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Analytical Methods

Multi-element analysis of mineral and trace elements in medicinal herbs and their infusions

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ABSTRACT

Twelve mineral and trace elements (Al, B, Ba, Fe, Zn, Mn, Mg, K, Na, P, Cu, Sr, and Ca) were determined in the herbs and their infusions consumed for medical purposes in Poland such as chamomile (*Matricaria chamomilla* L.), peppermint (*Mentha xpiperita*), melissa (*Melissa officinalis*), sage (*Salvia officinalis*), nettle (*Urtica dioica*), linden (*Tilia vulgaris*) and St. John's wort (*Hypericum calycinum*). Dry digestion procedure for total concentration and wet digestion procedure for infusions were applied under optimized conditions for dissolution of medicinal herbs. Element concentrations in herbs and their infusions were determined by ICP-OES. The accuracy and precision were verified against NCS DC 73349 – bush branches and leaves certified reference material. The result of total concentrations of elements in herb leaves shows that all herbs contain most of the elements, except K and P, in the μg/g range, and that elemental concentrations varied widely. Moreover, on the basis of experimental results for the extraction efficiencies, the elements in herb infusions were classified into three specific groups: highly-extractable (>55%) including K; moderately-extractable (20–55%) including Mg, Na, P, B, Zn and Cu and poorly-extractable (<20%) including Al, Fe, Mn, Ba, Ca and Sr. The results of analysis were evaluated statistically using ANOVA one-way and three-way analysis of variance, variance correlation test and Spearman's test.

1. Introduction

Traditionally, herbs are universally applied in popular and folk medicine, and play an important role in the general state of health in many populations. They are usually not biologically aggressive and do not have severe side effects. The therapeutic activity of herbs is associated with the content of biologically active organic compounds of varying structures and remedial powers such as alkaloids, tannins, oils, or vitamins. In addition to pharmacologically active substances, there is a growing interest in their chemical composition because of ongoing developments in nutrition and biochemical surveying and mineral prospecting (Kalny, Fijałek, Daszczuk, & Ostapczuk, 2007).

Raw plant materials are derived from sources subject to action of natural biogeochemical environment and may be easily contaminated with pesticides, microbial contaminants, heavy metals, chemical toxins and adulterated with orthodox drugs during growth and processing. Herbs constitute an important link in the transfer of trace elements from soil to man. The level of essential elements in plants varies, and content being affected by the geochemical characteristics of a soil and by the ability of plants to selectively accumulate some of these elements. Bioavailability of the elements depends on the nature of their association with the constituents of a soil. Plants readily assimilate elements through the roots. Additional sources of elements for plants are rainfall, atmospheric dusts, plant protection agents, and fertilizers that can be absorbed through the leaf blades (Łozak, Sołtyk, Ostapczuk, & Fijałek 2002). Therefore, it is important to use combined regimes for studies on medicinal herbs and analysis. Due to the importance of the mineral and trace elements present in herbs, several studies have been carried out to determine their levels by using graphite furnace atomic absorption spectrometry (GF-AAS) (Kalny et al., 2007; Sołtyk & Fijałek, 2000), flame atomic absorption spectrometry (F-AAS) (Abou-Arab & Abou Donia, 2000; Chizzola, Michitsch, & Franz, 2003; Maiga, Diablo, Bye, & Paulsen, 2005), inductively coupled plasma optical emission spectrometry (ICP-OES) (AL-Oud, 2003; Başgel & Erdemoğlu, 2006; Malik, Szakova, Drabek, Balik, & Kokoska, 2008), inductively coupled plasma-mass spectrometry ICP-MS (Nookabkaew, Rangkadilok, & Satayavivad, 2006; Raman, Patino, & Nair, 2004), INNA (Abugassa et al., 2008; Choudhury, Acharya, Nair, Reddy, & Garg, 2008; Garg, Kumar, Nair, & Reddy, 2007; Hamzah, Beh, Sarmani, Liow, & Abugassa, 2004) or X-ray fluorescence spectrometry (XRF) (Bumbálová, Komová, & Dejmková, 1992; Harangozó, Tölgyessy, Tomeček, Ružička, & Cejpek, 1999).

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Table 1
Major herbs used for medical purposes in Poland.

Herb (scientific name)	Indications
Chamomile (*Matricaria chamomilla* L.)	Antispasmodic, anti-inflammatory, antimicrobial, mild sedative, anti-mutagenic, cholesterol-lowering
Peppermint (*Mentha xpiperita*)	Anti-spasmodic, diaphoretic, digestive, antiseptic, slightly anesthetic, decongestant, and cooling
Melissa (*Melissa officinalis*)	Cooling and sedative, lowering fever, improving digestion, relaxing spasms, improving peripheral blood vessels, helps with hyperthyroidism, anti-viral, and anti-biotic
Sage (*Salvia officinalis*)	Astringent, antiseptic, relaxes spasms, suppresses perspiration and lactation, improves liver function and digestion, anti-inflammatory, and anti-depressant
Nettle (*Urtica dioica*)	Disturbances in metabolism against a shortage of microelements, inflammation of urine, tracts in gastric and colitis, anticancer, slightly reduces blood pressure, clears toxins, and reduces prostate enlargement
Linden (*Tilia vulgaris*)	Diuretic, expectorant and calming properties, lowering blood pressure, sedative, calming the digestive system, reducing respiratory mucus in colds and flu, and boosting the cardiovascular system
St. John's wort (*Hypericum calycinum*)	Cooling and astringent, calm the nerves, reduce inflammation, promote healing, moderate depression, nervous tension, insomnia, menopausal disturbances, premenstrual syndrome, shingles, sciatica and fibrosis's

