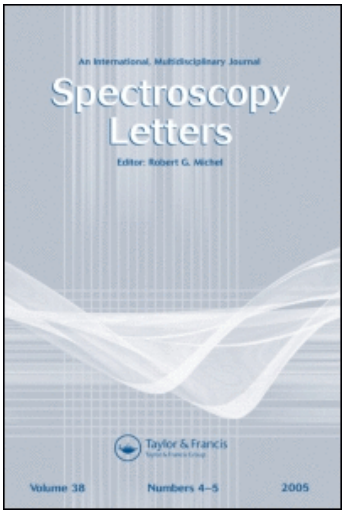

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Polarized Energy Dispersive X-ray Fluorescence Applications of Spice Samples

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Polarized Energy Dispersive X-ray Fluorescence Applications of Spice Samples

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ABSTRACT In this study, the elemental concentrations of some spice plants gathered from Mut-Mersin in Turkey were analyzed by polarized energy dispersive x-ray fluorescence (PEDXRF) spectrometry. The analyzed spices are peppermint (*Mentha piperita*), thyme (*Thymus vulgaris*), and sumac (*Rhus glabra*). These samples are very often used in the preparation of Turkish foods. The spice samples are indispensable in foods for Turkish people. The results of the analysis show that each sample has different compositions, and some of these elements have a great benefit in human health. The spice samples results reveal that the PEDXRF method is more reasonably sensitive in the determination of the elemental composition of the plants.

KEYWORDS EDXRF, multielemental analysis, spices

INTRODUCTION

Spices have been used in nutritionally insignificant quantities as a food additive for the purpose of flavoring for thousands of years.[1] Spices are generally aromatic and delicious matter. Most peoples prefer to use spices because of their different taste and different flavor.

Food purity is very important for human health. Spices may contain microbial contaminants or various trace or major elements via fertilizing and planting, drying, storage, transportation, grindinged handling.[1]

PEDXRF is a very sensitive, fast, easy, highly recommended, nondestructive, reliable, and useful technique to determine the chemical compositions of various types of samples.[2–10]

Recently, the XRF techniques are one of the most popular methods for food and plant samples analysis. Carvalho and coworkers analyzed heavy metals in wild edible mushrooms under different pollution conditions by XRF spectrometry.[11] Ekinci et al. analyzed iodine, and calcium concentrations in the bread improver.[12] Tıraşoğlu and coworkers studied samples of some plants.[13] Doğan and Tıraşoğlu detected potassium, calcium, and chlorine concentration in some vegetables by EDXRF.[14] Çevik et al. determined some elemental concentration in tobacco sample and its ash analyzed by XRF.[15] Aslan et al. analyzed some lichens and determined the elemental compositions of these plants by energy dispersive x-ray fluorescence spectroscopy.[16] Jayasekara et al. studied elemental analysis of curry and tumeric plant powder by energy dispersive x-ray fluorescence spectrometry.[17]

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Al-Bataina and coworkers analyzed some spice samples using XRF technique. They searched Mg, Al, Si, P, S, Cl, K, Ca, Ti, Mn, Fe, Cu, Zn, Br, Rb, and Sr in 10 oriental spice samples.[18] Queralt et al. analyzed some elements in medicinal plants and their infusions by XRF techniques.[19] Perring and coworkers analyzed infant cereal matrices by XRF technique.[20]

In this study, three dried and ground spice samples (*Mentha piperita*, *Thymus vulgaris*, and *Rhus glabra*) are analyzed by the PEDXRF method to figure out the elemental compositions.

MATERIALS AND METHODS

Dried spice samples were ground to a fine powder (<200 μm) using an agate mortar. Then about 4 g of the sample was taken and pressed into thick pellets of 32 mm diameter using wax as binder. Elemental concentration of spice samples was determined with the polarized energy dispersive x-ray fluorescence (PEDXRF) technique. The instrument used was a Spectro XLAB 2000 PEDXRF (Germany) spectrometer equipped with an Rh anode x-ray tube. The detector of the spectrometer was a Grefham Sirius 50/BE/XRF. The detector of the spectrometer is liquid nitrogen cooled Si(Li) with resolution of less than 150 eV at Mn Kα, 5000 cps. The sample to be analyzed was excited with linear polarized x-ray radiation, and ideally none of the primary radiation scattered by the sample reaches a suitably positioned detector. The PEDXRF spectrometer configures source beam, scattered beam, and fluorescent beam all at mutually orthogonal angles. The layout of the PEDXRF is presented in Fig. 1.[21]

