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ORIGINAL ARTICLE

Simultaneous extraction of phenolics and essential oil from peppermint by pressurized hot water extraction

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Abstract Phenolics and essential oil of peppermint were obtained by pressurized hot water extraction (PHWE). The best extraction conditions were found to be 130 °C for temperature, 10 min for extraction time, and 3 cycles for extraction number. There were no statistically significant differences between 130 and 160 °C in terms of essential oil content. Total phenolic contents (TPC) of the extracts were higher at 160 °C than that of 130 °C. However, further HPLC analysis of the extracts revealed that hydrolysis and/or decomposition of phenolics were observed in the extracts obtained at 160 °C. The main phenolic of peppermint was determined as eriocitrin by HPLC–DAD, while menthol was the dominant component in essential oil fraction of peppermint by GC-FID. The present study demonstrated that PHWE was a suitable technique for simultaneous extraction of phenolics and essential oil from peppermint. The technique might be used as an analytical extraction tool for determination of phenolics and essential oil. Moreover, the extracts of PHWE could directly be evaluated for the enrichment of liquid food formulations or be transformed into solid form by suitable techniques such as spray drying for easy storage and subsequent enrichment of food products.

Keywords Peppermint · Phenolics · Pressurized hot water extraction · Menthol · Eriocitrin

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Introduction

Peppermint (*Mentha piperita* L.), belongs to plant family of *Lamiaceae*, has been used in traditional medicine for its beneficial effects, and being cultivated worldwide mainly for its medicinal properties (Fecka and Turek 2007). Peppermint extracts have been used for centuries to treat irritable bowel syndrome, flatulence, indigestion, nausea, vomiting, cough and bronchitis (Kapp et al. 2013). Peppermint contains 1.2–3.9% (v/w) essential oil (Riachi and De Maria 2015). Peppermint essential oil is used in the manufacturing of consumer products such as toothpastes, chewing gums, and mouthwashes (Ciobanu et al. 2013; Farnad et al. 2014; Wojtowicz et al. 2010) because of its refreshing properties. Beverages containing peppermint essential oil might be a suitable alternative for persons having caffeine sensitivity. The percentage of phenolics in peppermint leaves (in dry matter basis) can reach up to 23%, of which 12% are flavonoids, such as eriocitrin, rosmarinic acid, hesperidin and luteolin (McKay and Blumberg 2006). Peppermint phenolics have been reported to exert antioxidant, hypolipidemic, antidiabetic, and

antitumoral activities (Figueroa-Pérez et al. 2014). Peppermint tea is a popular herbal tea, known for its refreshing taste and aroma, prepared by pouring hot water over dried peppermint leaves or peppermint tea bags (Figueroa-Pérez et al. 2014; Kapp et al. 2013). However, such preparation methods are insufficient for the extraction of essential oils from peppermint. When peppermint infusions are prepared, 75% of the phenolics are extracted, whereas 79% of the essential oil could not be extracted into the infusions (Kapp et al. 2013). Therefore, new effective techniques are needed for simultaneous extraction of phenolics and essential oils from aromatic plants.

Pressurized liquid extraction is a technique that uses solvents at elevated temperatures and pressures but below the critical point of water (Çam and Hişil 2010; Plaza and Turner 2015). This results in striking changes in mass transfer and solubility properties of the solvents (Mustafa and Turner 2011). When water is the solvent of choice, the technique is called as pressurized (hot) water extraction (PHWE). PHWE is regarded as a green technique (Heng et al. 2013) since it reduces/limits the usage of organic solvents. As the temperature of liquid water is raised under pressure, viscosity, polarity and surface tension decreases, diffusivity increases (Plaza and Turner 2015; Smith 2002). This improves the extraction ability of water. The main advantages of water as an extraction solvent are extensively discussed elsewhere (Mustafa and Turner 2011; Plaza and Turner 2015; Smith 2002).

PHWE was used for the extraction of bioactive components from plant materials e.g. phenolics (Çam and Hişil 2010; Gong et al. 2015; Matshediso et al. 2015), flavonoids (Gil-Ramírez et al. 2012) and essential oils (Dawidowicz et al. 2008; Ozel and Kaymaz 2004). Simultaneous extractions of phenolics and essential oils have great importance since their polarities are different. A literature study reported the possibility of phenolics and essential oil extraction by a conventional extraction method using ethanol–water mixtures (Durling et al. 2007). However, to the best of our knowledge, there is lack of publication regarding the possibilities of simultaneous PHWE of phenolics and essential oil from peppermint or any other plant materials. Hence, the objective of the present study was to determine the best PHWE conditions for phenolics and essential oil of peppermint.

