Extraction of Polyphenols from *Mentha aquatica* Linn. var. *crispa*

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Summary

Mentha aquatica Linn. var. *crispa* is commonly used as a spice in many Asian countries. Although its biological activities, such as its applications, antimicrobial properties, have been studied, its antioxidation properties have not been investigated. This study establishes the most suitable extraction conditions concerning the independent variables affecting the total polyphenol content (TPC) and antioxidant activity (AA) of *M. aquatica* extract (stem and leaf). Investigated factors include the type of solvent used; solvent concentration, the ratio of raw material to solvent, extraction time and extraction temperature. The efficiency of polyphenol extraction was evaluated by TPC and AA through the ability to neutralize the free radicals 1,1-diphenyl-2-picrylhydrazyl (DPPH) and 2,2'-azinobis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS), and the ferric reducing antioxidant power (FRAP) was used as the evaluation indicator. The results have shown that acetone at a concentration of 50%, at a ratio of 1:20 (w/v), extraction time of 2 h and a temperature of 40 °C give the highest values of TPC and AA, with values of 120.92 mg GAE g^{-1} dw for TPC, 169.36 μmol TE g^{-1} dw by DPPH assay, 264.03 μmol by ABTS assay, and 425.35 μmol Fe^{2+} g^{-1} dw by FRAP assay. This study demonstrates that extracts of *M. aquatica* can be used for research as food antioxidant.

Key words

antioxidant, extraction, *Mentha aquatica* Linn., polyphenols, spices

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Introduction

Currently, food preservation is increasing, helping food to avoid oxidation so food additives are often used. This method still causes some side effects. In some cases, overdosing can be harmful to human health.

The search for natural antioxidants to replace synthetic antioxidants is intensified because the use of food preservatives is increasingly regulated due to their toxicity (Puangpronpitag and Sittiwet, 2009). Several phenolic compounds found in natural products show better anti-free radical activity than synthetic antioxidants (Pinelo et al., 2005).

Mentha aquatica Linn. var. *crispa* is a plant of the mint genus, containing one of the most common essential oils, and grows wildly in Europe, Northwest Africa and Southwest Asia. It belongs to the genus *Mentha*, so it has the same characteristics as other plants of this genus. Previous studies revealed that these species possessed biological activities such as antioxidant, antimicrobial, insecticidal, anti-cancer, and anti-inflammatory properties. Essential oils and extracts of different mint genus species have been demonstrated to have antioxidant activity (AA) (Kunnumakkara et al., 2009). However, studies on the conditions for polyphenol extraction and evaluation of the AA of polyphenols from *M. aquatica* extract have not been carried out.

Nowadays, there are many polyphenol extraction methods, such as ultrasonic, microwave, or enzymatic methods, etc. However, the conventional extraction is still commonly used because of its advantages. This method is very simple and does not require complicated or expensive equipment and can be carried out with large numbers of samples (Aguilera, 2003).

In this study, the stem and leaves of *M. aquatica* were extracted and total polyphenol content (TPC) and AA of the extracts was determined.

Materials and Methods

Materials and Sample Preparation

M. aquatica used in the study was grown in Long An Province, Vietnam. Stems and leaves were dried at 40 °C until the moisture level of raw the materials was 8±2%. After that, the materials were ground smaller and passed through a 60 mesh sieve. The product was vacuum-packed and stored at 4 ± 1 °C before further experiment.

Chemicals: DPPH (1,1-diphenyl-2-picrylhydrazyl), ABTS (2,2'-azinobis (3-ethylbenzothiazoline-6-sulfonic acid), TPTZ (2,4,6-tripyridyl-s-triazine), gallic acid, and Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2- carboxylic acid) were obtained from Sigma-Aldrich (USA), while Folin-Ciocalteu (FC) reagent was obtained from Merck (Germany). Solvents and other chemicals were of analytical standard from Xilong (China).