The objective of this study was to investigate the levels of some of the mineral and trace elements (Al, B, Ba, Fe, Zn, Mn, Mg, K, Na, P, Cu, Sr, and Ca) in the most popular herbs and their infusions that are widely and habitually consumed for medicinal purposes in Poland (Table 1). Knowledge of the elemental content in medicinal plants is very important, because many trace elements play a significant role in the formation of active constituents which are responsible for their curative properties. Moreover, some of these elements are vitally important for various metabolic processes in the human body. They are closely linked to human growth and general health. Deficiency or imbalances of these elements could cause physiological disorders. Furthermore, plant materials, including herbs, are usually examined for the content of elements harmful to health. There are a few data on the content of the other elements, particularly in infusions which are quite willingly consumed by humans. For this reason, accessibility studies seem to be interesting as the total elemental concentration in herbs would be helpful for obtaining a realistic picture of the human intake of these elements. Not only is determination of elemental concentrations in these medicinal herbs very important as such; also, a reliable analytical procedure is an essential step in such studies. Therefore, prior to determining the elements in herbs and their infusions by ICP-OES, we aimed at optimizing the digestion procedure for dissolution of herbs. This included testing the analytical characteristics of the proposed method including its accuracy and precision as well as verifying data with the help of certified reference material. Additionally, extraction efficiency study of the infusion process and statistical analysis were performed to obtain more detailed information about global changes of the levels of the elements in herbs and their infusions.

2. Materials and methods

2.1. Apparatus

Measurements were carried out using a sequential spectrometer with an excitation in the ICP plasma (Spectro Analytical Instruments) and application of the following operation parameters: frequency – 27.12 MHz, RF output power – 1.1 kW, coolant gas – Ar, 14 L/min, auxiliary gas –Ar, 0.5 L/min, nebulizer gas – Ar, 1 L/min, observation height – 11 mm, nebulizer – concentric type Meinhard, monochromator with 2400 lines/mm, grating, sample uptake rate – 1 mL/min, wavelength – Al 396.152 nm, B 249.773 nm, Ba 455.403 nm, Cu 324.754 nm, Fe 259.94 nm, K 766.49 nm, Mg 383.826 nm, Mn 257.61 nm, Na 589.592 nm, P 213.618 nm, Sr 407.771 nm, Zn 213.856 nm, Ca 315,887.

2.2. Reagents

All chemicals purchased from commercial sources were of analytical grade. High purity deionized water from a Milli-Q system (Millipore, Milford, USA) was used for preparation of all solutions. All solutions of multielemental (Merck, Germany) standards were prepared daily by dissolving reference materials in water and used for calibration. The analyte concentration ranges, coefficients for regression equation ($y = ax + b$, where y – emission intensity, x – analyte concentration), μg/mL, sensitivity, repeatability and uncertainty were listed in Table 2.

Table 2
Analytical conditions for determination of some elements in the herbs and their infusions by using ICP-OES.

Element	Concentration range, μg/mL	Correlation coefficient	Sensitivity[1], imp · mL/ng	Repeatability[2], μg/g	Uncertainty[3], %
Al	**0.64–80**	0.9999	1.24	3.30	2.9
B	**0.8–100**	0.9999	3.93	0.14	0.9
Ba	**0.0064–0.8**	0.9998	55.8	0.09	0.5
Ca	**0.64–80**	0.9979	1.58	2.9	2.0
Cu	**0.016–2**	0.9998	80.5	0.02	2.6
Fe	**0.016–2**	0.9998	50.1	0.46	3.3
K	**1.6–200**	1.0000	0.54	15.6	0.6
Mg	**0.16–20**	0.9998	17.0	3.4	1.0
Mn	**0.008–1**	0.9998	242.3	0.14	2.0
Na	**1.6–200**	0.9998	0.14	4.1	2.5
P	**1.6–200**	0.9999	0.27	6.6	0.3
Sr	**0.0064–0.8**	0.9998	167.0	0.28	0.7
Zn	**0.16–20**	0.9998	11.6	0.27	0.5

[1] Sensitivity of the determination of each chemical element at a specific analytical line was expressed by the slope of the linear regression equation. Linearity was assessed by the correlation coefficients of calibration curves and was considered acceptable when $r = 0.9995$.

[2] Repeatability of the method was determined using the following formula: $r_p = t_{(\alpha,f)} \cdot s$ where $t_{(\alpha,f)}$ – Student's constant read from the tables at $\alpha = 0.05$ level of significance; s – standard deviation of the determination results for each studied element.

[3] Measurement uncertainties for each studied element were determined using the following formula: $u_c = \frac{r_p}{\overline{x}} \cdot 100\%$ where r_p – repeatability of the method, $\overline{x}$ – average content of the element in herb.