a novel study for the comparison of antioxidant levels of Turkish coffee with filter and espresso brewing techniques at different roasting temperatures to optimize a novel, fast, and simple method for the determination of antioxidant levels of coffee with CV, SWS, and DPV techniques.

2 | MATERIALS AND METHODS

2.1 | Electrochemical measurements

The electrochemical measurements were carried with in a Vertex® One (Ivium) electrochemical analyzer with a solid electrode cell stand, to which the three-electrode system was connected. The solid electrochemical cell connected with a carbon paste electrode (CPE, BASi MF-2010) was the working electrode, an Ag/AgCl (BASi, MF-2052) was the reference electrode, and a platinum wire was the auxiliary electrode (BASi, MW-1032). In order to obtain electrochemical signals, cyclic voltammetry (CV), differential pulse stripping voltammetry (DPSV) and square wave stripping voltammetry (SWSV) were used under the optimized conditions. For the SWSV, accumulation potential, accumulation time, pulse amplitude, step potential, and frequency were found 0 mV, 30 s, 50 mV, 5 mV, and 100 Hz, respectively. Furthermore, accumulation potential and accumulation time was deduced to be 0 mV and 30 s for the DPSV method at pulse time of 10 ms and pulse amplitude of 50 mV on CPE in pH 4.0 Brittion–Robinson (B–R) buffer solutions. Each of the voltammograms were measured thrice under optimum conditions and statistical calculations were made. The pH measurements were carried out by using a Mettler Toledo pH meter with an accuracy of ±0.05, and electrochemical processes were performed at room temperature.

2.2 | Reagents

Standard analytical phenolic compounds of caffeic acid and rutin were purchased from Sigma-Aldrich. All standard phenolic solutions were prepared at a concentration of 500 mg/L, and Britton–Robinson (B–R), pH 4.0 buffer solution using a support electrolyte was prepared at 0.04 mol/L of acetic acid, ortho phosphoric, and boric acid. The 2.0 M NaOH or 2.0 M HCl solutions were used to adjust the supporting electrolyte at B–R buffer at pH 4.0. Distilled water was used in all solution preparation, washing, and electrode cleaning processes. Also, all prepared stock solutions were stored in a refrigerator at 4°C in a dark environment when not in use.

2.3 | Process preparation of coffee samples

The handcrafted roasted coffee seed blends (Colombia, Costarica, and Guatemala) used in the experiments were obtained from third wave coffee producer company (Gastro Coffee Roaster). Equal amount of seeds (500 g for each seed) were mixed and the blend were prepared. The company roasted coffee beans with same standards in the roaster (A roasting machine with PLC control system and a 15 kg capacity destoner connection By HasGaranti) at three different

temperatures as light (105 Agtron Gourmet/180°C), medium (85 Agtron Gourmet/205°C), and dark (50 Agtron Gourmet/210°C) and then were ground in an industrial mill by HasGaranti mill as fine powder (≤120 μ) for Turkish coffee samples, coarse size (≤850 μ) for filter coffee, and bean size (≤350 μ) for espresso was prepared before brewing (The espresso machine grinds the core itself).

Turkish coffee was prepared using the automatic Turkish coffee maker (Arzum Okka). 7.0 g ground coffee and 80 ml distilled water at room temperature were added, mixed, and heated until foamed twice 92–95°C (Elmacı & Gok, 2021). Espresso coffee was prepared with 7.0 g ground coffee and nearly 35 ml distilled water by using the Jura Impressa XS9 Classic espresso machine at pressure of 9 bar (nearly 9 atm), and 90–95°C. Filter coffee samples were prepared with the 7.0 g ground coffee with 125 ml purified water at 95–97°C. All coffee brews were cooled to room temperature and then centrifuged for 5 min. The all coffee samples were stored at −20°C when not in use. Before the entire brewing process, the coffee machines were thoroughly washed with ultra-pure water.

