**ORIGINAL RESEARCH** 



# Extraction of Phenolic Compounds from Cornelian Cherry (*Cornus mas* L.) Using Microwave and Ohmic Heating Assisted Microwave Methods

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#### Abstract

In this study, it was aimed to extract bioactive compounds (phenolics and anthocyanins) from cornelian cherry (*Cornus mas* L.) using microwave (ME) and ohmic heating assisted microwave extraction (OH-ME) as an alternative to maceration (M). Total phenolic compound (TPC), total monomeric anthocyanin content (TMAC), antioxidant activity (AA), water-soluble solid matter (WSSM), color values, and pH of the extracts were determined. The effect of extraction time, solid to solvent ratio, microwave power, electrical field strength, and holding time during ohmic heating on extraction characteristics was investigated for different methods. Ohmic heating application prior to extraction has enhanced the TPC yield by 1.1 and 5.4-folds compared to that of ME and M, respectively. Moreover, the extraction time was decreased by approximately 42 and 95% as regards ME and M, respectively. The highest TMAC value (0.65 mg cya-3-glu/g dry matter) was observed with OH-ME. The experimental data obtained by ME and OH-ME methods was fitted to the Peleg model to describe the TPC yields. These results indicate that the combined application of ohmic heating and microwave extraction is a potential alternative method for total phenolic compounds extraction from cornelian cherry due to increased yields and reduced extraction times.

Keywords Microwave extraction · Ohmic heating · Cornelian cherry · Phenolic compounds · Anthocyanins

# Introduction

Phenolic compounds are secondary metabolites, which are involved in plant growth and plant defense against biotic and abiotic stresses (Pandey and Rizvi 2009). Therefore, all plant-rooted food materials always include various kinds of phenolic compounds at different amounts. In addition, phenolic compounds are partially responsible for the overall organoleptic properties (such as flavor, color, odor, and appearance) of plant foods. They are also important for human health. The phenolic compounds have effect on taste and odor formation

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and participate in color formation in plants. They also possess antimicrobial, antioxidant and enzyme inhibition activities. They can also be used as purity criteria in some food products (Saldamli 2007).

Cornelian cherry is grown notably in the Caucasus, and countries of the European continent such as Turkey, Romania, Bulgaria, and Italy (Dolejsi et al. 1991; Dinda et al. 2016). Cornelian cherry is a fruit which has beneficial health effects due to its high phenolic content. This fruit has also high anthocyanin pigment, so it has different color such as yellow, pink, or red (Hassanpour et al. 2011). Moldovan et al. (2016) showed that the dominant phenolic acids of the extract of cornelian cherry were quercetin, kaempferol, ellagic acid, epicatechin, pelargonidin, chlorogenic acid, and catechin. The fruits have been utilized in various European and Asian countries as medicine for ailments such as diarrhea, diabetes, gastrointestinal disorders, urinary infections, skin diseases, liver and kidney diseases, rheumatic, stomach ulcers, cancers, excessive sweating, menstrual bleeding, or cardiac problems (Zargari 1997; Genc and Ozhatay 2006; Celik et al. 2006; Zarei et al. 2014; Zlatkovic et al. 2014; Guler et al. 2015; Dinda et al. 2016). In literature, there are many studies about chemical and physical properties of cornelian cherry (Tural and Koca 2008; Moldovan et al. 2016; David et al. 2019; Dumitrașcu et al. 2019; Vakula et al. 2019).

Microwave extraction is a process that uses microwaves located between the radio-frequency and infrared in the electromagnetic spectrum, within the frequency band of 300 MHz to 300 GHz. Microwave heating has two distinct mechanisms namely, dipole reorientation and ionic polarization. Molecular collisions and heat generation occur when the dipoles attempt to realign themselves with the electric field during microwave application. In addition, the ions move accordingly to the electric field, causing ionic collisions and heating (Routray and Orsat 2012). Microwave extraction has been preferred by many researchers due to its numerous advantages such as lower extraction time, less solvent requirement, high yield efficiency, high quality of final product, and good reproducibility (Garofulic et al. 2013). On the other hand, the most important disadvantage of microwave is that it can cause degradation of extremely heat-sensitive compounds (Desai et al. 2010) due to increase in temperature especially at longer extraction times. It has been used to extract valuable compounds from purple corn cob, fig peel, lime peel waste, mango and grape seeds (Piyapanrungrueang et al. 2016; Backes et al. 2018; Rodsamran and Sothornvit 2019; Dorta et al. 2013a; Krishnaswamy et al. 2013), pectin from mango peel, jackfruit peel and eggplant (Matharu et al. 2016; Xu et al. 2018; Kazemi et al. 2019), prebiotic oligosaccharides from sweet potato (Guo et al. 2019), and soluble sugars from banana puree (Gomez et al. 2019).

Ohmic heating is also known as electro-conductive heating, electro-heating, direct heating, and Joule heating. Ohmic heating can be stated as a process in which alternating current is passed through a sample and the electrical resistance of the sample generates heat (Saldana et al. 2009; Varghese et al. 2012). Ohmic heating has led to high extraction yields in comparison to conventional thermal methods due to presence of electrical field, even under similar thermal conditions. The efficiency of ohmic heating depends on several factors such as electric field strength, electrical conductivity of food material, holding time, and equipment design (Varghese et al. 2012; İçier 2012; Aggarwal and Jain 2019). Recently, ohmic heating has been performed to extract valuable compounds from red grape pomace, bottle gourd, kohlrabi-turnip-potato-radish and chestnut juice (El-Darra et al. 2013b; Bhat et al. 2017; Farahnaky et al. 2018; Li et al. 2019), oil from Taiwanese citronella grass and microalgae (Gavahian et al. 2018; Sofi'i et al. 2019), and pectin from orange waste (Saberian et al. 2017). However, information regarding ohmic heated assisted extraction of phenolic compounds from cornelian cherry is lacking in literature.

To the best of our knowledge, previous studies did not evaluate the extraction of phenolic compounds from cornelian cherry using the assistance of ohmic heating system. Most studies have focused only on microwave extraction. In our study, our main goal was to increase the extraction efficiency of polyphenols with ohmic heating assistance. The extracts of cornelian cherry obtained by maceration, microwave, and ohmic heating assisted microwave extraction were compared with respect to (I) extraction yield; (II) physical (water-soluble solid matter and color) and chemical (total phenolic compounds, total monomeric anthocyanin content and antioxidant activity) properties; and (III) extraction model.