The sample was analyzed by PEDXRF mainly by three types of targets. The first target is suitable for the light elements with $Z > 22$, and this target is named the Barkla target. The second target is oriented crystal target and is suitable for light elements up to $Z = 22$ and is can be named the Bragg targets. The third target is pure metal target and is suitable for specific elements or small groups of adjacent elements. In addition, this target is useful for generating Compton scatter peaks, which can be used for matrix correction.[21]

The results of the measurement of the spectrometer system were controlled by determining the elemental concentration and were calibrated with the GBW-07605 (tea) an GBW-08501 (Peach leaf) standards.

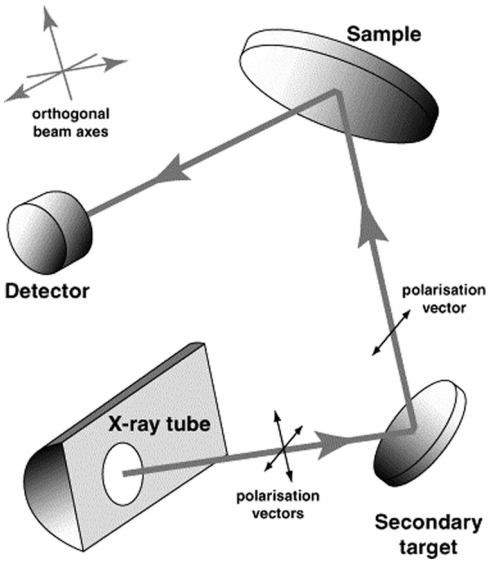

FIGURE 1 Schematic diagram of PEDXRF analysis.

TABLE 1 Elemental Concentration (Mean ± SD) of the Spice Samples by PEDXRF[a]

Element	*Mentha piperita*	*Thymus vulgaris*	*Rhus glabra*
Na	ND	ND	8000 ± 1100
Mg	2487 ± 93	3052 ± 99	570 ± 100
Al	449 ± 19	4157 ± 42	439 ± 20
Si	1586 ± 16	1.066 ± 0.004%	729 ± 13
P	2700 ± 12	1040 ± 9	205.7 ± 5.0
S	3564 ± 10	1561 ± 7	813.0 ± 5.3
Cl	1903 ± 6	1326 ± 5	1.309 ± 0.002%
K	2.010 ± 0.006%	5395 ± 34	9177 ± 46
Ca	1.945 ± 0.005%	3.108 ± 0.006%	2699 ± 19
Ti	19.6 ± 1.4	212.2 ± 3.4	17.2 ± 1.4
V	ND	2.3 ± 1.3	ND
Mn	54.7 ± 3.1	37.8 ± 2.8	5.5 ± 1.7
Fe	198.9 ± 4.3	1717 ± 13	200.4 ± 4.0
Ni	3.3 ± 0.4	3.4 ± 0.4	2.2 ± 0.3
Cu	31.1 ± 1.0	40.0 ± 1.2	20.9 ± 0.8
Zn	32.0 ± 0.8	31.7 ± 0.8	12.9 ± 0.5
Ga	ND	0.8 ± 0.3	0.4 ± 0.2
Br	2.8 ± 0.2	2.8 ± 0.2	12.3 ± 0.3
Rb	5.4 ± 0.2	8.4 ± 0.2	5.3 ± 0.2
Sr	62.5 ± 0.4	32.6 ± 0.4	25.8 ± 0.3
Y	3.5 ± 0.3	ND	ND
Zr	ND	11.7 ± 1.5	ND
Mo	4.0 ± 0.8	ND	ND
Te	ND	1.9 ± 0.6	ND
I	2.2 ± 1.0	ND	ND
Ba	21.6 ± 2.8	48.6 ± 2.8	11.3 ± 2.9
La	6.1 ± 3.9	27.2 ± 3.9	11.0 ± 4.1
Ce	8.6 ± 5.3	39.6 ± 5.4	ND

[a]Concentrations of elements are given in μg/g except for those of major elements in %; precision is given as standard deviation in each case for 5 different measurements.

ND, not detected.

RESULTS AND DISCUSSION

The results of all data are formed into the average of three measurements on each sample. The concentration values of spice samples were searched for 28 elements (mean + SD) as given in Table 1. XRF spectrums of the spice samples are shown in Figs. 2–4. Figure 2 is the XRF spectrum of *Mentha piperita*, Fig. 3 presents the XRF spectrum of *Thymus vulgaris*, and Fig. 4 shows the XRF spectrum of *Rhus glabra*.