3 | RESULTS AND DISCUSSION

3.1 | Cyclic voltammetry measurements

In this study, we obtained cyclic voltammograms of rutin and caffeic acid (1 mg/L) for the identification of peak signal and peak potential at a scan rate of 100 mV/s on the CPE between −200 and +1300 mV potentials (Figure 3). It is also known that the peak position of phenolic compounds varies depending on the pH values. Therefore, a single support electrolyte as a pH 4.0 B–R buffer solution was used for each substance. Among these substances, only the caffeic acid exhibited a reversible electrode reaction, while other has an irreversible reaction as oxidation according to the CV data's. Furthermore, the caffeic acid exhibited an oxidation peak at 0.38 V by CV on CPE. Rutin (0.44 V and 1.03 V) had two anodic peaks (Figure 3). However, rutin and caffeic acid have an oxidation peak at about 0.4 V and also. However, the first anodic peak of rutin and the oxidation signal of caffeic acid are quite close to each other. This peak, seen at about 0.4 V, is formed by the ketone oxidation of phenol groups in the structure of substances. Therefore, qualitative analyzes of coffee samples for the antioxidant capacity evolution were determined based on this peak potential for standard phenolic compounds. In addition, in order to reckon quantitate analysis for total antioxidant capacity (TAC) of coffee samples by CV, the concentration of rutin and caffeic acid was used as 1 mg/L. The main reason for this is that the peak of caffeic acid and rutin is very sensitive and very intense on CPE at pH 4.0 B–R buffer solution. As can be seen in Figure 3, the peak current values of the caffeic acid and rutin at approximately 0.4 V were found to be 0.801 ± 0.025 μA and 0.331 ± 0.009 μA, respectively, by CV at 100 mV/s scan rate in pH 4.0 B–R buffer solutions on CPE.

The CV's were taken to evolution total antioxidant capacity of the light, medium and dark roasted coffee beans by its brewing samples of Turkish coffee, espresso, and filter coffee at 100 mV/s on the CPE in pH 4.0 B–R buffer solution. Referring to Figure 3, the CV obtained at three roasted degree of Turkish coffee samples were seen, and the slightly roasted Turkish coffee sample has a reversible oxidation peaks at about 0.4 V. This obtained peak represents exactly caffeic acid standard due to its similar peak potential of caffeic acid has one reversible peak at nearly 0.4 V in pH 4.0. Furthermore, total antioxidant capacities of coffee beverages were calculated in terms of equivalent rutin and caffeic acid (Table 1).

According to the results obtained by CV for the three roasted temperature and three brewing techniques, the maximum amount of antioxidant capacity was found in espresso brewing technique with light roasted coffee seed. However, no peak was observed in the dark roasted bean samples. The higher roasting degrees removed caffeic acid, which is one of the important phenolic acids in coffee, by the effect of heat. Mehaya and Mohammad (2020) determined the gradual decrease in phenolic compounds like caffeic acid at increasing time and temperature as in this study. The thermostability of CGA and caffeic acid is 207°C and 223°C, respectively. That is why they are not resistant to high temperatures and their degradation rate increase at higher temperature (Mehaya & Mohammad, 2020). As a result, we can conclude that roasting level and brewing techniques directly affect antioxidant capacity of coffee beverage. The maximum antioxidant capacity was found in light roasted espresso as equivalent the rutin and caffeic as 1128 ± 41.0 mg/g and 2714 ± 49.5 mg/g, respectively, according to the CV technique.

3.2 | Square wave stripping voltammetry measurements

We researched electrochemical behavior of standard rutin (0.5 mg/L) and caffeic acid (0.5 mg/L) for identification of references peak signal and peak potential for the phenolic compounds under the optimum conditions in pH 4.0 on CPE. Caffeic acid exhibited an oxidation peak at 0.38 V, while rutin has two oxidation peaks at 0.45 V and 1.03 V (Figure 3). While the peak potential values obtained by SWSV were almost the similar for both items according to the CV data, a large increase in peak currents on SWS voltammograms was observed for the both phenolic compounds. This also proves that the % sensitivity difference is between them, even at 4 times less concentration. The basic common feature of both substances is that they have an oxidation peak at about 0.4 V. Therefore, for the qualitative determination of the antioxidant capacity of coffee beverages, the peak potential and peak intensity values at approximately 0.4 V of the standard phenolic compounds by SWSV were referenced. Furthermore, to make analysis of total antioxidant capacity (TAC) for the coffee beverages by SWSV, the concentration of rutin and caffeic acid was used at 0.5 mg/L under the optimum condition in pH 4.0 B–R buffer solutions. The main reason for this is that the peaks of caffeic acid and rutin in the SWSV is very sensitive than CV. As can be seen in Figure 3, the peak current of the caffeic acid and rutin at approximately 0.4 V