Materials and methods

Plant materials and chemicals

Peppermint (*Mentha piperita* L.) was harvested from the Agricultural Research and Application Centre of Erciyes University (Kayseri, Turkey) in July 2014. Peppermint leaves were open air dried at room temperature without direct sunlight exposure.

HPLC grade water (18.2 MΩ cm) was purified using a Millipore Simplicity 185 water purification system (Darmstad, Germany). Organic solvents, reagents, and authentic standards for GC and HPLC analyses were supplied either from Sigma or Merck unless otherwise stated.

Experimental design

Temperature, solvent type, pressure, time and the number of extraction cycles in pressurized liquid extraction have great effects on the yield of bioactive compounds. In this study, we selected water as extraction solvent (fixed factor) since our main aim was to extract phenolics and essential oil with water. The instrument, ASE 350, maintain extractions with a fixed pressure of 10.3 Mpa. Therefore, pressure was another fixed factor throughout the experiments in this study. Finally, temperature, time and the number of extraction cycles, were selected and the effects of the factors on extraction performance were investigated by changing the levels of one factor while keeping the other factors as constant. Total phenolic content (TPC), antioxidant capacity as EC_{50} and TEAC, contents of menthol and menthone were selected as system responses to investigate the effects of factors on extraction performance.

Pressurized hot water extraction

Phenolics and essential oil of peppermint were extracted by a pressurized liquid extractor (ASE 350, Dionex Corporation, Sunnyvale, CA, USA). Extractions were performed at the following basic conditions: Ten g of peppermint leaves were placed into a 100 ml of stainless steel extraction cell. A cellulose filter (30 mm, Dionex) was placed at the bottom of the cell to prevent coarse particles from penetrating into the collection vials. The cell was then closed with cell cap and placed into the carousel of the ASE 350. Once the oven temperature reached the desired point (40, 70, 100, 130 or 160 °C), the cell was transferred into the oven. The pump of the ASE 350 started to fill the cell with water. The pump kept on working until the pressure of the cell reached to 10.3 Mpa. Static extraction time (1, 5, 10 or 30 min) was applied, and then resulting extracts were collected in 250 ml of bottles. Fresh solvent (30% of the extraction cell volume) was pumped into the cell and nitrogen gas was purged for 90 s in order to collect the rest of the solvent into the bottles. By doing that, one extraction cycle was completed. Total 5 extraction cycles were performed and the extracts were collected in separate collection bottles. Three extraction factors, temperature, time and number of extraction cycles were studied to find the best extraction

conditions. Effects of factors on the extraction yield of phenolics and essential oil were investigated by changing the level of one factor and keeping the other factors as constant.

Peppermint was also extracted with methanol using a shaking water bath (Memmert, Germany) at 40 °C for 1 h in order to compare extract yields with PHWE. The extracts were filtered through a 0.45 μm membrane filter before analytical determinations.

Analysis of the extracts

Total phenolic content

Total phenolic content of the extracts was determined according to a literature method (Çam and İçyer 2015). Final results were expressed as mg gallic acid equivalent (GAE) per g of peppermint.

Antioxidant capacity by DPPH and ABTS methods

Two antioxidant capacity methods were used. In the first method, antioxidant capacity of the extracts was determined using DPPH radical (Fernandes et al. 2016). Final results were expressed as EC_{50} value.

In the second method, antioxidant capacity of the extracts was determined using ABTS radical (Fan et al. 2015). Final results were expressed as mg Trolox equivalent antioxidant capacity (TEAC) per g of peppermint.

Headspace solid phase micro extraction of essential oil

Two ml of extract obtained from PHWE was taken into a 15 ml of an amber headspace vial with a cylindrical stir bar (15 mm × 5 mm). The vial was tightly closed with a polypropylene hole cap and PTFE/silicone septa (Supelco, Bellefonte, PA, USA). The vial was let to equilibrate at 40 °C for 10 min with a stirring ratio of 150 rpm. A thermally conditioned solid phase micro extraction (SPME) fiber (DVB/CAR/PDMS) combined with a manual SPME holder (Supelco, Bellefonte, PA, USA) was inserted into the vial. The fiber was exposed to volatiles in headspace for 30 min. After completing the extraction, the fiber was inserted into the GC injection port. Five min thermal desorption was applied at 260 °C. Menthol and menthone standards were prepared ranging from 1 μg/ml to 150 μg/ml in order to obtain calibration curves. Accuracy of the method was determined by adding three concentration levels of 50, 100, and 150% of menthol and menthone into the extracts. The same extraction and analysis procedures were applied as above noted. The accuracy was expressed as the percentage of menthol and menthone recovered by the method.