As shown in Table 1, the major elements (≥1000 ppm) in some samples are generally Na, Mg, Si, P, S, Cl, K, and Ca. Na was detected in only *Rhus glabra* sample. Mg was detected as a major elements in *Mentha piperita* and *Thymus glabra* samples. Si, P, and S were major element level in these samples, but these elements are minor elements (<1000 ppm) for *Rhus glabra sample*. Al and Fe are major elements for only *Thymus vulgaris* sample. K and Cl are major elements in all samples. Ca, K, and P elements have great benefits in human health (Fig. 5).

The other investigated elements (Ti, V, Mn, Ni, Cu, Zn, Ga, Br, Rb, Sr, Y, Zr, Mo, Te, I, Ba, La, and Ce) are minor elements for the spice samples. Some of these elements are not detected for some samples. For example V, Y, Zr, Mo, Te, and I are detected in only one sample. They are not detected in the other samples. In addition, Mn, Ni, Cu, Zn, Br, Rb, Sr, Ba, and La are detected as minor element level in all samples. Ga, As, Cs, Ce, and Pb are detected in minor element level for only two types of samples (Fig. 6).

Concentrations of major and minor elements in spice samples are compared in Fig. 5 and Fig. 6, respectively.

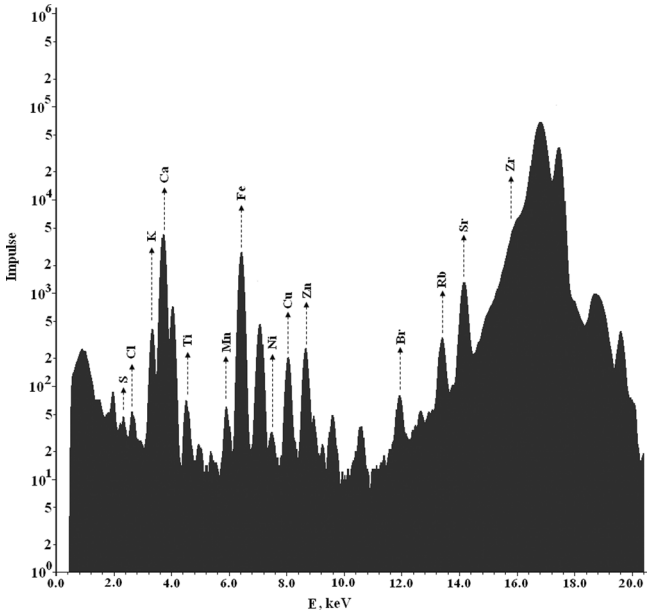

FIGURE 3 PEDXRF spectrum of *Thymus vulgaris* sample.

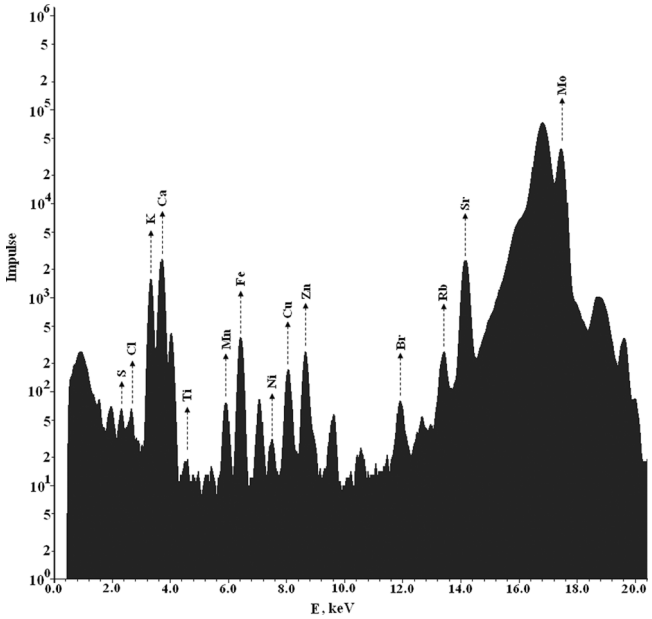

FIGURE 2 PEDXRF spectrum of *Mentha piperita* sample.

