



## Determination of Mineral Content in Indian Spices by ICP-OES

S. KUMARAVEL<sup>1</sup> and K. ALAGUSUNDARAM<sup>2</sup>

<sup>1</sup>Research Scholar, Annamalai University, Chidambaram & Scientist, Department of Food Safety & Quality Testing, Indian Institute of Crop Processing Technology, Thanjavur- 613005, India.

<sup>2</sup>Director, Indian Institute of Crop Processing Technology, Thanjavur- 613005, India.

<http://dx.doi.org/10.13005/ojc/300231>

(Received: March 01, 2014; Accepted: April 01, 2014)

### ABSTRACT

Vegetarian and non vegetarian Indian diet consists of various cereals and vegetables along with spices, often used in the preparation of curries. The nutritive potential of trace element has been evaluated using Inductively coupled plasma optical emission spectrometry (ICP-OES). In this study, 15 trace elements like As, Hg, Se, Zn, P, Pb, Cd, Fe, Mn, Cr, Mg, Cu, C, Na and K have been determined in Five Indian popular spices. The result shows that the spice Aniseed contains Phosphorous ( $2027.10 \pm 14.0$  mg/kg), Iron ( $5.40 \pm 2.0$  mg/kg), Magnesium ( $270.10 \pm 7.0$  mg/kg), Calcium ( $602.8 \pm 4.0$  mg/kg), Sodium ( $365.10 \pm 3.0$  mg/kg) and Potassium ( $887.80 \pm 11.0$  mg/kg). The spice Poppy seeds showed Phosphorous content of ( $3980.0 \pm 13.5$  mg/kg), Iron ( $5.475 \pm 2.5$  mg/kg), Magnesium ( $287.20 \pm 14.0$  mg/kg), Calcium ( $690.50 \pm 4.0$  mg/kg), Sodium ( $81.16 \pm 5.0$  mg/kg) and Potassium ( $746.70 \pm 6.0$  mg/kg). The spice Cloves showed that it contains Phosphorous ( $6355.0 \pm 20.0$  mg/kg), Iron ( $1.699 \pm 0.2$  mg/kg), and Potassium ( $318.01 \pm 5.0$  mg/kg). The spice Ajwain seeds showed the presence of Phosphorous ( $1764.0 \pm 16.0$  mg/kg), Iron ( $17.87 \pm 8.0$  mg/kg), Manganese ( $5.729 \pm 2.6$  mg/kg) and Calcium ( $1353.0 \pm 10.0$  mg/kg), The spice Fenugreek seeds showed that it contains Phosphorous ( $2950.0 \pm 17.5$  mg/kg) and Potassium ( $124.82 \pm 2.0$  mg/kg). Heavy metals As and Hg are absent in all five spices. Heavy metals Cr, Cd and Pb are absent in Cloves, Fenugreek and Ajwain species. The results showed that the spice powder is a good source of calcium, potassium, magnesium, iron and phosphorous.

**Key words:** Indian Spices, ICP-OES, Minerals, food quality, Quantitative analysis.

### INTRODUCTION

Spices refer to all of the edible parts of a plant used for flavoring or coloring foods, including fruit, seed, root, bark or vegetable substance<sup>1</sup>. Some are often used as preservative against the action of harmful bacteria or prevents their growth<sup>2</sup>. The specific uses of spices tend to vary considerably among cultures and countries, medicine, religious rituals, cosmetics, perfumery and in foods. As food,

they have been shown to play an important role in health partially as sources of nutrients<sup>3, 4</sup>.

The Indian vegetarian diet primarily consists, typically of bread baked at home, boiled rice and curries prepared from pulses (lentils) and vegetables, including spices added during cooking. Though bread is often made from wheat flour, other cereals, such as maize, barley, millet and sorghum, are also used, depending on the region or socio-

economic status. Indian curry is spiced with salt, red chillies and other spices, such as turmeric & coriander powder, depending on individual taste and other considerations. Besides, condiments such as cloves, black pepper, cardamom and bay leaves are also used to spread over cooked/fried rice and curry preparations.