GC-FID and GC–MS analyses of essential oils

Analyses of menthol and menthone were performed using a TR-5MS (Thermo Scientific, Waltham, MA, USA) column (60 m × 0.25 mm, 0.25 μm film thickness) on an Agilent 6890 N chromatographic system equipped with a flame ionization detector. H_2 was used as carrier gas at a flow rate of 1 ml/min. A split ratio of 1:10 was used throughout the analyses. GC oven temperature was kept at 40 °C for 2 min, then raised at a rate of 25 °C/min to 100 °C, and then raised at a rate of 7 °C/min to 280 °C, and kept at 280 °C for 5 min. The peaks were identified by comparing their retention times with authentic standards of menthol and menthone.

A literature method (Yilmaztekin 2014) was applied for the identification of essential oils with GC–MS.

HPLC–DAD analysis of phenolics

Anayses were done on a Shimadzu (Kyoto, Japan) chromatographic system, consisted of two LC-20ADXR pumps, a SIL-20ACXR auto-sampler, a DGU-20A5 degasser, a CTO- 20AS VP column oven and a SPD-M20A photodiode array detector (DAD). Data were processed using LC Solution 1.25 software. Phenolics of peppermint extracts were analyzed by an Acclaim 120 C18 column (150 × 2.1 mm, particle size 3 μm, Thermo Scientific) using acetonitrile (Solvent A) and water/acetic acid (98:2, v/v) as the mobile phases at a flow rate of 0.5 ml/min. The gradient elution program was 5% A from 0 to 5 min, 5–35%A from 5 to 25 min, 35–90%A from 25 to 28 min, 90–5%A from 28 to 30 min. The column was equilibrated for 5 min at starting conditions. The UV–Vis spectra was recorded over the range of 200–600 nm in order to determine component identity and peak purity. The chromatogram was monitored at 280, 329 and 350 nm for eriocitrin, rosmarinic acid and luteolin derivatives, respectively. Quantification was based on external standard method.

Statistical analysis

One-way ANOVA, and Tukey's HSD test were performed to identify the differences between treatments using the SPSS 22.0 statistical package for Windows (IBM SPSS Inc., Chicago, USA).

Results and discussion

Effects of extraction temperature on the content of phenolics and essential oil

Extraction temperature has critical roles and significant effects on analytes of interest in any extraction technique. In common extraction techniques such as Soxhlet, sonication and stirring, extraction temperature can be changed up to the boiling point of extraction solvent (Luthria 2012). However, PHWE is a suitable technique to observe the effects of temperature above the boiling point of water. Therefore, we studied the effects of extraction temperature at five different levels (40, 70, 100, 130, and 160 °C) in order to observe the effects of temperature on the content of phenolics and essential oil.

It should be noted that, 31 components were determined in essential oil fraction of peppermint and in the extracts obtained by PHWE based on GC–MS analyses. Among the identified essential oils, menthol (46.65%) and menthone (24.55%) were the main components of peppermint extracts. The amount of menthol and menthone was quantified by external standard method using a GC-FID method. As preliminary experiments, certain types of SPME fibers were tested in order to select the most suitable one for essential oil extractions. When PDMS fiber was used, the fiber coating had swollen during essential oil extraction and resulted in stripping from the needle during retraction of the fiber. Among the tested SPME fibers, DVB/CAR/PDMS resulted in higher extraction rates than the other fibers (PDMS, CAR/PDMS). After selecting the fiber type, recovery tests were performed to determine the accuracy of the proposed method for extraction of essential oils. The recovery tests revealed that 94.1% of menthol and 74.8% of menthone were recovered from the extracts using DVB/CAR/PDMS fiber. The recovery rates were enough to quantify the amount of menthol and menthone.

Figure 1a, b and Table 1 show the effects of temperature on the contents of menthol and menthone. There was significant improvement in the contents of menthol and menthone up to 130 °C, but no improvement was observed above 130 °C.