Extraction Methodology

The extraction process was carried out by liquid-solid extraction using organic solvent. In a typical experiment, the sample was prepared as described above and different solvents were added: water, ethanol, and acetone; with solvent concentrations of 25%, 50%, 75%, and 100%; ratios of raw material/solvent of 1:12, 1:20, 1:28, and 1:36 (w/v); extraction at room temperature (30±2 °C), and 40 °C, 50 °C, and 60 °C; extraction times of 1, 2, 3, and 4 h. During the extraction process, the temperature was kept constant by water bath and shaken frequently. The extracts were analyzed for TPC and AA.

Determination of Total Polyphenol Content

The determination of TPC was based on the method published by Singleton and Rossi (1965), with the procedure adjusted by Haron and Raob (2014). The reagent used was FC with gallic acid as standard. To 0.5 mL of the sample was added 2.5 mL of FC, shaken and incubated in the dark for 5 min. Then, 2.5 mL of 7.5% Na_2CO_3 was added, followed by vortexing and incubation at room temperature for 30 min. The absorbance of the obtained samples at 760 nm was measured. For each sample, the TPC was expressed as gallic acid equivalent per gram of dry weight (mg GAE g^{-1} dw).

Determination of Antioxidant Activity by DPPH Assay

The procedure according to Al-Rimawi et al. (2016) was used with several small modifications. To 0.2 mL of each sample was added 4 mL of 0.1 mM DPPH (solution in ethanol 99.5%), followed by vortexing and storage in the dark for 30 min. Absorbance was measured at 517 nm by UV-vis spectrophotometer (UVS-2800; Labomed, USA). The results were expressed as μmol TE g^{-1} dry weight (dw).

Determination of Antioxidant Activity by ABTS Assay

Experiments were performed according to Biskup et al. (2013) with some small modifications. ABTS and potassium persulfate were dissolved in distilled water at an ABTS concentration of 7 mM and potassium persulfate concentration of 2.45 mM. The two solutions were mixed in a 1:1 (v/v) ratio and the mixture was left in the dark at room temperature for 16 h before use to promote the production of ABTS radicals ($ABTS^{\bullet+}$). The ABTS radical solution was then diluted with distilled water until the absorbance at 734 nm of of 1 was achieved. The sample extract (0.1 mL) was then added to 2.9 mL $ABTS^{\bullet+}$ solution and the mixture was put in the dark at room temperature for 6 min. The absorbance at 734 nm was determined using the UV-Vis spectrophotometer. Results were expressed as μmol TE g^{-1} dry weight (dw).

Determination of Antioxidant Activity by FRAP Assay

Experiments were conducted according to Biskup et al. (2013) with some small modifications. FRAP reagent was freshly prepared each time: A mixture of acetate buffer 0.3 M (pH = 3.6), TPTZ 0.01 M in HCl 0.04 M and $FeCl_3.6H_2O$ 0.02 M, in a 10:1:1 ratio (v/v/v) was prepared and left in the dark. Then, 0.15 mL of sample or $FeSO_4.7H_2O$ standard (0-1.8 μmol Fe^{2+} mL^{-1}) was added to 4.5 mL of FRAP reagent solution and 0.45 mL of distilled water, followed by the vortexing and incubation at 37 °C for 30 min in the dark. UV absorbance was measured at 593 nm and results are expressed as μmol Fe^{2+} g^{-1} dw.

Data Analysis

The experimental data were analyzed by the one-way analysis of variance (ANOVA) method and significant differences among the means from triplicate analysis at significant level of 95%

($P \leq 0.05$) were determined by Fisher's least significant difference (LSD) procedure using IBM SPSS Statistics software, version 20 (IBM, USA). The results were expressed as the average of three samples ±standard deviation (Mean±SD).