Several workers have discussed the constituents of these spices in terms of their volatile oils and other vitamin contents<sup>5,6</sup> and have reported various characteristics for determination of chemical and toxic constituents of Indian spices. Ila and Jagam<sup>7</sup> reported 24 elements in six Indian spices. Abou-Arab and Abou-Donia<sup>8</sup> have determined heavy metal contaminants in Egyptian spices.

The used methodologies had been either time consuming and costly, or importantly did not allow simultaneous estimation of the micro minerals concerned. Inductively coupled plasma optical emission spectrometry (ICP-OES) is well established as a method for multi elemental analysis and the determination of isotope ratios<sup>9</sup> and overcomes many of these problems. This methodology allows simultaneous analysis of a wide range of trace elements in the same sample and has been used in this study.

The objective of the present study was the analysis of trace element (As, Hg, Se, Zn, P, Pb, Cd, Fe, Mn, Cr, Mg, Cu, Ca, Na and K) levels in the dehydrated powder of Indian spice powder using inductively coupled plasma optical emission spectrometry (ICP-OES).

## MATERIAL AND METHODS

The analysis of minerals in the following spices were used in this study Aniseed (*Pimpinella anisum*), Poppy seed (*Papaver somniferum*), Cloves (*Syzygium aromaticum*) Fenugreek seeds (*Trigonella foenum-graecum*) and Ajwain (*Trachyspermum ammi*) powder samples and they were obtained from a local supermarket and tested by using ICP-OES method (10). All minerals and heavy metals measurements were carried out using the Perkin Elmer optima ICP- OES (Model: OPTIMA 2000DV, serial number: 080N3041701) and the ICP-OES operating conditions are listed in table 1.

## Reagents and chemicals

Analytical reagents-grade chemicals were used in the preparation of all solutions. All the plastic and glassware were cleaned by soaking in dilute nitric acid (1 + 9) and were rinsed with distilled water prior to use. Nitric acid (65%) and hydrogen peroxide (30%) were supplied by Merck (India)

## Sample preparation

The samples were digested using microwave digestion method (Model: ETHOS One, Make – Milestone). The samples of approximately 1.0 g were digested with 6 ml of HNO<sub>3</sub> and 2 ml of H<sub>2</sub>O<sub>2</sub> in microwave digestion system. The samples and acid mixture are placed in suitably inert polymeric microwave vessels. The vessel is sealed and heated in the microwave digestion system. The temperature program was as follows: 2 min for 400 w, 6 min for 400 w, 5 min for 400 w, 8 min for 800 w and 8 min for vent. The resulting solutions were cooled and diluted to 10 ml with distilled water. The determination of metal contents in this clear solution was carried out by Inductively coupled plasma-optical emission spectrometry (ICP-OES).

## ICP-OES (Inductively Coupled Plasma-Optical Emission Spectrometry) Analysis of Samples

All samples were analyzed in triplicates by ICP-OES Perkin-Elmer; model Optima™ 2000 DV, using winLab32 software for the analysis. The analytical measurements were made with a simultaneous Perkin-Elmer ICP OES, model optima 2000DV, winLab32™, version 7.0 software equipped with a peristaltic pump, a cross-flow nebulizer (coupled to a ryton double pass spray chamber) and a ceramic central torch tube injector with an internal diameter of 2 mm. The operating parameters are listed in Table 1. The wavelengths, measurement parameters and standards for each element are given in Table 2.

## Analysis of certified reference material (CRM) of Minerals and calibration

Aliquots of ICP multielement standard solution (10 to 50 mg/L Merck) containing the elements such as (As, Hg, Se, Zn, P, Pb, Cd, Fe, Mn, Cr, Mg, Cu, Ca, Na and K) were used in the preparation of calibration solutions. Working standard solutions were prepared by dilution of the stock standard solutions to desired concentration

in 1% HNO<sub>3</sub>. The ranges of the calibration curves (5 points) were selected to match the expected concentrations for all the elements of the sample studied by ICP-OES. The correlation coefficient *r*<sup>2</sup> obtained for all cases was 0.9999

