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The Impact of Various Temperatures on Polyphenol and Flavonoid Extraction from *Fumaria officinalis* Herba †

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Abstract: The objective of the present study was to investigate the influence of high temperature on the extraction of polyphenols and flavonoids from *Fumaria officinalis*. The polyphenol yield varied from 16.56 to 18.33 mg gallic acid equivalent/g of dried plant material, achieving the highest value in the extract prepared using heat-assisted extraction (HAE) for 30 min. The same trend was noticed for the flavonoid concentration in the extracts (7.14–8.48 mg catechin equivalent/g of dried plant material): macerate after 60 min $\leq$ macerate after 90 min $\leq$ HAE extract after 15 min $\leq$ HAE extract after 30 min. Compared to maceration and taking into consideration the industrial requirements such as high extraction yield for a shorter time, HAE could be recommended as a convenient technique for polyphenol and flavonoid extraction from fumitory.

Keywords: fumitory; extracts; polyphenols; flavonoids

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1. Introduction

Polyphenols are a large group of plant secondary metabolites that can be employed as preservatives, antioxidants, and additives [1]. Flavonoids, as an important group of polyphenols and natural antioxidants, may reduce oxidative stress in cardiovascular and neurodegenerative diseases, diabetes mellitus, asthma, and eye disorders [2]. There is a growing interest in extracting these metabolites from plant sources to obtain a safe, natural, and low-cost alternative to synthetic compounds, out of which some possess toxic and mutagenic effects [3]. Maceration represents a simple extraction procedure that is performed at room temperature, but provides a low extraction yield and requires a long extraction time and a large amount of plant material and solvent. As an alternative to the traditional procedures for isolation and purification of bioactive phytochemicals, novel methods, including heat-assisted extraction (HAE), have been applied to improve the extraction efficiency and reduce the extraction time and solvent consumption [4]. Hence, the objective of the present study was to investigate the influence of high temperature on the extraction of polyphenols and flavonoids from fumitory or earth smoke (*Fumaria officinalis* L.). This research was an initial step in the production of polyphenol- and flavonoid-rich fumitory extracts aimed to be used for the formulation of foodstuffs and medicines.

2. Materials and Methods

The aerial part of the employed plant material, i.e., fumitory, was purchased from the Institute for Medicinal Plants Research "Dr Josif Pančić" (Serbia). A Simplicity UV® water purification system (Merck Millipore, Darmstadt, Germany) was used to obtain ultrapure water for the preparation of the extracts. Ethanol and sodium carbonate were purchased from Fisher Science (Loughborough, UK) for extract preparation and for the determination of the polyphenol yield, respectively. Folin–Ciocalteu liquid reagent, used for the determination of the polyphenol yield, and gallic acid, used for the preparation of the calibration curve, were both purchased from Merck (Darmstadt, Germany). The following reagents were employed for the measurement of the flavonoid yield: sodium nitrite (Alkaloid, Skopje, North Macedonia), sodium hydroxide (Alfapanon, Bački Petrovac, Serbia), and aluminum chloride (Sigma-Aldrich, St. Louis, MA, United States).

The extractions were performed at 25 °C (maceration) and 80 °C (HAE) in the incubator shaker KS 4000i control (IKA, Staufen, Germany) at a solid-to-solvent ratio of 1:30 g/mL employing 50% (*w*/*w*) ethanol. The extraction times used depended on the extraction procedure: 60 and 90 min for maceration, and 15 and 30 min for HAE. After the extraction, the samples were filtered through filter paper. Through varying extraction times in maceration and HAE, the extraction efficiency was examined by determining total polyphenol and flavonoid contents.