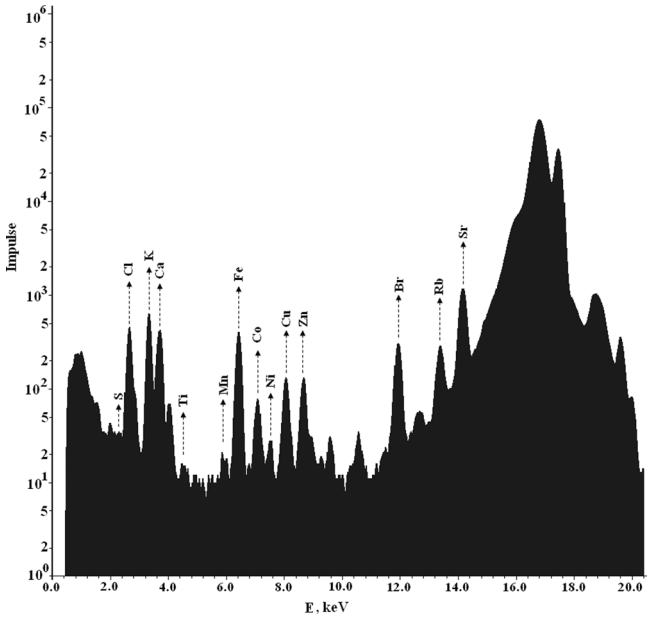

FIGURE 4 PEDXRF spectrum of *Rhus glabra* sample.

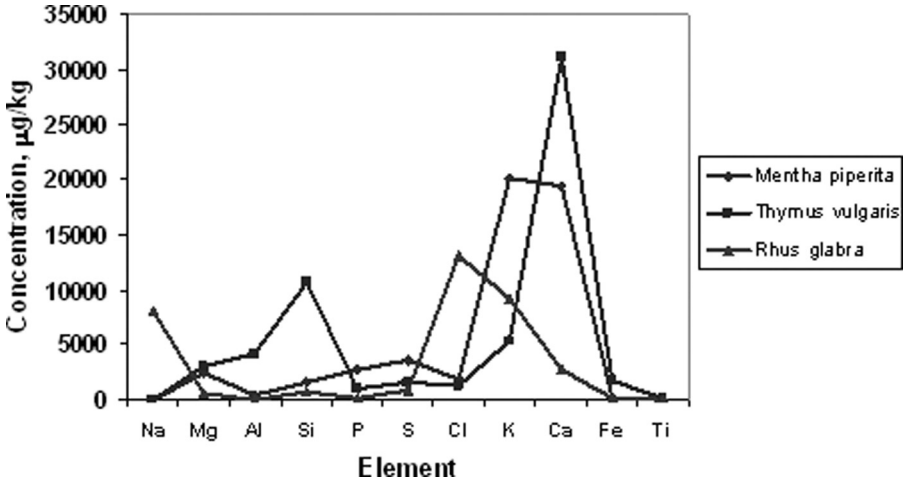

FIGURE 5 **Comparison diagram of major elements and Ti in the samples.**

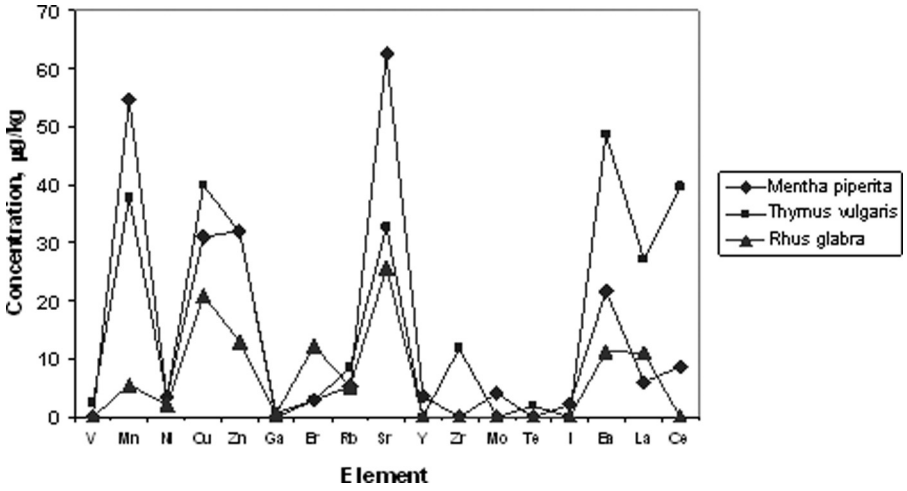

FIGURE 6 **Comparison diagram of minor elements in the samples.**

CONCLUSIONS

Mentha piperita, *Thymus vulgaris*, and *Rhus glabra* are the most important taste materials of Turkish foods. The chemical results of these spice samples reveal that they are rich in beneficial elements for human health. *The significant concentration of K, Zn, Al, and Fe is effect on the human body*. The results of these analyses show that Mentha piperita has a remarkable concentration of K, Zn, and P, which has a great effect on the development of the human body.