Results and Discussion

Effect of Type of Solvent on TPC and AA of the Extracts

The sample was extracted with three types of solvent: acetone, ethanol, and distilled water under the following conditions: raw material ratio/solvent ratio of 1:20 (w/v), extraction time of 2 h and extraction temperature of 40 °C, followed by filtration. Values of TPC and AA were determined according to DPPH, ABTS, and FRAP assay. The results are presented in Table 1.

The results show that when using acetone as solvent, the highest yield of TPC (102.99 mg GAE g^{-1} dw) was obtained and the product exhibited higher AA according to DPPH (147.63 µmol TE g^{-1} dw), ABTS (262.15 µmol TE g^{-1} dw), and FRAP (391.79 µmol Fe^{2+} g^{-1} dw) compared to using water or ethanol extraction.

That results can be attributed to the polarity of the solvents. Each solvent dissolves the substances in *M. aquatica* at different levels. A solvent's extraction efficiency depends mainly on the solubility of different specific phenolic groups, while these groups also have different antioxidant properties (Oreopoulou et al., 2019).

The obtained data show that extraction with acetone solvents was more effective than water or ethanol solvents. This result agrees with the research results of Iloki-Assanga et al. (2015) when studying *Bucida buceras* L. (leaves and trunk) while the obtained results of Tomsone et al. (2012) showed that ethanol and ethanol/water (80/20) were the most suitable solvent for the extraction of *Amoracia rusticana*. This difference can be explained by different chemical compositions of materials and the polarity of the solvents. The highest TPC yield obtained in this study is higher than that reported by Brahmi et al. (2017) who researched *Mentha pulegium* L. Their results showed a TPC of 25.3 ± 1.3 mg GAE g^{-1} dw and the AA according to DPPH was 147.63 µmol TE g^{-1} dw; the AA obtained was lower than that reported by Norhaiza et al. (2009) who researched *L. pumila* var. Alata, which gave an AA value of 299.84 µmol TE g^{-1} dw. Based on the results obtained, acetone was determined as the best solvents for the following survey.

Effect of Acetone Concentrations on TPC and AA of the Extracts

Experiments were conducted with fixed factors, including the raw materials/solvent ratio of 1:20 (w/v), extraction duration of 2 h, and extraction temperature of 40 °C. The solvent was acetone at concentrations of 25, 50, 75, and 100%. After the extraction, TPC and AA values of the extracts according to DPPH, ABTS, and FRAP assays were determined. The results are shown in Table 2.

The data in Table 2 show that when acetone concentrations increased from 25% to 50%, the TPC yield increased from 91.33 mg GAE g^{-1} dw at 25% to 118.87 mg GAE g^{-1} dw at 50%; for the AA of the extract by DPPH, ABTS, and FRAP assays, almost all increased strongly. However, there was a slight decrease when acetone concentration increased to 75% and 100%. With an acetone concentration of 75%, the AA according to DPPH showed a slight increase, but no difference compared to 50% ($P > 0.05$). The TPC obtained was reduced and the AA according to ABTS and FRAP assays also decreased compared to those at a solvent concentration of 50%.

The results above can be explained by the concentrations of organic solvents in the water, which affected the total quantity extract obtained and affected the number of specific compounds (Oreopoulou et al., 2019).

Table 1. Effect of type of solvent on TPC and AA of the extracts

Types of solvents	TPC (mg GAE g^{-1} dw)	DPPH (µmol TE g^{-1} dw)	ABTS (µmol TE g^{-1} dw)	FRAP (µmol Fe^{2+} g^{-1} dw)
Water	71.32^{a} ± 0.14	74.64^{a} ± 0.4	169.54^{a} ± 0.95	352.34^{a} ± 0.79
Ethanol	79.84^{b} ± 0.12	102.70^{b} ± 0.7	201.38^{b} ± 1.9	375.92^{b} ± 0.68
Acetone	102.99^{c} ± 0.21	147.63^{c} ± 0.8	262.15^{c} ±1.09	391.79^{c} ± 0.39