The content of total polyphenols (TPC) was determined by a modified Folin–Ciocalteu method [5]. The absorbance of the formed blue complex is proportional to the quantity of polyphenols. After 2 h of incubation in the dark at room temperature, the absorbance of the mixture (extract, water, Folin–Ciocalteu reagent, and sodium carbonate) was read at 765 nm against a blank (UV Spectrophotometer UV-1800, Shimadzu, Japan). The TPC was expressed as milligrams of gallic acid equivalent per gram of dried plant material (mg GAE/g).

The content of total flavonoids (TFC) was estimated spectrophotometrically [6] and the absorbance was recorded at 510 nm against the blank. The results were presented as milligrams of catechin equivalent per gram of dried plant material (mg CE/g).

3. Results and Discussion

The impact of high temperature on the polyphenol and flavonoid extraction from the fumitory aerial part was examined. The polyphenol yield in the extract obtained at 80 °C after 30 min was significantly higher than in the extracts obtained at room temperature (Table 1). The TPC of the extract obtained by 30 min HAE (18.33 mg GAE/g) was increased by 8.3–10.7% in comparison to the macerates obtained after 60 and 90 min (16.56 and 16.92 mg GAE/g, respectively). Comparing the TPC of the extracts obtained by maceration and HAE, it could be concluded that similar amounts of polyphenols were measured after 60 and 90 min of maceration and 15 min of HAE (16.80 mg GAE/g). The same trend can be observed for the flavonoid concentration in the extracts: macerate after 60 min $\leq$ macerate after 90 min $\leq$ HAE extract after 15 min $\leq$ HAE extract after 30 min. The TFC values varied from 7.14 to 8.48 mg CE/g.

According to the literature data, the use of thermal energy improves the efficiency of the extraction by disruption of cellular structures [7]. This feature leads to increased cell membrane permeability and breakdown of secondary metabolites—herb matrix interactions, which cause an enhancement of polyphenol solubility and mass transfer. The increment of solvent temperature could also decrease surface tension and consequently enhance the wetting of the herbal matrix, resulting in more efficient extraction [8]. High temperature decreases the viscosity of the extraction solvent, helping the medium to penetrate the plant particles and resulting in an improved and accelerated extraction process [9].

The results obtained follow the literature data, where the higher temperature had a positive impact on the polyphenol yield in ethanol plant extracts [9,10]. Compared to maceration and taking into consideration the industrial requirements such as high extraction efficiency for a reduced time, HAE could be recommended as an adequate technology for polyphenol and flavonoid extraction from *F. officinalis* herba.

Table 1. Total polyphenol and flavonoid contents of *Fumaria officinalis* extracts prepared using maceration or heat-assisted extraction.

Sample	Time	Total Polyphenols (mg GAE */g)	Total Flavonoids (mg CE/g)
Maceration	60 min	$16.56 \pm 0.25^{b,*}$	7.14 ± 0.52^{c}
	90 min	16.92 ± 0.13^{b}	$7.47 \pm 0.36^{b,c}$
Heat-assisted extraction	15 min	16.80 ± 0.32^{b}	$8.01 \pm 0.21^{a,b}$
	30 min	18.33 ± 0.70^{a}	8.48 ± 0.31^{a}

* GAE, gallic acid equivalent; CE, catechin equivalent; analysis of variance (one-way ANOVA) and Duncan's post hoc test (different letters indicated the differences that were considered statistically significant at $p < 0.05$, n = 3).

4. Conclusions

This study was an attempt to examine the impact of temperature on the extraction of polyphenols and flavonoids from *F. officinalis* herba using two different extraction techniques (maceration and HAE). The results indicated that temperature has significantly affected the content of total polyphenols and flavonoids. Thus, HAE could be used for future polyphenol and flavonoid extraction, due to obtained high TPC and TFC as well as short extraction time, compared to maceration. Furthermore, this study was an initial step in the production of polyphenol- and flavonoid-rich fumitory extracts aimed to be used for the formulation of foodstuffs and medicines. Further research should be conducted on the antioxidant, antimicrobial, anti-inflammatory, and other biological activities of the prepared extracts.

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