Extraction temperature affected antioxidant capacity results from 40 to 130 °C. Increase in temperature from 130 to 160 °C resulted in statistically significant ($p < 0.05$) improvement in TPC whereas the changes in antioxidant capacity results were marginal. Moreover, insignificant decreases were observed in the contents of menthol and menthone when temperature shifted from 130 to 160 °C. Therefore, it was worth to investigate the reason why total phenolics increased when the other system responses remained almost stable above 130 °C. Total phenolics by Folin-Ciocalteu's reagent, a spectrophotometric method, can be considered as an antioxidant capacity method that does not determine the individual phenolics, instead, the method suffers from interfering substances including non-phenolics, organic acids, sugars etc. (Prior et al. 2005). Figure 1c shows the individual phenolic profile of peppermint extracts obtained at 130 °C. Eriocitrin (peak no 2 in Fig. 1c) and rosmarinic acid (peak no 5 in Fig. 1c) were the main phenolics in the extracts. Every increment in temperature from 40 to 130 °C resulted in significant improvements on the contents of individual phenolics. However, the amount of each individual phenolics was lower in extracts obtained at 160 °C compared to 130 °C. The amount of eriocitrin was 33.97 ± 5.36 and 23.97 ± 0.53 mg/g in the extracts obtained at 130 and 160 °C, respectively. There was $\sim$ 30% loss in the amount of eriocitrin when extracts obtained at 160 °C compared to 130 °C. Similar decline was observed in the amount of rosmarinic acid. The amount of rosmarinic acid was 22.32 ± 2.86 and 17.49 ± 0.27 mg/g in the extracts obtained at 130 and 160 °C, respectively. The loss in the amount of rosmarinic acid was $\sim$ 22% when 160 °C was applied as extraction temperature instead of 130 °C. This may indicate the destruction and/or hydrolysis of phenolics at 160 °C. Moreover, the extracts were dark and cloudy when 160 °C was applied. Therefore, 130 °C was selected as optimum extraction temperature.

In a study (Luthria 2012), the authors studied to find an optimum pressurized liquid extraction point for potato phenolics and noted that there were statistically significant ($p < 0.05$) increase in the amount of phenolics from 40 to 100 °C, no significant ($p > 0.05$) increase between 100 and 160 °C, and decrease at 190 °C.

In a study, solvent types were compared for the extraction of thyme essential oil using pressurized liquid extraction (Dawidowicz et al. 2008). The authors tested certain solvents to extract essential oil of thyme. Among the tested solvents (water, hexane, ethyl acetate and dichloromethane), water reported as having the lowest yield. However, the amount of thymol, the main essential oil component of thyme, was obtained in higher amounts with water than the other solvents used. It should be noted that, thymol in thyme and menthol in peppermint, very similar chemical components, are classified as oxygenated terpenes. Combining the results of our study and above study, we may speculate that PHWE is a suitable technique for the extraction of oxygenated terpenes. The results presented in our study are in good agreement with a reported study (Kubátová et al. 2001). The authors applied subcritical water extraction (another naming of PHWE) using a home-made apparatus for the extraction of peppermint essential oils. Although the authors did not report any statistical comparison, they found that the amount of