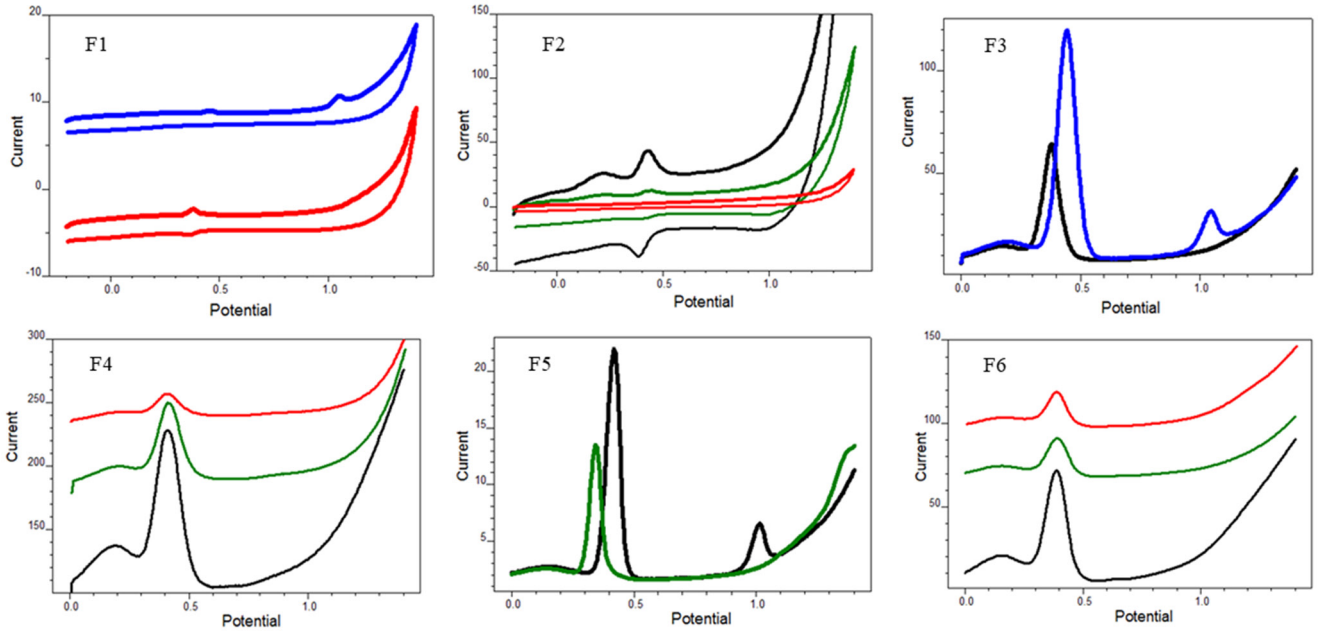

FIGURE 3 Peak currents of samples; F1: The CV for the standard phenolic compounds at 100 mV/s scan rate in pH 4.0 B-R buffer solutions. F2: The CV of Espresso coffee samples prepared with light, medium and dark roasted coffee beans at 100 mV/s scan rate in pH 4.0 B-R buffer solutions. F3: The SWSV for the standard phenolic compounds under the optimum condition in pH 4.0 B-R buffer solutions. F4: The SWSV for the filtered coffee samples brewing from various coffee beans (Eacc = 0 mV, tacc = 30 s, ΔE = 50 mV, Es = 5 mV, f = 100 Hz in pH 4.0 B-R buffer solutions). F5: The DPSV for the standard phenolic compounds under the optimum condition in pH 4.0 B-R buffer solutions. F6: DPSV for the Espresso coffee samples brewing from various coffee beans (Eacc = 0 mV, tacc = 30 s, ΔE = 50 mV and ΔEt = 10 ms in pH 4.0 B-R buffer solutions)

TABLE 1 Equivalent amount of rutin and caffeic acid of the Turkish coffee, filter coffee, and espresso brewing at three roasting temperatures determined by CV, SWSV, and DPSV