**RESULTS AND DISCUSSIONS**

The elements such as Arsenic (As),Mercury (Hg),Selenium (Se) ,Zinc (Zn) , Phosphorous (P) , Lead (pb),Cadmium (Cd),Iron (Fe), Manganese (Mn) ,Chromium (Cr),Magnesium

(Mg) ,Copper (Cu) Calcium (Ca),Sodium (Na) and Potassium (K) were analyzed and results given in Table 3 The result shows that the Aniseed contain Selenium (2.86±2.0 mg/kg), Zinc (1.110±0.5mg/kg), Phosphorous (2027.10±14.0 mg/kg), Iron (5.40±2.0 mg/kg), Manganese (3.140±1.2 mg/kg), Magnesium (270.10±7.0mg/kg), Calcium (602.8±4.0 mg/kg), Sodium (365.10±3.0 mg/kg) and Potassium (887.80±11.0 mg/kg). This spice has removes excess mucus in the gastrointestinal area. It is good for improving memory, colds, flu, cough, bronchitis, sinusitis, gas, colic, tension,

**Table 1. Instrumental Conditions and Parameters Parameter Setting**

<b>Nebulizer</b>	<b>Bergener PEEK Mira Mist</b>
Spray Chamber	Cyclonic
RF Power	1300 W
Sample Injector	2.0 mm i.d. Alumina
Nebulizer Argon Flow	0.80 L/min
Sample Uptake Rate	1.80 mL/min
Auto Integration	5 – 20 seconds (Min-Max)
Data Processing Mode	Peak Area
Read Delay	60 seconds
Rinse Delay	20 seconds

**Table 2: Wavelengths, Measurement Parameters and Standards for each Element**

<b>Element</b>	<b>Wave length (nm)</b>	<b>Plasma (L/min)</b>	<b>Aux (L/min)</b>	<b>Neb (L/min)</b>	<b>power</b>	<b>View Mode</b>	<b>Calibration (mg/Kg)</b>
Arsenic (As)	188.979	15	0.2	0.8	1300	Axial	10 - 50
Mercury (Hg)	194.168	15	0.2	0.8	1300	Axial	10 - 50
Selenium (Se)	196.026	15	0.2	0.8	1300	Axial	10 - 50
Zinc (Zn)	206.200	15	0.2	0.8	1300	Axial	10 - 50
Phosphorous (P)	213.617	15	0.2	0.8	1300	Radial	10 - 50
Lead (pb)	220.353	15	0.2	0.8	1300	Axial	10 - 50
Cadmium (Cd)	228.802	15	0.2	0.8	1300	Axial	10 - 50
Iron (Fe)	238.204	15	0.2	0.8	1300	Radial	10 - 50
Manganese (Mn)	257.610	15	0.2	0.8	1300	Radial	10 - 50
Chromium (Cr)	267.716	15	0.2	0.8	1300	Axial	10 - 50
Magnesium (Mg)	280.271	15	0.2	0.8	1300	Radial	10 - 50
Copper (Cu)	327.393	15	0.2	0.8	1300	Axial	10 - 50
Calcium (Ca)	393.366	15	0.2	0.8	1300	Radial	10 - 50
Sodium (Na)	589.592	15	0.2	0.8	1300	Radial	10 - 50
Potassium (K)	766.490	15	0.2	0.8	1300	Radial	10 - 50