# **Materials and Methods**

### **Materials**

Fresh Cornelian cherry (Cornus mas) was obtained from a local producer in Turkey. The geographic coordinates of local producer are 40° 11' 44.12" N 29° 03' 36.47" E. The cornelian cherry was harvested in September 2016. Initially, the seeds of cornelian cherry were removed mechanically. Afterwards, the flesh with the skin were ground using a laboratory blender (Waring Products Division New Hartford, Conn, USA) for 60 s. The Cornelian cherry samples were sealed in polyethylene bags and stored at -18 °C until further use. The moisture content of the cornelian cherry was measured as  $70 \pm 2\%$  (wet basis) by infrared moisture analyzer (MA150, Sartorius, Germany) at 105 °C. Each experiment contained 6, 3, or 2 g (dry basis) of cornelian cherry samples at solid to solvent ratios of 1/5, 1/10, and 1/15 g/mL, respectively. All chemicals used in this research were of analytical grade and supplied by Merck Co. (Germany) and Sigma-Aldrich Co. (USA) unless otherwise mentioned.

## Methods

The cornelian cherry samples were extracted by different techniques as explained below. After the extraction process, the extracts were filtered through filter paper (Whatman No 4) for all of the extraction methods. Total phenolic compounds, water soluble solid matters, and color values were determined for all of the samples collected during extraction. Total monomeric anthocyanin content, antioxidant activity, and pH analysis were performed for the samples that exhibited the highest total phenolic content for each extraction method. Water was used as a solvent for the extractions and all experiments were conducted in duplicates.

### Maceration

Maceration was performed at 25 °C in a water bath (SBD-313, Simsek Laborteknik, Turkey) for 24 h (Xu et al. 2016). Three different solid to solvent ratios (1/5, 1/10, and 1/15 g/mL) were studied during maceration. Extracts were stored at -18



Fig. 1 Ohmic heating system and glass ohmic heating cell

°C until analyses were performed. This method was selected as the control group for microwave and ohmic heating assisted microwave extraction.

#### **Microwave Extraction**

Microwave extraction was performed using a domestic microwave oven (GMOM 25, General Electric, USA) equipped with a cooling system (condenser). The frequency of microwave oven is 2450 MHz. The internal dimensions of the microwave oven are  $30 \times 30 \times 20$  (w×1×h) cm. The samples were extracted at different solid to solvent ratios (1/5, 1/10, and 1/ 15 g/mL), microwave power (180 and 360 W), and extraction times (10, 60, 120, 150, and 180 min.). The sample and solvent were mixed inside the extraction flask that had a capacity of 500 ml and placed in the middle of the microwave oven. The amount of microwave energy per gram of sample was 6 and 12 W/g for 180 and 360 W, respectively. The extracts were stored at – 18 °C until further use.

## **Ohmic Heating Assisted Microwave Extraction**

Ohmic heating system (CLOH-1000, CLS, Turkey), which was designed by our research group, was used for ohmic heating assisted microwave extraction (OH-ME) experiments (Fig. 1). The frequency of ohmic heating system was 50 Hz. The diameter and distance between the two electrodes were 5 and 15 cm, respectively. A built-in temperature controller maintained the set temperature within  $\pm 1$  °C during ohmic heating. The temperature of the samples during ohmic heating was measured by two thermocouples. The thermocouples were mounted inside the ohmic heating cell and the temperature measurements were recorded continuously. Recording of the thermocouples showed that the temperature was same at both points and the temperature was uniform along the sample. Heating up time was approximately 1 min and it was not included in the holding time. The electric field strengths and temperature were recorded every second using a data logger.

Ohmic heating applications were performed at electric field strengths of 20, 30, and 40 V/cm. The solid to solvent ratio and temperature of the cell was kept constant as 1/15 g/mL and 60 °C, respectively. The holding times for ohmic heating were 1, 10, and 20 min. The samples were then immediately removed from the cell and subjected to microwave extraction at 3 different extraction times (10, 60, and 120 min). The extracts were stored at -18 °C until further use.

#### **Determination of Water-Soluble Solid Matter**

Water-soluble solid matters were determined by using a refractometer (NR, Atago, Japan) and the results were expressed as percentage (Cemeroğlu 2010). The calculation was done by using Eq. 1. In this equation,  $B_x$  is the reading from refractometer, V is volume of sample (mL), and M is weight of sample (g).

Water-soluble solid matter (%) = 
$$\frac{B_x V}{M}$$
 (1)

## **Determination of Color Values**

The color of the extracts was measured using Chroma Meter (C-400, Konica Minolta, Japan) according to CIE L\* a\* b\* system. Prior to the analysis, the Chroma Meter was calibrated with a white ceramic standards plate. The analysis was



Fig. 2 The variation of TPC and WSSM of the extracts obtained by M at different solid to solvent ratios

Table 1Color values of theextracts obtained by M atdifferent solid to solvent ratios

L*	a*	b*
$25.76^{b} \pm 0.08$	$12.59^{a} \pm 0.17$	$11.47^{b} \pm 0.11$
$27.88^{a}\pm0.54$	$11.50^{ab}\pm0.34$	$13.44^a\pm0.21$
$29.21^{a}\pm0.61$	$10.33^b\pm0.77$	$13.68^a\pm0.25$
	L* $25.76^{b} \pm 0.08$ $27.88^{a} \pm 0.54$ $29.21^{a} \pm 0.61$	L*         a* $25.76^b \pm 0.08$ $12.59^a \pm 0.17$ $27.88^a \pm 0.54$ $11.50^{ab} \pm 0.34$ $29.21^a \pm 0.61$ $10.33^b \pm 0.77$

conducted in three repetitions for each extract. L\* is lightness (100) or darkness (0), a\* indicates red (+) or green (-) and b\* represents chromaticity yellow (+) or blue (-). Based on these parameters, total color difference ( $\Delta E$ ) was calculated using the Eq. 2. In this equation,  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  are the change of L\*, a\* and b\* values at time t with respect to values obtained by maceration.

$$\Delta E = \sqrt{\left(\Delta L^*\right)^2 + \left(\Delta a^*\right)^2 + \left(\Delta b^*\right)^2} \tag{2}$$

#### **Determination of Total Phenolic Compounds Content**

The total phenolic content was determined by following the Folin-Ciocalteu method (Slinkard and Singleton 1977). This method was modified according to Bobinaite et al. (2012). Briefly, 1 mL of extract (1 mg/mL) mixed thoroughly with 5 mL of Folin–Ciocalteu reagent (1/10 v/v) for 3 min, followed by the addition of 4 mL of 7.5% (w/v) sodium carbonate. The mixture was allowed to stand for a further 60 min in the dark at the room temperature. The absorbance of the mixture was measured at 765 nm against an appropriate blank using a spectrophotometer (Lambda35, Perkin Elmer, USA). Gallic acid was used for the standard curve (y = 0.0011x + 0.0051 and R<sup>2</sup> = 0.9990) and total phenolic compounds content (TPC) was recorded as milligram gallic acid equivalent per gram of sample (mg GAE/g).