Coffee Sample/ Method	Equivalent rutin (mg/g)			Equivalent caffeic acid (mg/g)		
	Brewing technique			Brewing technique		
	Turkish coffee	Espresso	Filter coffee	Turkish coffee	Espresso	Filter coffee
CV						
LR	72.9 ± 3.43^{aA}	1128 ± 41.0^{bF}	108.9 ± 5.36^{cA}	537 ± 21.7^{aC}	2714 ± 49.5^{bF}	262.5 ± 14.3^{cF}
MR	5.71 ± 0.0^{aB}	207 ± 5.51^{bE}	76.8 ± 14.3^{cB}	1141 ± 30.9^{aD}	499 ± 13.5^{bE}	185.7 ± 32.1^{aD}
DR	ND	ND	ND	ND	ND	ND
SWSV						
LR	26.3 ± 1.14^{aC}	47.0 ± 1.0^{bC}	92.9 ± 3.57^{cC}	57.1 ± 1.14^{aB}	98.5 ± 3.5^{bC}	208.9 ± 5.36^{cE}
MR	4.57 ± 0.0^{aD}	16.5 ± 0.5^{bAB}	42.86 ± 1.79^{cD}	10.3 ± 0.0^{aA}	37.0 ± 1.0^{bAB}	94.6 ± 3.57^{aAC}
DR	1.14 ± 0.0^{aE}	12.0 ± 0.5^{bA}	14.29 ± 0.0^{cE}	2.17 ± 0.0^{aA}	24.5 ± 0.5^{bA}	32.1 ± 1.79^{cA}
DPSV						
LR	34.3 ± 1.14^{aF}	67.0 ± 0.5^{bD}	26.8 ± 1.79^{cF}	65.1 ± 2.29^{aB}	129 ± 5.0^{bD}	50.0 ± 8.93^{cB}
MR	2.29 ± 0.0^{aG}	25.0 ± 0.5^{bB}	17.9 ± 0.0^{cG}	3.43 ± 0.0^{aA}	48.0 ± 1.0^{bB}	32.1 ± 0.0^{cA}
DR	ND	18.0 ± 0.5^{bAB}	10.7 ± 0.0^{cH}	ND	35 ± 0.5^{bAB}	19.6 ± 1.79^{cA}

Notes: a, b, c means within each row, and A, B, C, D, E, F, G, H means within each column with different letters are significantly different for each attribute ($p < .05$).

were found to be 48.212 ± 1.743 μA and 102.112 ± 3.682 μA, respectively, by SWSV under the optimum condition on CPE.SWS measurements were taken to determine the total antioxidant in different brewed coffee samples. Also, each measurement was repeated thrice under the optimum conditions in pH 4.0 B–R buffer solutions on CPE. As can be seen in Figure 3, in the SWS voltammograms for the Turkish coffee brews at three roasting temperature (light, medium and dark) have an oxidation peak at nearly 0.4 V (Figure 3). The obtained oxidation peak at 0.4 V by SWSV for coffee samples may represent the peak as an antioxidant

reference peak, since it generally has the same potential of phenolic compounds. In addition, the total antioxidant capacities of coffee samples were calculated in terms of rutin and caffeic acid based on peak intensities for the standards both phenolic compounds (Table 1).

According to the results obtained by SWSV for coffee brews, the maximum antioxidant capacity was found in the espresso brew from light roasted coffee beans. Moreover, while the antioxidant capacity of dark roasted coffee beans was not determined by CV as it is not as sensitive as other electroanalytical methods, it was seen that quantitative total antioxidant determination was possible by SWSV method. The obtained results by SWSV are consistent with the CV results. Different roasting levels and brewing techniques have a direct effect on the phenolic compound contents and thus have a significant effect on their antioxidant capacity. The antioxidant capacity of filter coffee at light roasting degree was the maximum amount when compared to the Turkish and espresso brewing as equivalent the rutin and caffeic by 92.9 ± 3.57 mg/g and 208.9 ± 5.36 mg/g, respectively.