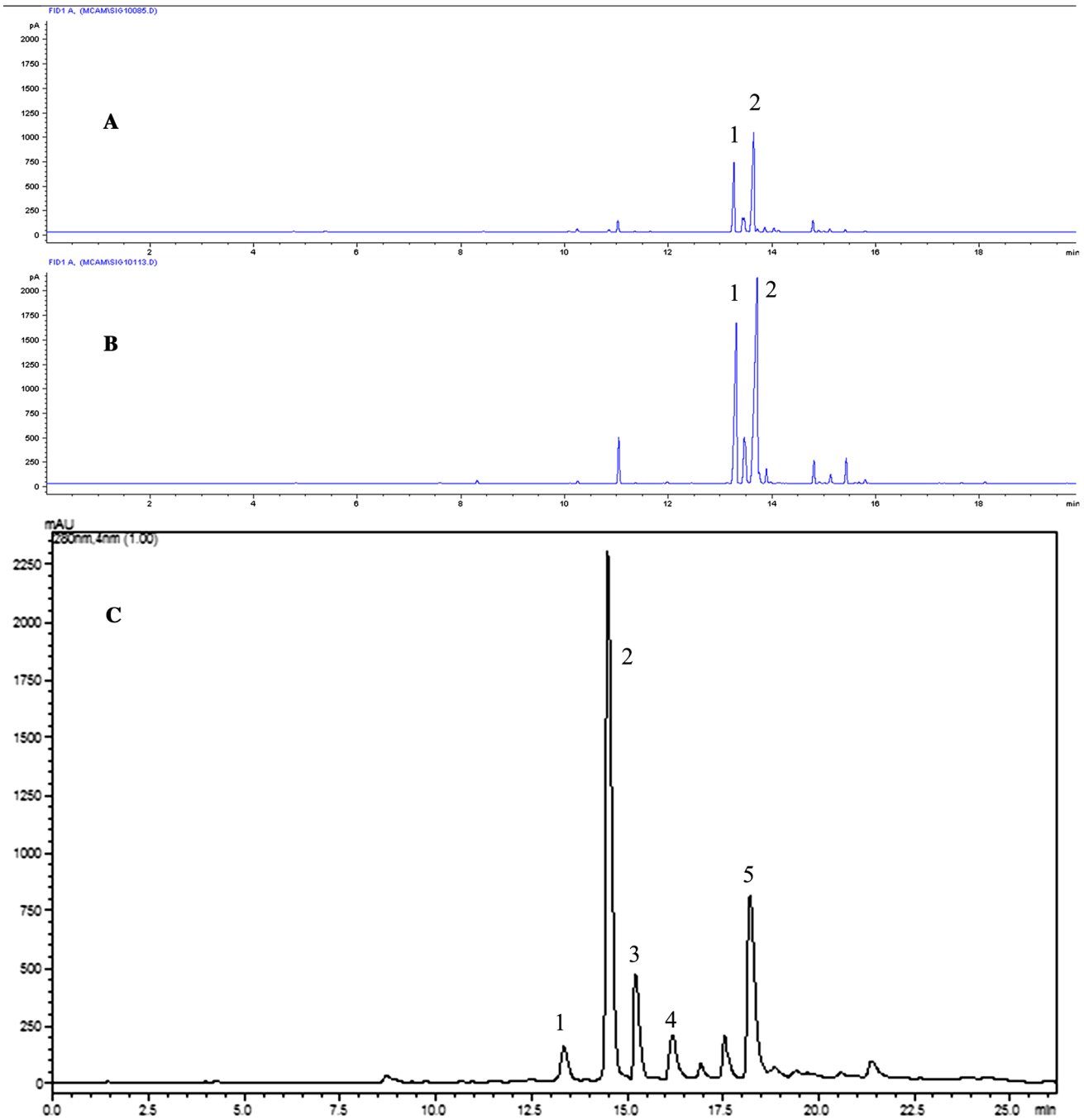

Fig. 1 GC-FID and HPLC chromatograms of essential oils and phenolics in peppermint extracts obtained by pressurized water extraction. **a** At 40 °C for 10 min. **b** At 130 °C for 10 min. Peaks for **a** and **b** are 1: Menthone; 2: Menthol. **c** At 130 °C for 10 min. Peaks for **c** are 1: Luteolin derivative 1; 2: Eriocitrin; 3: Luteolin derivative 2; 4: Luteolin derivative 3; 5: Rosmarinic acid

menthol and menthone were very close at 150 and 175 °C, thus the components could nearly be recovered from peppermint. In our study, we investigated the effects of temperature from 40 to 160 °C with 30 °C intervals. We suggest 130 °C for extraction temperature since there was no significant difference ($p > 0.05$) between 130 and 160 °C.

Effects of extraction time on the content of phenolics and essential oil

After selecting 130 °C as optimum extraction temperature, extraction time was investigated in the second stage of the study. Extractions were performed at 130 °C with four different extraction times 1, 5, 10, 30 min, while keeping the other factors constant. The results indicated that

Table 1 The effects of temperature on system responses at extraction time 10 min and at extraction cycle 1

Temperature (°C)	TPC (mg GAE/g sample)	EC_{50} (g sample/g DPPH)	TEAC (mg TEAC/g sample)	Menthol (mg/g sample)	Menthone (mg/g sample)
40	18.45 ± 0.14a	7.28 ± 2.49b	54.4 ± 0.5a	0.67 ± 0.10a	0.57 ± 0.06a
70	21.75 ± 9.57a	6.72 ± 0.41b	57.4 ± 7.7a	0.74 ± 0.09a	0.66 ± 0.11a
100	26.69 ± 8.61ab	5.40 ± 0.44ab	61.8 ± 9.2a	2.66 ± 0.32ab	1.15 ± 0.24ab
130	52.66 ± 3.79b	1.71 ± 0.06a	115.8 ± 4.9b	5.44 ± 0.91b	2.25 ± 0.31c
160	94.2 ± 6.83c	1.06 ± 0.01a	121.4 ± 5.2b	4.96 ± 0.53b	1.98 ± 0.49bc

The values are expressed as means ± standard deviations of 3 replicates. Different letters in the same column indicate significant differences ($p < 0.05$). TPC, total phenolic content by Folin-Ciocalteu's reagent; EC_{50}, the amount of sample to decrease the initial DPPH concentration by 50%; TEAC, trolox equivalent antioxidant capacity; GAE, Gallic acid equivalent

extraction time significantly affected the extraction responses (Table 2). There were statistically significant ($p < 0.05$) improvements in the TPC and antioxidant capacity results (Table 2). No improvement was observed in the contents of menthol and menthone when extraction time was between 1 and 30 min, indicating that menthol and menthone had faster extraction speed than phenolics. Moreover, both components have limited solubility in pressurized hot water since elongating the extraction time has no positive effects on the contents of menthol and menthone. We speculate that there is a saturation point in the extraction of menthol and methone with pressurized hot water, after that point, thermal load has no effect on essential oil extraction with pressurized hot water. We selected 10 min as extraction time to extract essential oils and phenolics together since there were no statistically significant improvements in the responses above 10 min.