# Determination of Total Monomeric Anthocyanin Content

Total monomeric anthocyanin content was determined following the pH differential method (Giusti and Wrolstad 2001) with minor modifications (Lee et al. 2005). The principle of the method is to transform the structure of anthocyanin manifested by different colorations in two distinct pH values (1.0 and 4.5). Two aliquots of the extract (500  $\mu$ L) were diluted to a final volume of 4 mL with potassium chloride buffer (0.025 M, pH 1.0) and sodium acetate buffer (0.4 M, pH 4.5). The dilutions are left to equilibrate for 15 min, then, the absorbance of each dilution was measured at 510 and 700 nm (Lambda35, Perkin Elmer, USA) against a blank of water. The sample absorbance (A) is equal to (Eq. 3):

Absorbance 
$$(A) = (A_{510} - A_{700}) \text{ pH1.0} - (A_{510} - A_{700}) \text{ pH4.5} (3)$$

Total monomeric anthocyanins were calculated by the following formula (Eq. 4):

TMAC (mg cya-3-glu/g) = 
$$\frac{A \times MW \times DF \times 10^3}{\varepsilon \times D}$$
 (4)

where A is the absorbance value which was calculated from Eq. 3, DF is the dilution factor,  $10^3$  is the conversion factor of g to mg, and D is the cell path length (1 cm). Total monomeric anthocyanin content (TMAC) was expressed as mg cyanidin-3-glucoside (molecular weight (MW) = 449.2 g/mol and a molar extinction coefficient  $\varepsilon = 26900$  L/cm.mol) per g of sample.

Μ ME OH-ME  $5.5 \pm 0.15^{\circ}$  $27.7 \pm 0.93^{b}$  $29.4\pm0.67^a$ TPC (mg GAE/g dry matter)  $0.65\pm0.08^{a}$ TMAC (mg cya-3-glu/g dry matter)  $0.36 \pm 0.01^{b}$  $0.32 \pm 0.02^{b}$  $4.09 \pm 0.04^{b}$  $4.51\pm0.04^a$  $4.13 \pm 0.01^{b}$ AA (mg TE/g dry matter)  $\phantom{-}3.05\pm0.06^a$  $2.29\pm0.02^{b}$  $\phantom{-}3.03\pm0.04^a$ pН WSSM (%)  $65.6 \pm 2.26^{b}$  $78.6 \pm 2.27^{a}$  $70.4 \pm 0.01^{b}$ L\*  $26.61\pm0.87^{ab}$  $25.67 \pm 0.17^{b}$  $29.21 \pm 0.62^{a}$ a\*  $10.33 \pm 0.77^{a}$  $9.31\pm0.86^a$  $9.83\pm0.64^a$ h\*  $2.43 \pm 0.84^{c}$  $5.17 \pm 0.27^{b}$  $13.68 \pm 0.25^{a}$ 

Different letters (a, b and c) in same row are significantly different (p < 0.05)

M: 1/15 g/mL, 25 °C and 24 h

ME: 1/15 g/mL, 180 W and 120 min

OH-ME: 1/15 g/mL, 60 °C, 20 V/cm, 10 min. HT, 180 W and 60 min

**Table 2**The results of analysis of<br/>extracts obtained by M, ME, and<br/>OH-ME at optimum conditions



Fig. 3 Change of TPC of extracts obtained by ME with respect to extraction time at different microwave powers (180 and 360 W) and solid to solvent ratios:  $\mathbf{a} \ 1/5 \ g/mL$ ,  $\mathbf{b} \ 1/10 \ g/mL$ , and  $\mathbf{c} \ 1/15 \ g/mL$ 

### **Determination of Antioxidant Activity**

The scavenging effects of the samples on DPPH (1,1diphenyl-2-picrylhydrazyl) radical were measured according to Bao et al. (2005), with some modifications. 0.1 mL of the

**Fig. 4** WSSM of the extracts obtained by ME at different process conditions

diluted extract was added to 3.9 mL of DPPH solution (6  $\times$  10<sup>-5</sup> M). The mixture was shaken vigorously and kept in dark for 30 min. The absorbance was measured at 515 nm using the spectrophotometer and the results were expressed in milligrams Trolox equivalents per gram of dry matter (mg TE/g).

#### **Kinetic Modelling**

Peleg's kinetic model (Peleg 1988) was fitted to the experimental data obtained from extraction of phenolics. Nonlinear regression was carried out using SigmaPlot 12.0 (Systat Software Inc., USA) program.

$$TPC(t) = TPC_0 + \frac{t}{k_1 + k_2 t}$$
(5)

where TPC(t) is the concentration of total phenolic compounds at time t (mg GAE/g), t the extraction time (min), TPC<sub>0</sub> is the initial concentration of phenolic compounds at time t = 0 (mg GAE/g),  $k_1$  is Peleg's rate constant (min g/ mg GAE), and  $k_2$  is Peleg's capacity constant (g/mg GAE) (El-Darra et al. 2013a).

# **Statistical Analysis**

Data were analyzed by the program MINITAB 16.1.1.0 (Minitab, State Collage, PA, USA) by one-way analysis of variance (ANOVA) followed by Tukey's test, considering p < 0.05 to be statistically significant.