3.3 | Differential pulse stripping voltammetry measurements

Moderated conditions for the DPSV such as pulse time of 10 ms, pulse amplitude of 50 mV, step potential of 5 mV, accumulation time of 30 s, and accumulation potential of 0 mV were optimized in pH 4.0 B–R buffer solutions. Afterward, electrochemical measurements were carried out by DPSV in the presence of standard rutin and caffeic acid at 0.5 mg/L, in order to deduce the total antioxidant amount in the coffee samples (Figure 3). There is a single oxidation peak for the caffeic acid at approximately 0.38 V. Over and above, two anodic peaks at 0.45 V and 1.03 V were obtained for the rutin by DPSV. Potential values of peaks designated by DPSV for both substances are almost identical to CV and SWSV. The total antioxidant capacities of coffee samples can be identified using the peak potential of 0.4 V for three proposed voltammetric methods of standard substances. Peak height values of standard substances were determined depending on the concentration under moderated conditions (pulse time of 10 ms, pulse amplitude of 50 mV, step potential of 5 mV, accumulation time of 30 s, and accumulation potential of 0 mV were optimized in pH 4.0 B–R buffer solutions). According to the Figure 3, the peak current values of the caffeic acid and rutin at approximately 0.4 V were found as 10.471 ± 0.450 μA and 20.101 ± 0.162 μA, respectively, by DPSV on CPE in pH 4.0 BR buffer solutions. According to these results, DPSV method is very sensitive compared to CV, and sensitivity is low compared to SWSV.The antioxidant capacity of three coffee beverages varied as espresso > filter coffee > Turkish coffee at three roasted levels by DPSV measurements (Table 1). No peak was obtained for dark roasted beans of Turkish coffee by the DPSV method in pH 4.0 B-R buffer solution (Figure 3). The obtained values by DPSV are consistent with CV and SWSV results. The highest antioxidant capacity of coffee brews between three roasting levels as equivalent rutin and caffeic acid was found in espresso at light roasted degree 67.0 ± 0.5 mg/g and 50.0 ± 8.93 mg/g, respectively.

Consequently, when we compare three voltammetric methods, according to the studies conducted with CV and DPSV, lightly roasted espresso showed the highest antioxidant capacity, while SWSV was obtained in lightly roasted filter coffee. The considering recommended methods, as the roasting degree increased, the peak currents decreased and therefore the antioxidant values in it were low.

3.4 | Effect of roasting and brewing

Antioxidant capacities of Arabica coffee at three different roasting temperatures brewed with espresso, Turkish, and filter coffee techniques were determined by electrochemical methods such as SWSV, DPSV, and CV in terms of the equivalent amounts according to the standard rutin and caffeic acid, which exhibited peaks at approximately 0.4 V in pH 4.0.

The antioxidant capacity of coffee beverages changed significantly with both brewing techniques and roasting temperatures. Antioxidant activities were highest in espresso and moderate in filter coffee; however, they were the lowest in Turkish coffee. The amount of rutin and caffeic acid decreased as the roasting temperature increased. Roasting intensity, brewing type, time of exposure, type of filtering, coffee grinding size, and amount of water used may affect the chemical composition of coffee beverage (Caprioli et al., 2015; Cordoba et al., 2020; López et al., 2016).

In order to observe the effect of roasting degree and brewing techniques on the antioxidant activity, Equation (1) was used. The effect was followed by using the antioxidant value at light roasting (LR) as an initial value. Percent loss was calculated by dividing the antioxidant level of medium and dark roasting to their initial LR value. The values of rutin and caffeic acid obtained are shown in Table 1 and represented in Figure 4.

$$\text{Loss}\,\% = \frac{\text{Antioxidant value}}{\text{Antioxidant value at LR}} \times 100 \quad (1)$$

The first variable that exhibits large differences between rutin and caffeic acid contents in three brewing techniques was the roasting degree where the amount of metabolites is decreased by the reaction occurring during the roasting. The temperatures were 180°C for light, 205°C for medium, and 210°C for dark roasting levels. As seen from the temperatures, very narrow temperature increase was needed to reach the dark roasting level from medium roasting, nearly 5–10°C heating. Coffee seeds reached the dark level in a short time and showed that after a point, as temperature increase the reaction rate increase rapidly, coffee seeds becomes more thermolabile. Rutin and caffeic acid have similar temperature sensitivity, where the decreasing rate of each brew was nearly similar at the same roasting degree (Figure 4). For example, reduction in both caffeic acid and rutin contents of filter coffee was 55% for SWSV, 29% for CV, and 34% for DPSV method at medium

