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Quantitative Analysis of Selenium and its Importance in Food Supplement Quality and Safety

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Abstract

Selenium (Se), a miniature metalloid element found in the environment in organic and inorganic forms, exhibits significant antioxidant, anti-inflammatory, anticancer, potentially antiviral, and antibacterial activity. Lately, selenium-enriched foods and selenium-based food supplements have been identified as important sources for Se supplementation in the human diet. Thus, verification of the composition of food supplements is essential to ensure consistency between the declared and actual content of this type of product, given the still insufficiently consolidated regulatory framework in Romania. The main goal of this work was to perform an analytical control of selenium-based food supplements using two analytical methods: X-ray fluorescence (XRF) and FT-IR. Total Se analysis, determined using a portable XRF (pXRF) device, was in good agreement with the value labeled on commercial supplements. For one sample (S2), the observed deviation is likely due to matrix effects and/or variations in the chemical form of selenium, both of which can affect the analytical response in XRF measurements. Therefore, it can be concluded that the combination of pXRF and FT-IR analyses provides rapid, high-performance quality control for selenium-based food supplements.

Keywords: food supplements, portable handheld X-ray fluorescence, FT-IR analysis, total selenium content

1. Introduction

Selenium is an essential trace element involved in the proper functioning of human and animal species due to its significant antioxidant, anti-inflammatory, anticancer, potentially antiviral and antibacterial activities. This metalloid is most often ingested through the diet. The presence of selenium in food is correlated with its cycle in the environment. The distribution of selenium in the environment primarily depends on its chemical speciation,

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whether inorganic or organic. Organic selenium compounds (selenoproteins) are absorbed and retained more efficiently than their inorganic counterparts (selenite (SeO_3^{2-}) and selenate (SeO_4^{2-})) [1-4]. In living organisms, selenium is found as a component of more than 25 selenoproteins, while the best-known selenium-containing amino acids are selenomethionine (SeMet) and selenocysteine (SeCys). Organic selenium species represent important forms of selenium in food and play a crucial role in human health. These organic compounds are notable for their redox activity and anti-inflammatory properties [5]. Moreover, selenium can be used as selenium nanoparticles (SeNPs) to develop packaging with important antioxidant and antimicrobial properties [6].

Food-based supplementation is more widely acceptable among the general population than pharmaceutical supplementation. Foods rich in Se, whether natural or artificially processed, have proven to be high-quality sources for supplementing Se in the human diet [7, 8]. However, current trends indicate an increasing consumption of selenium-containing food supplements from various manufacturers as an alternative to ensure adequate intake.

In this context, verifying the composition of food supplements is essential to ensure consistency between the declared and actual content of this type of product, given the still insufficiently consolidated regulatory framework in Romania. The determination of total selenium in various matrices can be achieved using a series of spectral techniques, such as XRF, ICP-MS, or XAS, which have proven to be the most suitable for quantifying total selenium [9-11]. The selection of an appropriate selenium quantification technique is typically developed in conjuction with a suitable sample preparation method, taking into account the matrices to be tested and the expected selenium concentrations [11, 12].

In this work, an analytical control method for selenium-based food supplements using a portable X-ray fluorescence (pXRF) device and FT-IR analysis was developed. A rapid quantification of total Se in various commercially available food supplements was performed using a portable handheld X-ray fluorescence (pXRF) instrument. The total Se content of food supplements using the pXRF device was in good agreement with the value labeled on the commercial supplements. In conclusion, the combination of pXRF and FT-IR analysis provides rapid, high-yield quality control for selenium-based food supplements.

2. Materials and methods

2.1. Materials

To perform the analytical control, five selenium-based food supplements obtained from community pharmacies were considered. The supplements were first classified according to the generic selenium form declared on the label: inorganic selenium as sodium selenite and organic selenium as L-selenomethionine. Among the investigated supplements, two contained selenium in the inorganic form as sodium selenite, whereas the other three contained selenium in the organic form as L-selenomethionine. The five commercially available selenium-containing food supplements investigated in the present study were: (S1) Premium Formula Seleniu, (S2) Seleniu Forte, (S3) Selenium, (S4) Selenium and (S5) Selenium non-yeast source. According to the product labels, S1 and S2 contained sodium selenite, while S3, S4 and S5 contained L-selenomethionine. Additional information regarding the manufacturer, batch number, and expiry date has been included in Table 1 to ensure better traceability of the analysed samples.