# **Results and Discussions**

## Maceration

The variation of total phenolic content with solid to solvent ratio is given in Fig. 2. The minimum value of TPC was found as



Fig. 5 The variation of the WSSM and TPC of extracts with respect to solid to solvent ratios and microwave powers during ME



3.9 mg GAE/g dry matter at 1/5 g/mL of solid to solvent ratio. It increased approximately 41% to 5.5 mg GAE/g dry matter, when the amount of solvent used for the extraction was tripled. This finding indicated that the amount of solvent was not enough to extract all of the total phenolic compounds at solid to solvent ratio of 1/5 g/mL. In addition, temperature might have played a critical role in extracting phenolic compounds during maceration. The temperature was kept constant (25 °C) during maceration and the low extraction temperature together with the reduced solvent concentration might have resulted in limited TPC extraction. In addition, the increase in the amount of solvent caused an increase in concentration gradient, and hence, more phenolic compounds were extracted. Similar results were reported for sour cherry pomace (Simsek et al. 2012), caper (Durmaz et al. 2015), and cranberry bush (Alifakı 2019). Popovic et al. (2012) extracted total phenolic compounds from different genotypes of cornelian cherry (1/15 g/mL) at 10 °C for 70 h and using 80% methanol-water mixture as a solvent. It was reported that the amount of total phenolic compounds was determined in the range of 4.94-7.04 mg GAE/g dry matter. Similar results for certain genotypes were obtained with comparison to our study, despite using a different solvent. Moldovan et al. (2016) reported that the total phenolic content of the cornelian cherry (1/10 g/mL) fruits was 16.3 mg GAE/g dry matter by traditional extraction using acetone as a solvent. It was thought that the difference in TPC determined in that study and in our study could be dependent on solvent variation.

The change of water-soluble solid matter (WSSM) with respect to solid to solvent ratio is also depicted in Fig. 2. WSSM was found to be in the range of 61–65% and there were no differences between solid to solvent ratios (p > 0.05). Although the effect of the solid to solvent ratio on WSSM was not observed in our study, there are studies in literature that have reported its impact. For example, grape pomace was extracted (Pinelo et al. 2005) at different solvent to solid ratios (1, 3, and 5 mL/g). It was found that WSSM was increased with an increase in the amount of solvent. Similarly, Pavela (2017) has also reported that the extracts of *Saponaria*  *officinalis* had higher WSSM contents when more solvent per gram of feedstock was used. The discrepancy may be due to differences in solvents and material types, as well as extraction conditions. It may be concluded that the amount of solvent at 1/5 (g/mL) solid to solvent ratio was sufficient in our case to solubilize all of the water-soluble materials, and hence, an increase in the solvent did not have a significant impact on WSSM.

The variation of L\*, a\*, and b\* values of extracts with solid to solvent ratios was given in Table 1. According to Table 1, the maximum L\* and minimum a\* values were found as 29.2 and 10.3 at solid to solvent ratio of 1/15 g/mL, respectively. The increase in the amount of solvent caused the extract color to lighten (increase in L\* value). When b\* values were examined, the lowest value was found as 11.5 at 1/5 g/mL. This finding may suggest that the compounds that provide the yellow color (such as flavonols and carotenoids) might have been extracted more as the amount of solvent per grams of sample was increased.

The total monomeric anthocyanin content (TMAC), antioxidant activity (AA), and pH value of the samples, extracted at which TPC was maximum (1/15 g/mL), were also determined (Table 2). The TMAC of the extract was found as 0.4 mg cya-3-glu/g dry matter. Our result aligns well with the literature. The TMAC of dried cornelian cherries (10 different genotypes) were reported to be in the range of 0.05– 3.03 mg cya-3-glu/g dry matter by Popovic et al. (2012). The antioxidant activity (AA) and pH value of the corresponding extract was determined as 4.0 mg TE/g dry matter and 3.05, respectively. The pH values of cornelian cherry were reported to be in the range of 3.11–3.53 by Tural and Koca (2008), 2.92-3.37 by Yilmaz et al. (2009) and 2.7-3.0 by Gunduz et al. (2013).

### **Microwave Extraction**

The change of total phenolic compounds at different microwave powers (180 and 360 W), and solid to solvent ratios (1/5, **Table 3** Color values of theextracts obtained by ME atdifferent process conditions

1/10, 1/15 g/mL) with respect to extraction time were shown in Fig. 3a–c. The final temperatures of the extracts were measured as  $82 \pm 2$  °C after microwave heating. Microwave power, extraction time, and solid to solvent ratio had statistically significant impact on TPC of the extracts. Regardless of the solid to solvent ratio, TPC was increased for the first 120 min and remained relatively constant afterwards. This finding has shown that the extraction has reached equilibrium at around 120 min. Moreover, increasing the amount of solvent per unit sample has enhanced the extraction of TPC. High amount of solvent has accelerated mass transfer and subsequently has increased the extraction efficiency due to concentration gradient increase. The highest TPC was found as 27.7 mg GAE/g dry matter at microwave power of 180 W, solid to solvent ratio of 1/15 g/mL, and extraction time of 120 min. In literature, TPC extraction from cornelian cherry by using microwave technology has not been studied. However, when ultrasound extraction was employed, TPC of the extracts was found in the range of 12.22–21.63 mg GAE/g dry matter (Vakula et al. 2019). Although ethanol was used as a solvent in this study, the value of TPC was found lower compared to our study. This finding showed that microwave extraction had

