

## Evaluation of trace element contents of powdered beverages from Turkey

MUSTAFA TUZEN – SIBEL SARACOGLU – MUSTAFA SOYLAK

### Summary

Trace element contents of powdered beverages from Turkey were determined by atomic absorption spectroscopy after microwave digestion. Verification of the method was demonstrated by analysis of standard reference material. Trace element contents in powdered beverage samples were 0.10–1.33 mg.kg<sup>-1</sup> for copper, 0.22–1.90 mg.kg<sup>-1</sup> for manganese, 0.39–4.19 mg.kg<sup>-1</sup> for iron, 0.22–4.54 mg.kg<sup>-1</sup> for zinc, 0.11–0.47 mg.kg<sup>-1</sup> for selenium, 3.21–71.5 µg.kg<sup>-1</sup> for chromium, 20.9–80.1 µg.kg<sup>-1</sup> for aluminium and 6.12–217 µg.kg<sup>-1</sup> for nickel. The results were compared with literature data and levels set by legislative documents.

### Keywords

trace element; powdered beverage; atomic absorption spectroscopy; evaluation

Trace heavy metal analysis is an important part of public health studies due to well documented negative effects of heavy metal ions on the human metabolism [1, 2]. Some transition metals at trace levels play a positive role in human physiology. Heavy metals normally occurring in nature are not harmful to the environment, because they are only present at low levels. However, if their levels increase, their role may become negative. For this reason, environmental contamination by traces of heavy metals caused by human activities including industry, traffic etc. has been extensively studied [3, 4]. One of the main sources of heavy metal ions is also food [5–7] and the analysis of food samples for trace heavy metal contents has been performed [8–11]. However, there is only a limited information on trace element contents in powdered beverages from Turkey.

The reliability of trace heavy metals determination in complex matrices mainly depends on the dissolution process used. Both the wet and dry ashing procedures are time consuming. In recent years, microwave digestion procedures in closed vessels have been developed as a rapid and reproducible sample preparation method for a great variety of complex matrices [3, 12]. For analysis, flame atomic absorption spectrometry is the most widely used technique. Lower concentrations are

determined using graphite furnace atomic absorption spectrometry [13].

In the present study, the contents of trace elements in powdered beverage samples produced in Turkey were determined by flame and/or graphite furnace atomic absorption spectrometry (AAS) after microwave digestion.

## MATERIAL AND METHODS

### Sampling

Nineteen different powdered beverage samples of four brands were purchased from local markets in Kayseri and Tokat, Turkey during 2006. The samples were dried at 105 °C for 24 h. Dried samples were homogenized in an agate mortar and stored in polyethylene bottles until analysis.

### Reagents

All reagents were of analytical reagent grade unless otherwise stated. Double distilled deionized water Milli-Q (Millipore, Massachusetts, USA) of 18.2 MΩ.cm<sup>-1</sup> resistivity was used for all dilutions. HNO<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>, and HCl were of suprapure quality (Merck, Darmstadt, Germany). All the plasticware and glassware were cleaned by soaking in dilute HNO<sub>3</sub> (1/9, v/v) and were rinsed with distilled

**Mustafa Tuzen**, Gaziosmanpasa University, Faculty of Science and Arts, Chemistry Department, 60250 Tokat, Turkey.

**Sibel Saracoglu**, Erciyes University, Faculty of Education, 38039 Kayseri, Turkey.

**Mustafa Soylak**, Erciyes University, Faculty of Art and Science, Department of Chemistry, 38039 Kayseri, Turkey.

*Correspondence author:*

Sibel Saracoglu, tel./fax: + 90 352 4378834, e-mail: saracs@erciyes.edu.tr

water prior to use. The standard solutions of elements used for calibration were produced by diluting of a stock solution of 1000 mg.l<sup>-1</sup> of the given element supplied by Sigma Chemical (St. Louis, Missouri, USA).