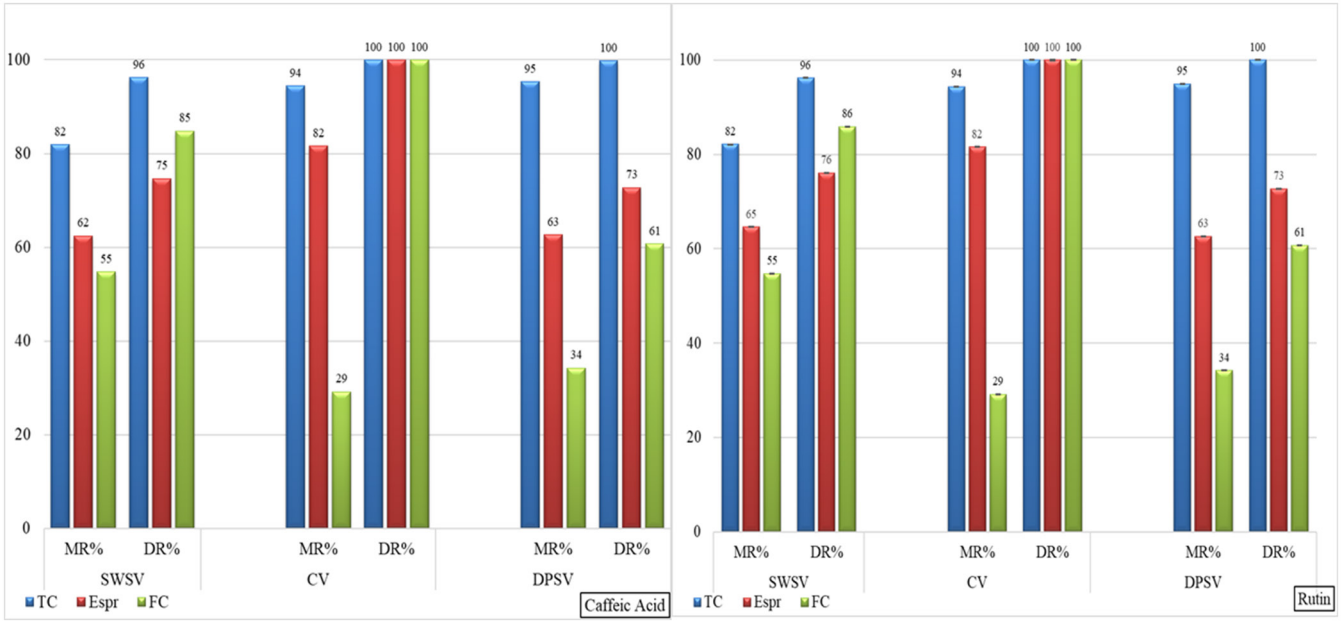

FIGURE 4 Percent loss in caffeic acid and rutin at medium roasting (MR) and dark roasting (DR) of coffee beverages prepared by three different brewing technique (TC: Turkish coffee, Espr: Espresso, FC: Filter coffee)

roasting. Compared to brewing techniques, espresso has highest rutin (225.5 g/L) and caffeic contents (542.8 g/L), then filter coffee, and Turkish coffee (CV method) at light roasting degree. There were no available data that studied the rutin and caffeic acid contents of coffee beverages in the literature to compare.

For determination of effects of geographic regions, roasting degree profiles of CGA isomers on coffee beverages brewed by 10 min were estimated. Contact of coffee with boiling water (100°C) at a ratio of 1:20 (w/v) showed the highest decrease, which was higher than 85% at dark roasting, compared to light and medium roasting degree. Also, the antioxidant activity of coffee samples obtained from different regions was changing at same conditions (Liang et al., 2016).

Total phenol content of espresso, Turkish, and Americano coffee was 22.01 mg GAE/ml, 9.87 mg GAE/ml and 5.05 mg GAE/ml, and the total antioxidant activity was 10.91 mg Trolox/ml for 5.45 mg Trolox/ml, and 3.47 mg Trolox/ml, respectively, in the research of Derossi et al. (2018). They found that grinding levels had slight effect on the caffeine and phenol activity of coffee prepared by espresso, Turkish, and Americano brewing. Controversial to the Derossi et al. (2018), Severini et al. (2015) concluded that among the variables of the quality of espresso coffee, grinding was one of the most important factors at constant volume, 9 bar pressure, and 92°C. Coarsest coffee ground provided more water flow and thus the highest percolation rate and high caffeine amount (Derossi et al., 2018; Severini et al., 2015).