Note: Different superscript letters in the same column indicate significant differences according to LSD test at $P < 0.05$ level

Table 2. Effect of acetone concentration on TPC and AA of the extracts

Concentration of acetone (v/v)	TPC (mg GAE g^{-1} dw)	DPPH (µmol TE g^{-1} dw)	ABTS (µmol TE g^{-1} dw)	FRAP (µmol Fe^{2+} g^{-1} dw)
25%	91.33^{a} ± 0.45	142.51^{a} ± 1.18	264.03^{a} ± 1.09	393.61^{a} ± 0.68
50%	118.87^{b} ± 0.23	168.31^{b} ± 1.02	274.95^{b} ± 0.95	421.04^{b} ± 0.39
75%	109.43^{c} ± 0.60	169.32^{b} ± 1.02	272.20^{c} ±1.90	403.36^{c} ± 0.79
100%	103.00^{d} ± 0.39	147.26^{c} ± 0.59	267.80^{d} ± 1.09	395.87^{d} ± 1.04

Note: Different superscript letters in the same column indicate significant differences according to LSD test at $P < 0.05$ level

This result is consistent with the study of Do et al. (2014) on *Limnophila aromatica*, which showed the extraction of polyphenol antioxidants to be the most effective at 50% acetone concentration, when the highest yield of TPC in this study was 118.87 mg GAE g^{-1} dw. This is higher than the results reported by Benabdallah et al. (2016) who researched six species of *Mentha* from the northeast of Algeria. The highest TPC from *M. aquatica* was only 43.21 mg GAE g^{-1} dw, while the AA according to DPPH (168.31 µmol TE g^{-1} dw) and ABTS (274.95 µmol TE g^{-1} dw) assays in this study are also higher than those of Thoo et al. (2010) on mengkudu (*Morinda citrifolia*), where the DPPH and ABTS levels were 1928.5 µmol TE 100 g^{-1} dw and 791.71 µmol TE 100 g^{-1} dw, repectively. Hence, an acetone concentration of 50% was selected for the further steps.

Effect of Material to Solvent Ratio on TPC and AA of the Extracts

The extraction was conducted with fixed factors, including a solvent concentration of 50%, extraction duration of 2 h, and extraction temperature of 40 °C. The ratios of raw material to solvent were 1:12; 1:20; 1:28; 1:36 (w/v). After the extraction process, the TPC and AA of the extracts according to DPPH, ABTS, and FRAP assays were determined. The results are shown in Table 3.

The results in Table 3 show that a ratio of material to solvent of 1:20 obtained the highest TPC (119.44 mg GAE g^{-1} dw) and AA according to DPPH (168.57 µmol TE g^{-1} dw), ABTS (269.31 µmol TE g^{-1} dw), and FRAP (428.06 µmol Fe^{2+} g^{-1} dw) assays. These results are reasonable because a higher solvent/material ratio increases the mass transfer rate due to a greater difference in concentration between the solid substrate and the solvent's general phase.

Therefore, the extraction takes place at faster rate. However, the concentration of phenolic compounds in the extract is lower, while the purity of the extract may be poor due to the solubility of undesirable compounds (Oreopoulou et al., 2019). This result was consistent with the results on the leaves of *Origanum vulgare* reported by Majeed et al (2016).

The highest TPC was 119.44 mg GAE g^{-1} dw, lower than that reported by Sulaiman et al. (2011), who extracted *Portulaca oleracea* with 70% acetone in water (138.2 ± 2.1 mg GAE g^{-1} dw) and higher than that obtained in the study by Uribe et al (2016), who researched on natural antioxidants of *Mentha piperita* L. (27.12 ± 0.71 mg GAE g^{-1} dw). The AA according to DPPH assay (168.57 µmol TE g^{-1} dw) was also lower than that obtained in the study by Al-Rimawi et al. (2016) on *Tragopogon porrifolius*, who extracted with 80% ethanol in water (324 µmol TE g^{-1} dw) and. Thus, the material/solvent ratio of 1:20 was considered as desirable value for extracting *M. aquatica* polyphenols.