Microwave power	Extraction time (min)	Solid to solvent ratios (g/mL)			
		1/5	1/10	1/15	
		L*			
180 W	10	$24.38\pm1.19^{b\mathrm{A}}$	$24.05\pm1.60^{cA}$	$24.46\pm1.22^{bA}$	
	60	$26.10\pm0.89^{bA}$	$25.21\pm1.23^{bcA}$	$25.14 \pm 1.26^{ab}$	
	120	$28.27\pm0.88^{abA}$	$27.08\pm0.92^{abcA}$	$26.61 \pm 1.29^{ab}$	
	150	$31.38\pm0.04^{aA}$	$28.98 \pm 1.24^{abAB}$	$27.55\pm0.70^{abl}$	
	180	$31.73\pm2.10^{aA}$	$30.05\pm0.14^{aA}$	$28.95\pm0.51^{aA}$	
360 W	10	$24.25\pm0.99^{bA}$	$24.57\pm0.98^{cA}$	$24.81 \pm 1.51^{bA}$	
	60	$26.10\pm1.32^{abA}$	$25.46\pm1.24^{bcA}$	$25.13\pm1.16^{bA}$	
	120	$28.49 \pm 1.30^{abA}$	$28.06\pm0.52^{abcA}$	$27.91\pm0.68^{ab}$	
	150	$30.24\pm1.59^{aA}$	$29.30\pm0.81^{abA}$	$28.08 \pm 1.13^{ab}$	
	180	$31.37\pm1.99^{aA}$	$29.79\pm1.45^{aA}$	$29.64\pm1.62^{aA}$	
		a*			
180 W	10	$12.58\pm1.77^{aA}$	$10.11\pm1.61^{aA}$	$10.50\pm0.75^{aA}$	
100 11	60	$13.29\pm0.77^{aA}$	$10.59\pm0.88^{aA}$	$9.83\pm0.89^{aA}$	
	120	$12.96\pm0.48^{aA}$	$10.14\pm0.26^{aB}$	$9.31\pm0.86^{aB}$	
	150	$14.49\pm0.92^{aA}$	$9.58\pm0.41^{aB}$	$8.48\pm0.39^{aB}$	
	180	$12.86\pm0.53^{aA}$	$9.87\pm0.25^{aB}$	$8.30\pm0.53^{aB}$	
360 W	10	$11.82\pm0.72^{aA}$	$10.64\pm0.82^{aA}$	$10.45\pm0.76^{aA}$	
	60	$12.65\pm1.18^{aA}$	$10.51\pm0.86^{aA}$	$10.17 \pm 0.92^{ab}$	
	120	$12.73\pm1.13^{aA}$	$9.99\pm0.08^{aAB}$	$8.36\pm0.25^{abcE}$	
	150	$11.97\pm1.80^{aA}$	$9.08\pm0.02^{aA}$	$8.09\pm0.15^{bcA}$	
	180	$12.09\pm1.38^{aA}$	$8.71\pm0.17^{aB}$	$7.30\pm0.13^{cB}$	
		b*			
180 W	10	$7.73 \pm 1.07^{aA}$	$7.04\pm2.44^{aA}$	$7.99 \pm 1.73^{aA}$	
	60	$4.77\pm0.19^{aA}$	$4.01\pm0.48^{abA}$	$4.35\pm1.08^{abA}$	
	120	$4.11\pm0.10^{aA}$	$2.81\pm0.39^{abA}$	$2.43\pm0.84^{b\rm A}$	
	150	$3.59\pm1.87^{aA}$	$1.92\pm0.09^{bA}$	$2.00\pm1.33^{b\rm A}$	
	180	$4.51\pm1.05^{aA}$	$2.04\pm0.04^{bA}$	$2.11\pm0.54^{b\rm A}$	
360 W	10	$7.23\pm0.36^{aA}$	$7.13\pm1.29^{aA}$	$7.30\pm1.45^{a\mathrm{A}}$	
	60	$5.07\pm0.58^{abA}$	$4.07\pm0.21^{bA}$	$5.09 \pm 1.77^{abA}$	
	120	$4.28\pm0.48^{bA}$	$2.58\pm0.59^{bA}$	$1.88\pm0.80^{b\rm A}$	
	150	$4.10\pm0.23^{b\rm A}$	$1.96\pm0.42^{b\mathrm{A}}$	$2.00\pm0.92^{b\mathrm{A}}$	
	180	$4.50\pm0.95^{b\mathrm{A}}$	$1.99\pm0.83^{bAB}$	$1.01\pm0.22^{bB}$	

Different letters (a, b and c) in same column are significantly different (p < 0.05)

Different letters (A and B) in same row are significantly different (p < 0.05)

**Fig. 6** The variation of  $\Delta E$  of extracts obtained by ME with different process conditions



high efficiency in the extraction process, even if, the solvent is water. Stankovic et al. (2014) extracted total phenolic compounds from cornelian cherry using Soxhlet apparatus with different solvents (methanol, water, ethyl acetate, acetone, and petroleum ether). The values of TPC were measured as 31.36, 12.77, 179.05, 55.38, and 27.14 mg GAE/g extract for methanol, water, ethyl acetate, acetone, and petroleum ether, respectively. Our result of TPC was found to be higher compared to this study considering that the solvent is water.

Irrespective of the solid to solvent ratio, extracts obtained using low microwave power provided slightly more efficient (7% on average) TPC extraction (p < 0.05). High microwave power causes rapid increase in sample temperature which may be destructing some of the phenolic compounds in cornelian cherry. The reduction in TPC of the extracts with an increase in microwave power was also reported by other researchers (Song et al. 2011; Teng et al. 2013; Nazeri and Zain 2018).

The TPC of the microwave extracts were found to be approximately 5 times higher compared to that of M. Moreover, the extraction time decreased from 1440 to 120 min with ME. Microwave energy has been found to be very effective to extract TPC from cornelian cherry. When the plant cell is subjected to microwaves, water vapor is formed inside the cell. This causes the cell to swell and thus creates a great pressure on the cell wall. The pressure builds up rapidly, disintegrates the cell wall (Mandal et al. 2007), and enhances the extraction efficiency. In addition, the dipole molecules align approximately  $4.9 \times 10^9$  times per second at 2450 MHz. Consequently, heat is generated due to friction (Lopez-Avila 2000). This phenomenon contributes to achieving higher extraction efficiencies with microwave. In addition, the extraction temperature might have played an important role in facilitating the extraction of TPC. The maceration temperature was 25 °C, whereas the temperature of the extract at optimum microwave extraction (ME) condition was  $82 \pm 2$  °C.