The study by Cordoba et al. (2020) showed that in espresso brews, CGA was higher than dripped/filtered or other brewing methods. According to results, CGA and caffeine content of espresso was 3–6 times higher than moka and filtered coffees (Cordoba et al., 2020). The reasons were explained by higher ratio of ground roasted coffee per ml of water and high-pressure techniques used for extraction in the espresso machine (mostly 9 bar). Lower CGA in cold brewing method was attributed to low water temperature used in extraction. Water temperature of cold brew is generally between 20–25°C or at lower temperatures, and infusion periods can change from 6 to 24 h (Cordoba et al., 2021; Fuller & Rao, 2017; Portela et al., 2021). The studies concluded that heating up to 95°C provides higher extraction of CGA but holding coffee brews at elevated temperatures (>95–98°C) may result in reduction in antioxidant values such as in Turkish coffee brewing method (Cordoba et al., 2020; Farah & de Paula Lima, 2019; Fuller & Rao, 2017).

Yıldırım et al. (2020) and Sridevi et al. (2011) showed in their research that the method of coffee brewing and roasting temperature had significant effect on total solid content and chemical composition in coffee brew. Coffee prepared with espresso had highest solid content than Turkish coffee and the filter coffee was the least one (Sridevi et al., 2011; Yıldırım et al., 2020). Zhang et al. (2012) explained that cafestol extraction yield of coffee beverage depends on the brewing type and degree of roasting. Coffee beverage prepared by boiling and using French press for light roasted coffee seeds had the higher cafestol level than Turkish coffee and Mocha, and the lowest level was found at dark roasted brewed with Turkish coffee and Mocha (Zhang et al., 2012).

Gorjanović et al. (2017) determined the antioxidant capacity of coffee for instant, espresso, filter and Turkish brews by DC polarographic assay method. They found that the antioxidant capacity of espresso, filter, and Turkish coffee samples were similar and that instant coffee has the highest value. There was not any information about the origin and roasting degree of coffee in the study (Gorjanović et al., 2017). Result of Gorjanović et al. (2017) are different than Yıldırım et al. (2020).

Yıldırım et al. (2020) determined the antioxidant capacity of light (180°C), medium (205°C), and dark roasted (210°C) Arabica seed brewed Turkish coffee and filer coffee as equivalent gallic acid and

quercetin by electrochemical methods. Results showed that light roasted coffee beverage has the highest antioxidant capacity and filter coffee brew antioxidant capacity was higher than that of Turkish coffee at all three-roasting level (Yıldırım et al., 2020), as in this study. According to the most of studies, decrease in the CGA content of coffee beverages were observed with the increasing roasting temperature and extended time as in our study. Loss in the amount of CGA in the browning reactions such as Maillard reactions does not mean a decrease in antioxidant capacity because there are also browning products formation (Liang et al., 2016). Maillard reactions are noncovalent reactions of CGA isomers and melanoidins that result in products with increasing antioxidant activity (Kocadağlı & Gökmen, 2016; Liang et al., 2016). But as the time and temperature increase, caffeic acid and CGA degradation rate increase due to their thermal sensitivity (Mehaya & Mohammad, 2020).

3.5 | Effect of extraction time

Extraction time is explained as another crucial parameter that determine the coffee beverage quality (Caprioli et al., 2015; Cordoba et al., 2020; López et al., 2016). It is contact time of water with ground coffee seed in the brewing methods. Soluble compounds are dissolved based on the extraction techniques and washed away with water. When extraction time is completed, coffee beverage with extracted soluble solids is ready for drinking (Figures 1 and 2). Extraction time was 3 min for Turkish coffee, 2 min for filter coffee and 25–30 s for espresso. The particle size of ground coffee seed used for Turkish coffee was fine powder which smaller than other two methods and, ratio of water/ground coffee seed used was also different. Lowest antioxidant level in Turkish coffee than other two methods may be due to differences in long extraction time, amount of water used and smallest particle size. In the study of López et al. (2016), they explained that there is significant effect of applied pressure and water temperature between espresso brewing techniques on extraction kinetic. High pressure (11 bar) increased volatiles with increased water temperature (López et al., 2016). Pressure creates driving force for flow of water through compact coffee cake and makes easy for extraction of the soluble particles and oil droplets from ground coffee beans. They also explained that most polar compounds were extracted within the first s and at longer extraction time, and decreases in their concentrations were observed. Ludwig et al. (2012) found the higher phenolic compounds in filter coffee than espresso. The reason was explained as contact time of water with ground coffee, and they concluded that brewing time has one of the key factors determining the antioxidants level in coffee beverage (Ludwig et al., 2012).