Table 1. Coding and labeling information of the analyzed samples

Sample coding	Sample name	Manufacturer	Batch number	Expiry date
S1	Premium Formula Seleniu	Aronia Charlottenburg	S20250331	31.03.28
S2	Seleniu Forte	Sara Natural	0825	31.08.27
S3	Selenium	G&G Food Supplies	09725251	08.09.28
S4	Selenium	Solgar	5148UM0610	30.09.27
S5	Selenium non-yeast source	Nature's way	20194795	31.01.28

All samples, each containing 200 µg selenium per capsule, were ground into fine powders and stored at –20 °C until analysis.

2.2. FT-IR spectra

Infrared spectra of selenium food supplements were obtained with a Bruker ALPHA FT-IR spectrophotometer (Bruker Optik GmbH, Ettlingen, Germany) in the range of 4000-500 cm^{-1}.

2.3. Quantification of Se by portable X-ray fluorescence (pXRF) device

Elemental analysis was performed using a portable X-ray fluorescence (pXRF) spectrometer (ElvaX

series, Model CEP-01; Elvatech Ltd., Kyiv, Ukraine). The instrument operates on a power supply range of AC 100–242 V (50/60 Hz), with a maximum power consumption of 50 W, and is rated IP30. The ElvaX spectrometer enables rapid, non-destructive multi-elemental analysis across a wide range of sample types using X-ray fluorescence principles, making it applicable to environmental, materials, and geochemical studies. The pXRF system allows for multi-element detection over a broad atomic number range, typically from Na to U. Under the standard atmospheric conditions applied in this study, reliable quantification was achieved for elements ranging from approximately Mg/Al to U, with the highest sensitivity observed for medium and heavy elements, including transition metals and metalloids such as Se. An acquisition time of 300 s per sample was selected to ensure an appropriate balance between analytical precision and measurement efficiency. All food supplement samples were analyzed in triplicate.

3. Results and Discussion

3.1. FT-IR analysis of inorganic and organic forms of selenium from food supplements

Usually, pure sodium selenite exhibits symmetric and asymmetric stretching vibrations of Se-O at 725 and 807 cm^{-1} [13]. In the spectra of the two supplements containing selenium in inorganic form, broader, weaker peaks were identified (S1 – 871.69 and 1017.74 cm^{-1}; S2 – 1026.57 cm^{-1}) (Figure 1).

L-selenomethionine is often found in food supplements because it is better absorbed than inorganic forms of selenium and is more stable in the body. According to its chemical structure, it is an organoselenium compound that has a characteristic methylseleno group (-$SeCH_3$). According to Gul et al., the vibration of the Se-C bond exhibits at 792 cm^{-1} [14]. In case of the three food supplements with selenium in organic form, the vibration of Se-C was recorded at 757.62 cm^{-1} (S3), 885.63 cm^{-1} (S4), and 895.63 cm^{-1} (S5) (Figure 1). Moreover, in these FT-IR spectra, a series of characteristic peaks for amino acids was identified, including the C-H stretching band around 2800-3000 cm^{-1} [15]. Thus, the three samples with L-selenomethionine showed different broad bands corresponding to the asymmetric and symmetric stretching vibrations of the C-H bond (S3 - 2927.70 cm^{-1}, S4 - 2850.50 cm^{-1}, and S5 - 2892.57 cm^{-1}) (Figure 1). Therefore, the broad bands indicate hydrophobic regions in the composition of these food supplements containing selenium in organic form.