Phenolics were obtained from parsley using pressurized liquid extraction (Luthria 2012). The author reported that different times between 5 and 15 min had no significant effect on phenolic yield. In another study, rather than time itself the importance of time–temperature combinations were indicated for PHWE of essential oils from peppermint (Kubátová et al. 2001). The authors suggested either 150 °C for 30 min or 175 °C for 12 min.

Effects of extraction cycle on the content of phenolics and essential oil

After determining the extraction temperature and time as 130 °C and 10 min, respectively, next stage was to determine the number of extraction cycles. Extractions were performed 5 times at 130 °C for 10 min, and then resulting extracts were collected separately in bottles. Results showed that still there were antioxidants and essential oils in peppermint after four extractions. Theoretically, one needs infinite number of extraction cycles to obtain 100% of the target compounds in the materials to be extracted. However, adding one more cycle to extraction results in the dilution of target compounds in the extracts. Therefore, suitable number of extraction cycle can be selected based on the purpose of the study. We suggest 3 consecutive extraction cycles for this study since approximately 90% of the phenolics, menthol and methone were extracted from peppermint (Table 3).

Comparison of extraction methods

Figure 2 shows the results of PHWE at (130 °C for 10 min) and methanol extraction (40 °C for 1 h) of peppermint. Among others, methanol is known as one of the

Table 2 The effects of extraction time on system responses at extraction temperature 130 °C and at extraction cycle 1

Time (min)	TPC (mg GAE/g sample)	EC_{50} (g sample/g DPPH)	TEAC (mg TEAC/g sample)	Menthol (mg/g sample)	Menthone (mg/g sample)
1	41.98 ± 4.18a	1.94 ± 0.08b	82.1 ± 7.1a	5.67 ± 0.76a	2.41 ± 0.12a
5	46.53 ± 4.17ab	1.77 ± 0.06ab	108.8 ± 13.1ab	5.77 ± 1.09a	2.46 ± 0.59a
10	52.81 ± 1.83b	1.54 ± 0.04a	127.1 ± 1.0c	5.95 ± 1.35a	2.55 ± 0.23a
30	56.38 ± 3.56b	1.47 ± 0.06a	121.7 ± 3.6b	6.64 ± 0.61a	2.98 ± 0.07a

The values are expressed as means ± standard deviations of 3 replicates. Different letters in the same column indicate significant differences ($p < 0.05$). TPC, total phenolic content by Folin-Ciocalteu's reagent; EC_{50}, the amount of sample to decrease the initial DPPH concentration by 50%; TEAC, trolox equivalent antioxidant capacity; GAE, Gallic acid equivalent

Table 3 The effects of extraction cycles on system responses at extraction temperature 130 °C and at extraction time 10 min

Cycles*	TPC (mg GAE/g sample)	EC_{50} (g sample/g DPPH)	TEAC (mg TEAC/g sample)	Menthol (mg/g sample)	Menthone (mg/g sample)
1st	47.79 ± 4.39c	1.22 ± 0.13a	110.2 ± 2.3d	5.74 ± 1.05c	2.29 ± 0.38c
2nd	11.31 ± 0.11b	7.57 ± 1.00b	17.3 ± 1.8c	4.55 ± 0.84bc	2.07 ± 0.07bc
3rd	4.13 ± 0.23ab	19.00 ± 0.97c	4.7 ± 2.6b	2.81 ± 0.20ab	1.38 ± 0.19b
4th	2.62 ± 0.14a	36.23 ± 1.35d	2.2 ± 0.5ab	1.18 ± 0.38a	0.52 ± 0.07a
5th	1.63 ± 0.15a	55.35 ± 6.58e	1.4 ± 0.1a	0.33 ± 0.01a	0.23 ± 0.11a

The values are expressed as means ± standard deviations of 3 replicates. Different letters in the same column indicate significant differences ($p < 0.05$). TPC, total phenolic content by Folin-Ciocalteu's reagent; EC_{50}, the amount of sample to decrease the initial DPPH concentration by 50%; TEAC, trolox equivalent antioxidant capacity; GAE, Gallic acid equivalent