4 | CONCLUSIONS

In our work, we presented a new electrochemical method such as square wave stripping voltammetry (SWSV), differential pulse stripping voltammetry (DPSV), and cyclic voltammetry (CV) for constructing antioxidant capacity of coffee brews prepared with three different brewing techniques at three roasting compounds. To the best of our knowledge, there were no study using these methods for the determination of antioxidant capacity of coffee beverages, which are fast, reliable, fully validated, and may be used for determination of antioxidant capacity of coffee beverages in the future.

Rutin and caffeic acid substances were used to determine the antioxidant levels of coffee brews, because these two substances exhibited the anodic peak signal at a potential of about 0.4 V in pH 4.0 B–R buffer solutions on a carbon paste electrode (CPE). This signal is due to the oxidation of phenols to ketones. Thus, it shows that antioxidants containing phenolic groups can be determined by electrochemical methods. The antioxidant capacities of the samples prepared with three different brewing and three different roasting techniques were successfully investigated without any pretreatment. The highest antioxidant capacity was seen in espresso coffee prepared with lightly roasted coffee beans and was calculated as 1128 ± 41.0 mg/g and 2714 ± 49.5 mg/g, routine and caffeic, respectively, by using the CV technique. They are important organic compounds of coffee and have health benefits. Our data showed that roasting temperature, brewing method, extraction time, and ratio of ground coffee seed/water have a considerable effect on the antioxidant capacity of coffee brews and influence the potential health benefits.In this study, it was difficult to compare the data with literature. Because antioxidant activity data of coffee beverages in the most research was obtained from various methods and thus result in the differences between studies with inconsistent data. Also, the amount of ground coffee and water ratio, the extraction time, temperatures of roasting, type of coffee seeds, variation between brewing techniques, type of data representation with used units such as w/v, v/w, w/cup, and w/dose changes and makes it difficult for comparison and correlation between results. But these inconsistent results provide new opportunities to make further research.

AKNOWLEDGEMENTS

This study is performed as a multidisciplinary Master of Science thesis study of Sevinc Yildirim with advisor Ilkay Gok and co-advisor Ersin Demir supported by Istanbul Okan University, Faculty of Applied Sciences, Gastronomy and Culinary Art Department and Afyonkarahisar Health Sciences University, Analytical Chemistry Department, respectively. The authors thank Kurukahveci Mehmet Efendi Mahdumları Ltd. Şti. for purchasing chemicals and some equipments used during analysis, Arzum Elektrikli Ev Aletleri San. ve Tic. A.Ş. for donation of Turkish coffee machine, and Gastro Coffee Roastery Ltd. Şti. for preparation and donation of handcrafted specialty coffee samples and providing the espresso coffee machine.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest.

AUTHOR CONTRIBUTIONS

Sevinc Yildirim: Conceptualization; data curation; formal analysis; investigation; methodology; software; validation; visualization. **İlkay Gök:** Conceptualization; data curation; formal analysis; funding acquisition; investigation; methodology; project administration; writing – original draft; writing – review and editing. **Ersin Demir:** Conceptualization; data curation; formal analysis; investigation; methodology; software; validation; visualization; writing – original draft. **Ozlem Tokusoglu:** Investigation; validation; writing – original draft; writing – review and editing.

DATA AVAILABILITY STATEMENT

Data available on request from the authors.

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How to cite this article: Yildirim, S., Gok, I., Demir, E. & Tokusoglu, O. (2022). Use of electrochemical techniques for determining the effect of brewing techniques (espresso, Turkish and filter coffee) and roasting levels on total antioxidant capacity of coffee beverage. *Journal of Food Processing and Preservation*, *00*, e16626. https://doi.org/10.1111/jfpp.16626