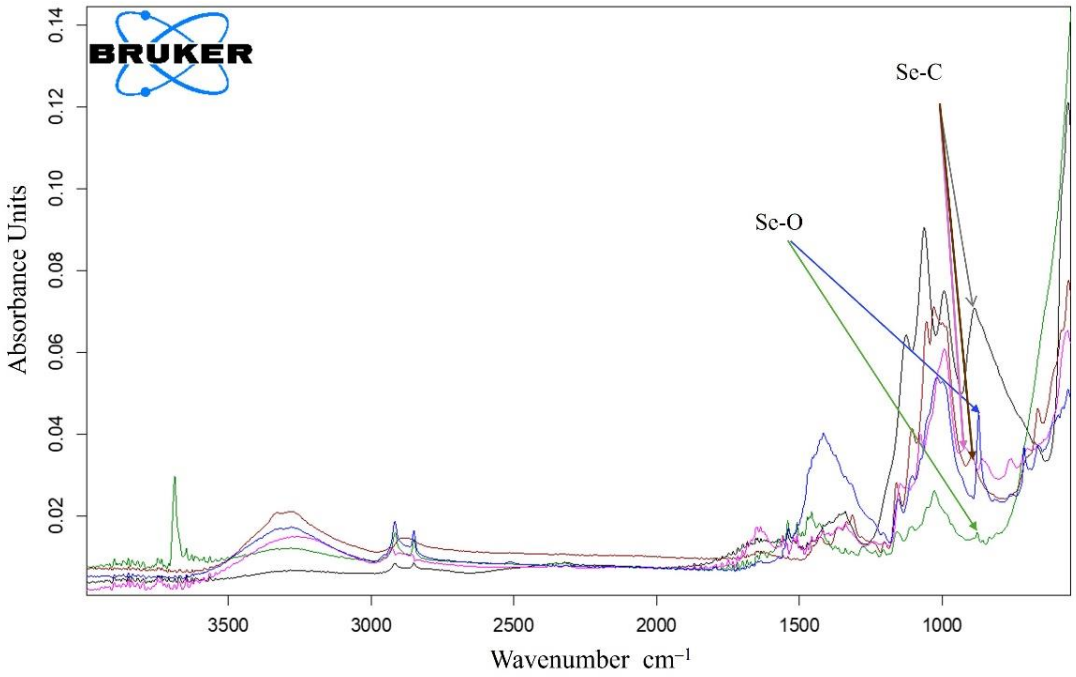

Figure 1. FT-IR spectra of selenium food supplements (blue line – S1, green line – S2, purple line – S3, black line – S4, brown line – S5)

3.2. Total Se analysis in food supplements

Analysis of trace elements in soils, sediments, rocks, and various food matrices can be performed using a portable X-ray fluorescence (pXRF) device. This elemental analysis using the pXRF device is non-destructive, fast, environmentally friendly, and low-cost, and is suitable for a wide range of sample types (solid or liquid). At the same time, pXRF eliminates the need for sample pretreatment, making this device a promising tool for elemental analysis of food samples. In addition, the XRF device is easy to transport and requires no special equipment at the experimental site [16].

Total selenium in five food supplements containing selenium in inorganic or organic form was determined in triplicate using a pXRF. The experimental results are presented in Table 2.

Table 2. Determination of total Se using a pXRF device

Sample	Se content (µg/g)		Recovery (%)
	pXRF (mean) ± SD	Labeled total Se	
S1	664 ± 0.34	660.06	100.6
S2	117.3± 0.17	197.43	59.4
S3	388.6± 0.46	387.6	100.3
S4	481.3± 0.28	382.41	125.9
S5	676.7± 0.29	675.67	100.2

*S1 and S2 – food supplements with inorganic Se (sodium selenite); S3, S4 and S5 – food supplements with organic Se (L-selenomethionine) The XRF values are reported as mean values (n = 3).

The experimentally obtained data were, for the most part, in good agreement with the labeled values, with most recovery values close to 100%, indicating satisfactory accuracy of the pXRF method. The deviations observed in some samples may be attributed to matrix effects and differences in selenium chemical form, which can influence the analytical response. Therefore, the rapid *in situ* quantification of Se in various food supplements using a pXRF device with minimal sample pretreatment proved to be a rapid method for the analytical control of these supplements.

4. Conclusion

Due to selenium's significant antioxidant, anti-inflammatory, and antibacterial activity, various Se-enhanced food products and selenium-based food supplements have been developed recently. Therefore, verifying the composition of food supplements is essential to ensure consistency between the declared and actual contents of this type of product. At present, the regulatory framework for these food supplements is still insufficiently consolidated in Romania.

In this paper, an analytical control of Se in various commercially available food supplements was performed using a pXRF device and FT-IR analysis. FT-IR analysis was used to identify the chemical species (selenite (SeO_3^{2-}) or selenomethionine (SeMet)). The rapid quantification of total Se content of food supplements was performed using the pXRF device. The experimentally obtained values were in good agreement with those listed on the commercial supplements. In the case of a single sample, the observed deviation may be attributed to matrix effects and/or differences in the chemical form of selenium, which may influence the pXRF analytical response. Therefore, the combined use of pXRF and FT-IR techniques enables rapid and efficient quality control of selenium-based food supplements. While pXRF provides quantitative information on the total Se content, FT-IR contributes to the qualitative characterization of the chemical species present. However, additional analyses using complementary methods are required to fully confirm and validate the results.

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