Table 3: Mineral content estimation in Indian spice extracts by ICP-OES

Element with Wave length	Wave length (nm)	Results mg/kg of spice powder (dry basis)							Detection Limit
		Aniseed	Poppy Seed	Cloves	Fenugreek	Ajwain			
Arsenic (As)	188.979	BDL	BDL	BDL	BDL	BDL	BDL	BDL	1-10
Mercury (Hg)	194.168	BDL	BDL	BDL	BDL	BDL	BDL	BDL	1-10
Selenium (Se)	196.026	2.86±2.0	0.013	0.071	0.060	0.015	0.060	0.015	1-10
Zinc (Zn)	206.200	1.110±0.5	2.575±1.0	0.099	2.239±0.3	BDL	2.239±0.3	BDL	0.1-1
Phosphorous (P)	213.617	2027.10±14.0	3980.0±13.5	6355.0±20.0	2950.0±17.5	1764.0±16.0	2950.0±17.5	1764.0±16.0	1-10
Lead (pb)	220.353	0.215±0.08	0.184±0.05	BDL	BDL	BDL	BDL	BDL	1-10
Cadmium (Cd)	228.802	0.006	0.007	BDL	BDL	BDL	BDL	BDL	<0.1
Iron (Fe)	238.204	5.40±2.0	5.475±2.5	1.699±0.2	4.094±1.0	17.87±8.0	4.094±1.0	17.87±8.0	<0.1
Manganese (Mn)	257.610	3.140±1.2	3.848±1.8	1.839±0.6	0.670±0.1	5.729±2.6	0.670±0.1	5.729±2.6	0.1-1
Chromium (Cr)	267.716	0.156±0.05	0.114±0.05	BDL	BDL	BDL	BDL	BDL	0.1-1
Magnesium (Mg)	280.271	270.10±7.0	287.20±14.0	70.88±4.2	89.53±5.5	112.30±11.0	89.53±5.5	112.30±11.0	<0.1
Copper (Cu)	327.393	1.933±0.5	2.581±0.8	0.195±0.5	0.324±0.1	0.729±0.3	0.324±0.1	0.729±0.3	0.1-1
Calcium (Ca)	393.366	602.8±4.0	690.50±4.0	243.20±2.0	108.0±1.0	1353.0±10.0	108.0±1.0	1353.0±10.0	<0.1
Sodium (Na)	589.592	365.10±3.0	81.16±5.0	45.22±1.5	123.10±2.6	228.47±2.0	123.10±2.6	228.47±2.0	0.1-1
Potassium (K)	766.490	887.80±11.0	746.70±6.0	318.01±5.0	124.82±2.0	90.43±1.0	124.82±2.0	90.43±1.0	0.1-1

\* BDL - Below Detection Limit

lactation (*not in pregnancy*), eye brightener and fresh breath. Potassium is an important component of cell and body fluids that helps to control heart rate and blood pressure. The Poppy seeds composed of Zinc ( $2.575 \pm 1.0$  mg/kg), Phosphorous ( $3980.0 \pm 13.5$  mg/kg), Iron ( $5.475 \pm 2.5$  mg/kg), Magnesium ( $287.20 \pm 14.0$  mg/kg), Calcium ( $690.50 \pm 4.0$  mg/kg), Sodium ( $81.16 \pm 5.0$  mg/kg) and Potassium ( $746.70 \pm 6.0$  mg/kg). Magnesium is the sixth most abundant mineral and last of the major minerals. It is a small, mobile and strongly electropositive divalent cation in the plants, found both in bound as well as free form(11). The Cloves seeds showed mineral content of Phosphorous ( $6355.0 \pm 20.0$  mg/kg), Iron ( $1.699 \pm 0.2$  mg/kg), Magnesium ( $70.88 \pm 4.2$  mg/kg), Calcium ( $243.20 \pm 2.0$  mg/kg), Sodium ( $45.22 \pm 1.5$  mg/kg) and Potassium ( $318.01 \pm 5.0$  mg/kg). Internally, cloves are also good for pain relief, nausea, vomiting and digestive problems. They may be chewed for toothaches and eating cloves is said to be an aphrodisiac. Cloves are effective in killing malaria, tuberculosis, cholera, scabies and bacteria, parasites, viruses and fungi including Candida. Cloves also destroy all species of shigella, staphylococcus and streptococcus.

The Ajwain seeds composed of Phosphorous ( $1764.0 \pm 16.0$  mg/kg), Iron ( $17.87 \pm 8.0$  mg/kg), Magnesium ( $5.729 \pm 2.6$  mg/kg), Calcium ( $1353.0 \pm 10.0$  mg/kg), Sodium ( $228.47 \pm 2.0$  mg/kg) and Potassium ( $90.43 \pm 1.0$  mg/kg). The cloves showed the composition of Phosphorous ( $2950.0 \pm 17.5$  mg/kg), Iron ( $4.094 \pm 1.0$  mg/kg), Magnesium ( $89.53 \pm 5.5$  mg/kg), Calcium ( $108.0 \pm 1.0$  mg/kg), Sodium ( $123.10 \pm 2.6$  mg/kg) and Potassium

( $124.82 \pm 2.0$  mg/kg). Guerinot and Ying (12) reported that Fe plays a role in rib nucleotide dinitrogen reduction and energy yielding electron transfer chain.