WSSM of the extracts obtained by ME was in the range of 61.8-81.8% (Fig. 4). At solid to solvent ratio of 1/5 g/mL, the microwave power did not have a statistically significant effect on WSSM. The extraction time, on the other hand, had a positive impact on WSSM (p < 0.05). Both microwave power and extraction time had no statistically significant effect on WSSM for solid to solvent ratios of 1/10 and 1/15 g/mL. Increment in the amount of solvent has not affected the WSSM. In other words, all solids that could be dissolved were solved in the ratio of 1/5 solid to solvent. The increase in the amount of solvent did not cause any change in the value of WSSM. Moreover, the insignificant effect of extraction time could be explained by the capture of all the soluble solid matter that is available at the beginning of the extraction. The variation of the WSSM and TPC of extracts with respect to solid to solvent ratios and microwave powers during ME were given in Fig. 5. A positive correlation (Pearson correlation coefficient range 0.801-0.987) between the WSSM and the TPC was detected for all solid to solvent ratios. Higher WSSM values were acquired by ME compared to M (approximately 21%), which could be mainly due to high temperature observed during ME. Lee et al. (2006) have observed this effect during the extraction of banana juice. WSSM was increased by approximately 12% when the temperature was increased from 35 to 95 °C. Stevenson et al. (2008) have also reported 4-fold rise in WSSM when the temperature was tripled during ME of oat bran concentrate. The obtained high values of TPC by microwave method are also relevant to the values of WSSM, because most of the phenolic compounds are in water-soluble form (Cassidy 2008).

The variation of L\*, a\*, and b\* values of extracts obtained by ME was shown in Table 3. L\* values increased significantly with increasing extraction time at constant microwave power and constant solid to solvent ratio. The effect of extraction time on the a\* value was only seen at solid to solvent ratio of



**Fig. 7** TPC of extracts obtained by OH-ME at different electrical field strength (20, 30, 40 V/cm), extraction time (10, 60, and 120 min) and holding time (HT) values: **a** 1 min, **b** 10 min, and **c** 20 min

1/15 g/mL and microwave power of 360 W. The decrement of a\* values during extraction suggested that some anthocyanins which are responsible for the redness criterion was degraded with the effect of time and temperature in the presence of more solvent at the solid to solvent ratio of 1/15 g/mL. These results of a\* values supported the increase in L\* value. The b\* values were significantly reduced with increasing extraction time at constant solid to solvent ratios (180 W, except for 1/5 g/mL solid to solvent ratio). With the increase of extraction time, the duration of temperature exposure of extracts increased and the compounds causing yellow color were deteriorated. The L\*, a\*, and b\* values were found as 26.6, 9.3, and 2.4 at optimum conditions (180 W, 120 min, and 1/15 g/mL), respectively. It

was observed that L\* value of the extracts obtained by ME was lower than that of obtained by M. This finding indicated that microwave extracts were darker in color. This result aligns well with the TPC results. Since more phenolic compounds were extracted with ME, a darker solution was expected. a\* values of ME extracts, on the other hand, were similar to what was obtained by M. Based on these findings, it could be concluded that the phenolic compounds extracted with ME were mostly colorless.

The change of total color differences of extracts which were obtained by microwave extraction versus maceration was given in Fig. 6. It was observed that the  $\Delta E$  values increased with increasing extraction time at each microwave powers and solid to solvent ratios. Differences can be expected in  $\Delta E$  values since higher temperatures were achieved by microwave compared to maceration. The increase in total color difference during the extraction time can be explained by the sharp decrease in a\* and b\* values for ME. Since these values of extracts obtained by ME were lower compared to maceration, the total color difference was found high between the two methods.



**Fig. 8** WSSM of extracts obtained by OH-ME at different electrical field strength (20, 30, 40 V/cm), extraction time (10, 60, and 120 min), and holding time (HT) values: **a** 1 min, **b** 10 min, and **c** 20 min



Fig. 9 The variation of the WSSM and TPC of extracts with respect to holding times (1, 10, 20 min) and electrical field strengths during OH-ME: a 20 V/cm, b 30 V/cm, and c 40 V/cm

The total monomeric anthocyanin concentration, antioxidant activity, and pH value of the sample, extracted at which TPC was maximum (at 180 W, 120 min, and 1/15 g/mL), were determined for microwave extraction (Table 2). The TMAC of the extract was found as 0.3 mg cya-3-glu/g dry matter. Although TPC of microwave extracts were very high, there were no significant differences between the methods ME and M in terms of TMAC. This could be due to degradation of anthocyanins at high extraction temperatures when they are exposed to microwaves for a long period of time. Similar findings with microwave extraction were reported in literature. Vazquez-Espinosa et al. (2019) extracted anthocyanin from maqui berry using ME. It was claimed that ME was not effective in anthocyanin extraction due to degradation of anthocyanins as a result of increase in temperature and pressure during ME. The AA of extract was found as 4.5 mg TE/g dry matter. This value was found to be 12.5% higher than that of M (p < 0.05). Similarly, there is a study reporting that extracts obtained by ME method showed higher antioxidant activity (Dorta et al. 2013b). In our study, even though microwave extracts' TPC was 5 times higher, the antioxidant content did not increase to the same extent. This indicated that not all of the phenolic compounds that are extracted during ME presented antioxidant activity. The pH value of the extract was found as 2.3 for microwave extraction at optimum conditions. This value was found to be lower compared to that of obtained by M method. With increasing temperature, more hydrogen ions and hydroxide ions are formed from water molecules and this would increase the value of Kw (dissociation constant) (Cummings et al. 2003). Therefore, the pH decreases as the temperature increases. Similarly, Stevenson et al. (2008) reported that microwave treatment applied to extract phenolic compounds from oat bran at different temperatures (50–150 °C) has caused a decrease in the pH value.

#### **Ohmic Heating Assisted Microwave Extraction**

The effects of electrical field strengths, holding time, and extraction time on TPC of OH-ME extracts were depicted in Fig. 7a-c. The TPC have increased significantly with increasing extraction time for all of the holding times (p < 0.05). However, the change in electrical field strengths did not have a statistically significant impact on TPC. Loypimai et al. (2015) have also studied ohmic heating assisted extraction of anthocyanins from black rice bran. Parallel to our finding, they have reported field strengths had no effect on the extraction efficiency of valuable compounds. In addition, in our study, no significant effect of holding time was observed in general. Likewise, Pare et al. (2014) used ohmic heating at different voltages (600-900 V), temperatures (70-90 °C), and holding time (0-10 min) for oil extraction from soy bean. It was observed that the effect of holding time was not significant on free fatty acid (% oleic acid) of extracted oil.