The PEDXRF measurement is fast, practical, useful, practically easy, and nondestructive, and is suitable for the analysis of elemental compositions of the samples. As a result, the PEDXRF technique is recommended for the routine analysis of many kinds of the plant samples.

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REFERENCES

1. Ristory, C. A.; dos Santos Pereira, M. A.; Gelli, D. M. *Food Control* **2007**, *18*, 268.
2. Üstündağ, Z.; Kalfa, O. M.; Erdoğan, Y.; Kadıoğlu, Y. K. *Nucl. Instrum. Methods Phys. Res. B* **2006**, *251*, 213.

3. Zararsiz, A.; Kirmaz, R.; Önertoy, Ş.; Arikan, P. *J. Radioanal. Nucl. Chem.* **1997**, *221*, 173.
4. Küçükönder, A.; Çam, H.; Küçükönder, E.; Söğüt, Ö. *J. Quant. Spectrosc. Radiat. Transfer* **2003**, *77*, 329.
5. Lagarde, G.; Larcher, J. *J. Radioanal. Chem. Lett.* **1986**, *105*, 341.
6. George, R. *J. Radioanal. Nucl. Chem.* **1984**, *83*, 135.
7. Custo, G.; Boeykens, S.; Dawidowski, L.; Fox, L.; Gómez, D.; Luna, F.; Vázquez, C. *Anal. Sci.* **2005**, *21*, 751.
8. Üstündağ, İ.; Üstündağ, Z.; Kalfa, O. M.; Kadıoğlu, Y. K. *Nucl. Instrum. Methods Phys. Res. B* **2007**, *254*, 153.
9. Kurahashi, M.; Mizutani, J.; Hioki, A. *Anal. Sci.* **2005**, *21*, 827.
10. Misra, N. L.; Mudher, K. D. S. *Progr. Crystal Growth Characteriz. Mater.* **2002**, *45*, 65.
11. Carvalho, M. L.; Pimentel, A. C.; Fernandes, B. *Anal. Sci.* **2005**, *21*, 747.
12. Ekinci, N.; Ekinci, R.; Şahin, Y. *J. Quant. Spectrosc. Radia. Transfer* **2002**, *74*, 783.
13. Tıraşoğlu, E.; Çevik, U.; Ertuğral, B.; Apaydin, G.; Baltaş, H.; and Ertuğrul, M. *J. Quantitative Spectros. Radiat. Transfer* **2005**, *94*, 181.
14. Doğan, O.; Tıraşoğlu, E. J. *Quant. Spectrosc. Radiat. Transfer* **2006**, *101*, 141.
15. Çevik, U.; Ergen, E.; Budak, G.; Karabulut, A.; Tıraşoğlu, E.; Apaydin, G.; Kopya, A. I. *J. Quant. Spectrosc. Radiat. Transfer* **2003**, *78*, 409.
16. Aslan, A.; Budak, G.; Tıraşoğlu, E.; Karabulut, A. *J. Quant. Spectrosc. Radiat. Transfer.* **2006**, *97*, 10.
17. Jayasekera, R.; Freitas, M. C.; Araújo, M. F. *J. Trace Elements Med. Biol.* **2004**, *17*, 221.
18. Al-Bataina, B. A.; Maslat, A. O.; Al-Kofahi, M. M. *J. Trace Elements Med. Biol.* **2003**, *17*, 85.
19. Queralt, I.; Ovejero, M.; Carvalho, M. L.; Marques, A. F.; Llabrés, J. M. *X-ray Spectrom.* **2005**, *34*, 213.
20. Perring, L.; Andrey, D.; Basic-Dvorzak, M.; Blanc, J. *J. Agri. Food Chem.* **2005**, *53*, 4696.
21. Stephens, W. E.; Calder, W. E. *Anal. Chim. Acta* **2004**, *527*, 89.