Effect of Extraction Temperature on TPC and AA of the Extracts

The research was conducted with fixed factors, including a solvent concentration 50%, extraction duration of 2 h, and the ratio of raw material to solvent of 1:20 (w/v). Temperatures were room temperature (28-32 °C), and 40, 50 and 60 °C. After extraction, the TPC and AA of the extract were determined according to DPPH, ABTS, and FRAP assays, and the results are shown in Table 4.

The obtained data show that extraction at 40 °C gave the best results regarding TPC (120.12 mg GAE g^{-1} dw) and AA according to DPPH (168.83 µmol TE g^{-1} dw), ABTS (267.17 µmol TE g^{-1} dw), and FRAP (419.59 µmol Fe^{2+} g^{-1} dw) assays. Although the results show that the FRAP capacity in the sample extracted at 50 °C was the highest, when compared at the 95% significance level, there was no difference.

Table 3. Effect of ratio of material to solvent on TPC and AA of the extracts

Ratio of material: solvent (w/v)	TPC (mg GAE g^{-1} dw)	DPPH (µmol TE g^{-1} dw)	ABTS (µmol TE g^{-1} dw)	FRAP (µmol Fe^{2+} g^{-1} dw)
1/12	86.68^{a} ± 0.11	126.53^{a} ± 0.28	244.80^{a} ± 0.00	382.71^{a} ± 1.39
1/20	119.44^{b} ± 0.18	168.57^{b} ± 0.78	269.31^{b} ± 3.08	428.06^{b} ± 0.52
1/28	106.5^{c} ± 0.17	150.36^{c} ± 0.62	262.02^{c} ± 2.75	404.59^{c} ± 0.87
1/36	95.65^{d} ± 0.21	138.43^{d} ± 0.78	261.85^{c} ± 2.64	401.31^{d} ± 1.15

Note: Different superscript letters in the same column indicate significant differences according to LSD test at $P < 0.05$ level

Table 4. Effect of temperature on TPC and antioxidant activity of the extracts

Temperature (°C)	TPC (mg GAE g^{-1} dw)	DPPH (µmol TE g^{-1} dw)	ABTS (µmol TE g^{-1} dw)	FRAP (µmol Fe^{2+} g^{-1} dw)
28-32 °C	102.15^{a} ± 0.21	144.01^{a} ± 1.18	243.33^{a} ± 2.88	408.80^{a} ± 0.39
40	120.12^{b} ± 0.12	168.83^{b} ± 0.45	267.17^{b} ± 1.09	419.59^{b} ± 0.91
50	113.55^{c} ± 0.24	151.51^{c} ± 0.78	253.99^{c} ± 2.17	420.59^{b} ± 1.04
60	94.59^{d} ± 0.36	120.22^{d} ± 0.90	251.48^{c} ± 0.00	411.07^{c} ± 0.79

Note: Different superscript letters in the same column indicate significant differences according to LSD test at $P < 0.05$ level

Table 5. Effect of extraction time on TPC and AA of the extracts

Extraction time (h)	TPC (mg GAE g^{-1} dw)	DPPH (μmol TE g^{-1} dw)	ABTS (μmol TE g^{-1} dw)	FRAP (μmol Fe^{2+} g^{-1} dw)
1	$107.35^{a} \pm 0.42$	$152.13^{a} \pm 0.25$	$241.98^{a} \pm 3.23$	$417.17^{a} \pm 0.52$
2	$120.92^{b} \pm 0.24$	$169.36^{b} \pm 0.22$	$264.03^{b} \pm 1.09$	$425.35^{b} \pm 0.79$
3	$121.20^{b} \pm 0.84$	$169.88^{bc} \pm 0.22$	$262.02^{b} \pm 0.00$	$423.99^{c} \pm 0.39$
4	$119.24^{c} \pm 0.48$	$168.84^{bd} \pm 0.39$	$265.29^{b} \pm 2.88$	$423.52^{c} \pm 0.52$