The maximum TPC in OH-ME extracts was 29.4 mg GAE/ g dry matter at electrical field strength of 20 V/cm, holding time of 10 min, and extraction time of 60 min (the microwave extraction after ohmic heating was performed at constant solid to solvent ratio (1/15 g/mL) and microwave power (180 W)). The value was approximately 5.4 and 1.1-fold higher than that of M and ME, respectively. Even though the difference in TPC values of OH-ME and ME appears to be small, the extraction time was reduced (approximately 50%) remarkably with ohmic heating. This might be due to electroporation. Electroporation is a process which damages the cell membrane by electrical pulses. The cell membrane is fragmented by the effect of electrical resistance and its temporary permeability increases (Loypimai et al. 2015). This permeability allows the passage of molecules between matrices by diffusion (Gehl 2003). Hence, OH assistance during extraction significantly reduces the extraction time. El-Darra et al. (2013b) performed polyphenol extraction by pulsed ohmic heating from red grape pomace. As a result, it was found that polyphenols were in the range of 44-62 mg GAE/g dry matter in 60 min. These results were found to be higher compared to **Table 4**Color values of theextracts obtained by OH-ME atdifferent process conditions

Electrical field strength (V/cm)	Extraction time (min)	1 min HT	10 min HT	20 min HT	
		L*			
20	10	$24.75\pm0.40^{b\mathrm{A}}$	$24.45\pm0.19^{bA}$	$24.83\pm0.47^{bA}$	
20	60	$25.36\pm0.46^{abA}$	$25.67\pm0.36^{abA}$	$25.37\pm0.41^{bA}$	
20	120	$26.81\pm0.25^{aA}$	$26.27\pm0.44^{aA}$	$26.13\pm0.28^{aA}$	
30	10	$24.82\pm0.08^{cA}$	$24.44\pm0.18^{bA}$	$24.17\pm0.43^{bA}$	
30	60	$25.84\pm0.09^{bA}$	$24.78\pm0.21^{bB}$	$25.10\pm0.22^{abAB}$	
30	120	$26.45\pm0.03^{aA}$	$26.72\pm0.01^{aA}$	$25.74\pm0.12^{aB}$	
40	10	$24.71\pm0.25^{bA}$	$24.52\pm0.11^{b\mathrm{A}}$	$24.36\pm0.28^{bA}$	
40	60	$25.11\pm0.14^{b\mathrm{A}}$	$25.09\pm0.16^{abA}$	$25.35\pm0.27^{aA}$	
40	120	$26.78\pm0.29^{aA}$	$26.01\pm0.38^{aA}$	$26.34\pm0.84^{aA}$	
		a*			
20	10	$10.56\pm0.45^{aA}$	$10.35\pm0.31^{a\mathrm{A}}$	$10.70\pm0.30^{aA}$	
20	60	$10.44\pm0.11^{abA}$	$9.83\pm0.56^{aA}$	$9.90\pm0.14^{b\rm A}$	
20	120	$9.45\pm0.24^{b\mathrm{A}}$	$9.19\pm0.30^{aA}$	$9.44\pm0.05^{bA}$	
30	10	$10.16\pm0.09^{aA}$	$10.39\pm0.19^{a\mathrm{A}}$	$10.33\pm0.27^{aA}$	
30	60	$10.34\pm0.31^{aA}$	$10.23\pm0.09^{aA}$	$9.88\pm0.30^{\mathrm{aA}}$	
30	120	$9.09\pm0.08^{bA}$	$9.35\pm0.10^{b\rm A}$	$8.94\pm0.21^{bA}$	
40	10	$10.88\pm0.20^{a\mathrm{A}}$	$10.51 \pm 0.51^{aA}$	$10.26\pm0.15^{\mathrm{aA}}$	
40	60	$10.39\pm0.09^{aA}$	$9.91\pm0.14^{aA}$	$9.77\pm0.34^{abA}$	
40	120	$9.19\pm0.20^{b\mathrm{A}}$	$9.30\pm0.23^{aA}$	$8.87\pm0.20^{b\mathrm{A}}$	
		b*			
20	10	$8.24\pm0.40^{aA}$	$8.26\pm0.48^{aA}$	$8.42\pm0.44^{aA}$	
20	60	$5.02\pm0.16^{b\mathrm{A}}$	$5.17\pm0.23^{bA}$	$4.73\pm0.22^{bA}$	
20	120	$3.09\pm0.26^{cA}$	$3.17\pm0.38^{cA}$	$2.88\pm0.10^{cA}$	
30	10	$8.39\pm0.34^{aA}$	$8.36\pm0.39^{aA}$	$8.09\pm0.52^{aA}$	
30	60	$4.82\pm0.10^{b\mathrm{A}}$	$5.05\pm0.08^{bA}$	$4.90\pm0.17^{b\mathrm{A}}$	
30	120	$3.03\pm0.25^{cA}$	$2.88\pm0.13^{cA}$	$3.68\pm0.13^{b\rm A}$	
40	10	$8.44\pm0.24^{aA}$	$7.58\pm1.39^{aA}$	$7.86\pm0.11^{a\mathrm{A}}$	
40	60	$5.09\pm0.40^{b\rm A}$	$4.80\pm0.10^{b\rm A}$	$4.71\pm0.77^{bA}$	
40	120	$3.13\pm0.67^{b\rm A}$	$3.68\pm0.23^{b\rm A}$	$2.88\pm0.44^{b\rm A}$	

Different letters (a, b and c) in same column are significantly different (p < 0.05)

Different letters (A and B) in same row are significantly different (p < 0.05)

our study. This is mainly due to differences in feedstock, solvent, and process parameters.

A statistically significant (p < 0.05) increase in WSSM was also observed with an increase in extraction time (Fig. 8a–c). However, similar to TPC findings, electrical field strength and holding time had no influence on WSSM values, too. The average WSSM values decreased in the order of ME>OH-ME>M. This might be attributed to the temperature rise in microwave extraction. The effect of temperature on WSSM was also reported by Mannozzi et al. (2018). Ohmic heating pretreatment was applied at different temperatures (40–80 °C) to obtain puree from carrots and apples. WSSM value was increased along with the temperature rise for both of the food materials. The variation of the WSSM and TPC of extracts with respect to electrical field strengths and holding times during OH-ME was given in Fig. 9a–c. For all electrical field strengths and holding times, positive correlations were also observed between the WSSM and the TPC values with Pearson correlation coefficient ranging between 0.937 and 0.999.