## CONCLUSIONS

From the results, it is seen that elements like As, Hg, Se, Zn, P, Pb, Cd, Fe, Mn, Cr, Mg, Cu, Ca, Na and K were successfully estimated in the Indian spice powder samples by using ICP-OES with microwave digestion procedures. The use of ICPOES provides a simpler, effective, faster, and less contamination procedure of determining the quality of spice extracts. The results show that spice powder is a good source of calcium sodium, potassium, magnesium, selenium and Phosphorous and spice was a very important human nutrient since their consumption has increased in recent years. Heavy metals in spice are a problem for human consumption and hence a detailed study of minerals in spices is needed. Heavy metals As and Hg are absent in all five spices. Heavy metals Cd, Cr and Pb are absent in Fenugreek, Cloves and Ajwain spices.

## ACKNOWLEDGEMENTS

Special thanks to Dr. K. Alagusundaram, Director, Indian Institute of Crop Processing Technology (IICPT), Ministry of Food Processing Industries, Govt. of India, Thanjavur-613 005 TamilNadu, for the support of this study and guidance throughout the work.

## REFERENCES

1. U. R. Susheela, "Handbook of Spices, Seasoning, and Flavorings," TECHNOMIC Publishing Co., Inc., Lancaster, 2000; 329.
2. J. Bitting and P. W. Sherman, "Antimicrobial Functions of Spices—Why Some Like It Hot," *Quarterly Review of Biology*, **1998**; *73*(1): 3-49. doi:10.1086/420058
3. A. A. Barakat, A. O. Maslat and M. M. AL-Kofahi, "Element Analysis and Biological Studies on Ten Oriental Spices Using XRF and Ames Test," *Journal of Trace Element Medicine Biology*, 2003; **17**(2): 85-90. doi:10.1016/S0946-672X(03)80003-2
4. H. R. H. Takruri and A. F. M. Dameh, "Study of the Nutritional Value of Black Cumin Seeds (*Nigella sativa* L)," *Journal of the Science of Food and Agriculture*, 1998; **76**(3): 404-410. doi:10.1002/(SICI)1097-0010(199803)76:3<404::AID-JSFA964>3.0.CO;2-L

5. Gopalan, C., Ramasastry, B. V., Balasubramanian, S. C., Narsinagarao, B. S., Deosthale, Y. G., & Pant, K. C. Nutritive value of Indian foods (p. 156). Hyderabad, India: National Institute of Nutrition **1999**.
6. Pruthi, J. S. Quality assurance in spices and spice products, modern methods of analysis. New Delhi: Allied Publishers Ltd. **1999**.
7. Ila, P., & Jagam, P. Multielement analysis of food spices by instrumental neutron activation analysis. *Journal of Radioanalytical Chemistry*, **1980**; *57*: 205–210.
8. Abou-Arab, A. A. K., & Abou Donia, M. A. Heavy metals in Egyptian spices and Medicinal plants and the effect of processing on their levels. *Journal of Agricultural Food Chemistry*, **2000**; *48*: 2300–2304.
9. A.R.Date and Gray, A. L. Development progress in *plasma source mass spectrometry*. *Analyst*, **1983**; *105*(1283): 159-165.
10. Dilek Bakircioglu, Yasemin Bakircioglu Kurtulus and Gokhan Ucar. . Determination of some traces metal levels in cheese samples packaged in plastic and tin containers by ICP-OES after dry, wet and microwave digestion *Food and Chemical Toxicology* **2011**; *49*: 202–207.
11. Gilbert, F. A. Mineral nutrition and the balance of life. University of Oklahoma Press, Norman. 1957; 350.
12. Guerinot, M. L. and Ying, Y. Iron: nutritious, noxious, and not readily available. *Plant Physiol.*, **1994**; *104*: 815–820.