Note: Different superscript letters in the same column indicate significant differences according to LSD test at $P < 0.05$ level

These results can be explained by the increase in extraction temperature leading to greater permeability of the cell wall, phenolic compounds dissolving better into the solvent, and higher heat and mass transfer phenomena, leading to increased extraction efficiency; however, too high a temperature causes decomposition of some compounds (Oreopoulou et al., 2019).

This result is different from those of Thoo et al. (2010), who extracted mengkudu (*Morinda citrifolia*) with 40% ethanol in water and reported the best temperature to be 65 °C, this probably being due to the different composition and structure of the two materials. The TPC (120.12 mg GAE g^{-1} dw) is higher than that reported by Fernandes et al. (2016), who studied 13 plants, with *Origanum vulgare* showing the highest TPC (74.01 mg GAE g^{-1} dw). However, the AA according to DPPH assay (168.83 μmol TE g^{-1} dw) was lower than that obtained by Fernandes et al. (2016), with the highest value for *Origanum vulgare* by DPPH assay of 9.06 g TE 100 g^{-1} dw (361.98 μmol TE g^{-1} dw) and lower than that reported by Uribe et al (2016), who extracted phenolic compounds from *M. piperita* (135.20 ± 0.40 mmol TE g^{-1} dw). Hence, the extraction temperature of 40 °C was selected for the next experiment.

Effect of Extraction Time on TPC and AA of the Extracts

From the results above, the experiments were continued with fixed factors, including a solvent concentration of 50%, a ratio of raw material to solvent of 1:20, and a temperature of 40 °C. Extraction times were performed for 1, 2, 3, and 4 h. After the extraction process, TPC was measured and AA was established according to DPPH, ABTS, and FRAP assay of the extract. The results are shown in Table 5.

Based on Table 5, we can see that the extraction time of 2 h is reasonable. Although the TPC and the AA according to DPPH assay were the highest at an extraction time for 3 h and the AA according to ABTS assay was the highest at time of 4 h, there was no difference at the 95% significance level between 2, 3, and 4 h. These results can be explained by equilibrium being reached after about 2 h. When extracting by a conventional method with organic solvents and mild temperature conditions, there is no destruction of phenolic compounds (Oreopoulou et al., 2019).

The best extraction time coincides with the study results of Chew et al. (2011) researching *Orthosiphon stamineus* powder, but the obtained TPC (120.92 mg GAE g^{-1} dw) and AA according to DPPH (169.36 μmol TE g^{-1} dw) and ABTS (264.03 μmol TE g^{-1} dw) assays were higher than the results of Chew et al. (2011), who reported a TPC of 2003.4 mg GAE 100 g^{-1} dw, an AA according to DPPH assay of 180.9 μmol TE 100 g^{-1} dw, and according to ABTS assay of 765.4 μmol TE 100 g^{-1} dw, TPC was lower than the results of Benedec et al. (2013), they isolated polyphenols from *Mentha viridis* L. var *crispa* (246.7 ± 0.47 mg GAE g^{-1} dw).

Conclusions

Based on the experimental results obtained, the extraction efficiency depended on the type of solvents, solvent concentration, the ratio of raw materials-to-solvent, extraction time, and extraction temperature. The most suitable extraction conditions to obtain the highest TPC as well as the best antioxidant activity according to DPPH, ABTS, and FRAP assays were: aqueous acetone solution (50%, v/v), a ratio of material to solvent of 1:20 (w/v), extraction temperature of 40 °C, and extraction time of 2 h. This study showed that the TPC and AA of the extracts of *M. aquatica* were quite high and that it could be used as a source of polyphenols or applied to food to prevent oxidation.

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