*For first cycle, peppermint was extracted with water at 130 °C for 10 min and the resulting extract was collected. The same extraction conditions were applied for 2nd, 3rd, 4th, and 5th cycles by feeding fresh water into the same extraction cell containing peppermint from previous cycle. Five extracts were independently collected and analyzed

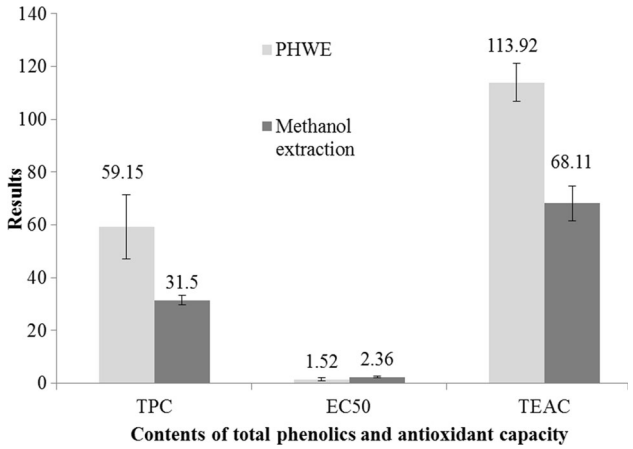

Fig. 2 Comparison of extraction methods (bars show mean ± standard deviations of three replicates. White and black bars are for PHWE and conventional methanol extraction, respectively. Individual averaged values of analyses are shown on top of the bars)

best solvent for the extraction of phenolics from plant materials. However, the present results clearly indicated that PHWE method was more effective than conventional methanol extraction in terms of TPC, EC_{50}, and TEAC. This might be due to the changes in the properties of water at 130 °C, e.g. decrease in viscosity, and polarity together with increase in diffusivity (Plaza and Turner 2015; Smith 2002).

Conclusions

This study clearly showed the potentiality of the PHWE technique for extraction of bioactive components. Changing the levels of PHWE resulted in two–fivefold improvements in the yield and antioxidant related properties. Among studied alternative experimental points, 130 °C for extraction temperature, 10 min for extraction time and 3 extraction cycles would be the best extraction conditions for simultaneous extraction of phenolics and essential oil from peppermint. The yield of the extraction at optimum conditions gave approximately twofold higher results than conventional methanol extraction. Extracts of peppermint that contain significant amount of eriocitrin in phenolic fraction and menthol in essential oil fraction can be exploited in functional food development either directly or in dried form. Future studies might focus on the effects of factors on system responses in a narrower factor levels. The PHWE method could be used as an analytical method instead of conventional extraction techniques for phenolics and essential oils.

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References

Çam M, Hişil Y (2010) Pressurised water extraction of polyphenols from pomegranate peels. Food Chem 123:878–885. https://doi.org/10.1016/j.foodchem.2010.05.011

Çam M, İçyer NC (2015) Phenolics of pomegranate peels: extraction optimization by central composite design and alpha glucosidase inhibition potentials. J Food Sci Technol 52:1489–1497. https://doi.org/10.1007/s13197-013-1148-y

Ciobanu A, Mallard I, Landy D, Brabie G, Nistor D, Fourmentin S (2013) Retention of aroma compounds from *Mentha piperita* essential oil by cyclodextrins and crosslinked cyclodextrin polymers. Food Chem 138:291–297. https://doi.org/10.1016/j.foodchem.2012.10.106

Dawidowicz AL, Rado E, Wianowska D, Mardarowicz M, Gawdzik J (2008) Application of PLE for the determination of essential oil components from *Thymus vulgaris* L. Talanta 76:878–884. https://doi.org/10.1016/j.talanta.2008.04.050

Durling NE, Catchpole OJ, Grey JB, Webby RF, Mitchell KA, Foo LY, Perry NB (2007) Extraction of phenolics and essential oil

from dried sage (*Salvia officinalis*) using ethanol-water mixtures. Food Chem 101:1417–1424

Fan R, Yuan F, Wang N, Gao Y, Huang Y (2015) Extraction and analysis of antioxidant compounds from the residues of *Asparagus officinalis* L. J Food Sci Technol 52:2690–2700

Farnad N, Heidari R, Aslanipour B (2014) Phenolic composition and comparison of antioxidant activity of alcoholic extracts of Peppermint (*Mentha piperita*). J Food Meas Charact 8:113–121. https://doi.org/10.1007/s11694-014-9171-x

Fecka I, Turek S (2007) Determination of water-soluble polyphenolic compounds in commercial herbal teas from *Lamiaceae*: Peppermint, melissa, and sage. J Agric Food Chem 55:10908–10917. https://doi.org/10.1021/jf072284d