The color values were demonstrated in Table 4 for different conditions. L\* value increased significantly with the increase of the extraction time at constant electric field strength in all holding times (p < 0.05). a\* value decreased with increasing extraction time (except samples treated with 20 and 40 V/cm-10 min HT). This finding showed that both temperature and extraction time interaction cause degradation of anthocyanins. It was observed that the b\* value decreased with the increase of extraction time in all electric field strength and holding times. Likewise, it was thought that the compounds that cause



**Fig. 10**  $\Delta E$  of extracts obtained by OH-ME at different electrical field strength (20, 30, 40 V/cm), extraction time (10, 60, and 120 min), and holding time (HT) values: **a** 1 min, **b** 10 min, and **c** 20 min

yellowness were degraded. The L\*, a\*, and b\* values were found as 25.7, 9.8, and 5.2 at optimum conditions (1/15 g/mL, 20 V/cm, 10 min HT-180 W and 60 min), respectively. It was observed that the L\* value of extract obtained by OH-ME was lower than that of obtained by M, but similar to the one obtained by ME. Moreover, the a\* value of extract obtained by OH-ME was similar to those of obtained by M and ME. In addition, the b\* value was found to be lower than M, but higher than ME.

**Table 6**Coefficients of determination and constants for Peleg Model(ME 1/15 g/mL, 180 W, and 120 min; OH-ME 1/15 g/mL, 60 °C, 20V/cm, 10 min HT, 180 W, and 60 min)

Extraction methods	k <sub>1</sub>	k <sub>2</sub>	$\mathbb{R}^2$
ME	0.2809	0.03251	0.97
OH-ME	0.3836	0.02841	0.99

The  $\Delta E$  values calculated from the difference of color values of extracts obtained by OH-ME and color values obtained in M method were given in Fig. 10a–c.  $\Delta E$  values increased with an increase in extraction time at each electrical field strength and holding time. However, electrical field strengths and holding time had no significant effect on  $\Delta E$  (p < 0.05). It was observed that the values of  $\Delta E$  were similar to  $\Delta E$  values obtained by ME.

The total monomeric anthocyanin concentration, antioxidant activity, pH value, and color values of the sample, extracted at which TPC was maximum (at 20 V/cm, 10 min HT, and 60 min), were determined for ohmic heating assisted microwave extraction (Table 2). The TMAC was determined as 0.65 mg cya-3-glu/g dry matter. This value was approximately 45% and 51% higher than that of M and ME, respectively. As discussed above, the temperature had adverse effects on anthocyanins after a certain period of extraction time (Liu et al. 2018). However, it is worth noting that anthocyanin degradation was prevented by OH-ME due to decrease in extraction time. The AA of the OH-ME extract was found as 4.1 mg TE/ g dry which was similar to M, but lower than the value obtained by ME. The WSSM was found to be higher in the extracts obtained by ME. The pH value of extract was determined as 3.0 for OH-ME. This value was found to be similar to M, but it was significantly higher compared to ME. Even though the temperature was also high in this method (OH-ME) like ME, it was thought that it prevented the reduction of pH due to the short extraction time. Extracts obtained by M had high values of L\* and b\*. However, there were no differences between a\* values of all methods.

Table 5 Experimental and predicted TPC values (mg GAE/g dry matter) of different extraction methods at optimum conditions (ME 1/15 g/mL, 180 W, and 120 min; OH-ME 1/15 g/mL, 60 °C, 20 V/cm, 10 min HT, 180 W, and 60 min)

Extraction time (min)	ME			OH-ME		
	Experimental	Predicted	Error*	Experimental	Predicted	Error*
10	16.8	16.5	0.3	14.8	15.0	- 0.2
60	26.0	26.9	-0.8	29.4	28.8	0.6
120	27.7	28.7	- 1.0	31.1	31.6	- 0.5
150	30.4	29.1	1.3			
180	29.6	29.3	0.3			

\*Error is the difference between the experimental and predicted TPC yields.

### **Kinetic Modelling**

The experimental and predicted values of TPC extraction at optimum ME and OH-ME conditions were given in Table 5 for Peleg model. The constants ( $k_1$  and  $k_2$ ) and coefficient of determinations ( $\mathbb{R}^2$ ) for Peleg model were calculated and presented in Table 6. The coefficients of determination of ME and OH-ME were found as 0.97 and 0.99, respectively. It was observed that Peleg model had high compatibility for these methods to describe yield of TPC. Similar studies were reported in the literature. Okiyama et al. (2018) stated that Peleg's model applied to flavanols extraction from cocoa bean shell provided a reasonable agreement with the experimental results. In another study, it was reported that Peleg's model ( $\mathbb{R}^2 > 0.94$ ) showed a good fitness to the extraction of bioactive compounds from coffee silverskin by Wen et al. (2019).

## **Future Aspects for Ohmic Heating**

Ohmic heating offers a number of process advantages. It has extreme potential for achieving rapid and uniform heating in foods, and can be used in extraction of bioactive compounds from food materials. The fast response/controllability of the technology allows rapid start-up, and good turndown ratios (Maloney and Harrison 2016). Even though it has many advantages, there are only 18 commercial ohmic heating plants in the world (Castro 2007; İçier 2012) due to its limitations. One of the biggest issues is that it is only suitable for pumpable samples with suitable electrical conductivity. As the conductivities of food material changes, the design of the process may have to be revised. During commercial applications, a suitable electrical supply and the cost of electrical energy have to be considered (Maloney and Harrison 2016). Besides these disadvantages, continuous efforts are being made to integrate ohmic heating in other applications in near future. Further studies are required to assess the performance and to reduce the overall cost of ohmic heating,

# Conclusions

The aim of this study was to extract phenolic compounds from cornelian cherry by novel and environmentally friendly methods (microwave and ohmic heating assisted microwave extraction) as an alternative to traditional extraction method (maceration). It was found that OH-ME and ME were more effective in extraction of phenolic compounds from cornelian cherry. Moreover, the ohmic heating application has been successfully integrated into the innovative extraction method as a pretreatment and not only increased the amount of TPC, but also significantly reduced the extraction time. The decrease in extraction time helped to preserve anthocyanins in the OH-ME extracts (0.65 mg cya-3-glu/g dry matter). The antioxidant activity of extract obtained by ME was the highest among other methods. In addition, OH-ME method ( $R^2 = 0.99$ ) was found to be the best fitting method to the Peleg model. These results indicate that the combined application of ohmic heating and microwave extraction is a potential alternative method for total phenolic compounds extraction from plant materials.

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