Fernandes R et al (2016) Evaluation of antioxidant capacity of 13 plant extracts by three different methods: cluster analyses applied for selection of the natural extracts with higher antioxidant capacity to replace synthetic antioxidant in lamb burgers. J Food Sci Technol 53:451–460

Figueroa-Pérez MG, Rocha-Guzmán NE, Pérez-Ramírez IF, Mercado-Silva E, Reynoso-Camacho R (2014) Metabolite profile, antioxidant capacity, and inhibition of digestive enzymes in infusions of peppermint (*Mentha piperita*) grown under drought stress. J Agric Food Chem 62:12027–12033. https://doi.org/10.1021/jf503628c

Gil-Ramírez A, Mendiola JA, Arranz E, Ruíz-Rodríguez A, Reglero G, Ibáñez E, Marín FR (2012) Highly isoxanthohumol enriched hop extract obtained by pressurized hot water extraction (PHWE). Chem Funct Charact Innov Food Sci Emerg Technol 16:54–60. https://doi.org/10.1016/j.ifset.2012.04.006

Gong Y, Zhang X, He L, Yan Q, Yuan F, Gao Y (2015) Optimization of subcritical water extraction parameters of antioxidant polyphenols from sea buckthorn (*Hippophaë rhamnoides* L.) seed residue. J Food Sci Technol 52:1534–1542

Heng MY, Tan SN, Yong JWH, Ong ES (2013) Emerging green technologies for the chemical standardization of botanicals and herbal preparations TrAC. Trends Anal Chem 50:1–10. https://doi.org/10.1016/j.trac.2013.03.012

Kapp K et al (2013) Commercial peppermint (*Mentha piperita* L.) teas: antichlamydial effect and polyphenolic composition. Food Res Int 53:758–766. https://doi.org/10.1016/j.foodres.2013.02.015

Kubátová A, Lagadec AJM, Miller DJ, Hawthorne SB (2001) Selective extraction of oxygenates from savory and peppermint using subcritical water. Flavour Fragr J 16:64–73. https://doi.org/10.1002/1099-1026(200101/02)16:1%3c64:AID-FFJ949%3e3.0.CO;2-D

Luthria DL (2012) Optimization of extraction of phenolic acids from a vegetable waste product using a pressurized liquid extractor. J Funct Foods 4:842–850. https://doi.org/10.1016/j.jff.2012.06.001

Matshediso PG, Cukrowska E, Chimuka L (2015) Development of pressurised hot water extraction (PHWE) for essential compounds from *Moringa oleifera* leaf extracts. Food Chem 172:423–427. https://doi.org/10.1016/j.foodchem.2014.09.047

McKay DL, Blumberg JB (2006) A review of the bioactivity and potential health benefits of peppermint tea (*Mentha piperita* L.). Phytother Res Int J Devot Pharmacol Toxicol Eval Nat Prod Deriv 20:619–633

Mustafa A, Turner C (2011) Pressurized liquid extraction as a green approach in food and herbal plants extraction: a review. Anal Chim Acta 703:8–18. https://doi.org/10.1016/j.aca.2011.07.018

Ozel MZ, Kaymaz H (2004) Superheated water extraction, steam distillation and Soxhlet extraction of essential oils of *Origanum onites*. Anal Bioanal Chem 379:1127–1133. https://doi.org/10.1007/s00216-004-2671-5

Plaza M, Turner C (2015) Pressurized hot water extraction of bioactives TrAC-. Trends Anal Chem 71:39–54. https://doi.org/10.1016/j.trac.2015.02.022

Prior RL, Wu X, Schaich K (2005) Standardized methods for the determination of antioxidant capacity and phenolics in foods and dietary supplements. J Agric Food Chem 53:4290–4302. https://doi.org/10.1021/jf0502698

Riachi LG, De Maria CAB (2015) Peppermint antioxidants revisited. Food Chem 176:72–81. https://doi.org/10.1016/j.foodchem.2014.12.028

Smith RM (2002) Extractions with superheated water. J Chromatogr A 975:31–46. https://doi.org/10.1016/s0021-9673(02)01225-6

Wojtowicz E, Zawirska-Wojtasiak R, Adamiec J, Wasowicz E, Przygoński K, Remiszewski M (2010) Odor active compounds content in spices and their microencapsulated powders measured by SPME. J Food Sci. https://doi.org/10.1111/j.1750-3841.2010.01794.x

Yilmaztekin M (2014) Characterization of potent aroma compounds of cape gooseberry (*Physalis peruviana* L.) fruits grown in Antalya through the determination of odor activity values. Int J Food Prop 17:469–480. https://doi.org/10.1080/10942912.2011.642446