#### Atomic absorption spectrometry

A Perkin Elmer Analyst 700 atomic absorption spectrometer equipped with HGA graphite furnace and with deuterium background corrector (Shelton, Connecticut, USA) was used. Zinc and iron were determined in air-acetylene flame. The operating parameters for individual elements were set as recommended by the manufacturer. For flame measurements, a 10 cm long slot-burner head, a lamp and an air-acetylene flame were used. Other elements were determined in graphite furnace. For graphite furnace measurements, argon was used as inert gas. Pyrolytic-coated graphite tubes (Perkin Elmer part No. B3 001264) with a platform were used. Samples were injected into the graphite furnace using Perkin Elmer AS-800 autosampler. The atomic absorption signal was measured as a peak area and peak height mode against an calibration curve.

#### Microwave digestion

Milestone Ethos D microwave (Sorisole, Italy) closed system (maximum pressure, 1.10<sup>7</sup> Pa; maximum temperature, 300 °C) was used for digestion of the samples. One gram of the sample was digested with 6 ml of concentrated HNO<sub>3</sub> and 2 ml of concentrated H<sub>2</sub>O<sub>2</sub> in the microwave digestion system and diluted to 10 ml with double distilled deionized water. A blank digest was carried out in the same way (digestion conditions for microwave system were applied as 2 min for 250 W, 2 min for 0 W, 6 min for 250 W, 5 min for 400 W, 8 min for 550 W, vent: 8 min, respectively).

The accuracy of the entire method involving microwave digestion and atomic absorption was verified by the analysis of certified reference materials (NIST SRM 1573a Tomato leaves).

## RESULTS AND DISCUSSION

Trace metal contents were analysed in quartet and were expressed on dry weight basis. Relative standard deviations were less than 10% and the student T-test was used to determine significant differences between mean values. Both the detection limit (based on three times the standard deviations of the reagent blank) and the characteristic masses (based on 0.0044 absorbance) were calculated for the investigated analyte ions. The

detection limit values of the investigated elements for flame AAS were found to be 0.013 mg.l<sup>-1</sup> for Cu, 0.019 mg.l<sup>-1</sup> for Zn, 0.011 mg.l<sup>-1</sup> for Fe, and 0.010 mg.l<sup>-1</sup> for Mn. The characteristic mass values were Ni: 20 pg, Al: 17 pg, Cr: 15 pg and Se: 22 pg in graphite furnace AAS.

An accuracy of 95–98% was demonstrated for our method by means of trace metal determination in the standard reference material, within 95% confidence levels. The results for iron, copper, manganese, zinc, chromium, selenium, aluminium and nickel, which had been chosen as representative trace metals for environmental pollution, are shown in Tab. 1. A summary of trace element contents found in the analysed samples is given in Tab. 2. The contents of investigated trace elements in powdered beverage samples were found to be in the range of 0.10–1.33 mg.kg<sup>-1</sup> for copper, 0.22–1.90 mg.kg<sup>-1</sup> for manganese, 0.39–4.19 mg.kg<sup>-1</sup> for iron, 0.22–4.54 mg.kg<sup>-1</sup> for zinc, 0.11–0.47 mg.kg<sup>-1</sup> for selenium, 3.21–71.5 µg.kg<sup>-1</sup> for chromium, 20.9–80.1 µg.kg<sup>-1</sup> for aluminium and 6.12–217 µg.kg<sup>-1</sup> for nickel.

The lowest and highest copper levels in powdered beverage samples were found as 0.10 mg.kg<sup>-1</sup> in sour cherry (brand A and C) and apple (brand B) samples, and 1.33 mg.kg<sup>-1</sup> in the orange sample (Brand A). In the literature, copper levels have been reported in the range of 0.25–3.94 mg.kg<sup>-1</sup> in Nigerian foods [8], 0.2–3.1 mg.kg<sup>-1</sup> in Brazil foods [14]. Our copper values are in agreement with literature values. The maximum copper level permitted for food is 5 mg.kg<sup>-1</sup> according to Turkish Food Codex [15]. Copper levels in the analysed powdered beverage samples were found to be lower than the legal limits. Copper is known to be vital and toxic for many biological systems. It may enter the food materials from soil through mineralization by crops, food processing or environmen-

**Tab. 1.** Trace element contents in certified reference material (NIST SRM 1573a Tomato leaves).

Element	Certified value [mg.kg <sup>-1</sup> ]	Our value [mg.kg <sup>-1</sup> ]	Recovery [%]
Cu	4.7	4.60 ± 0.25	98
Mn	246	236.2 ± 10.5	96
Fe	368	356.9 ± 21.6	97
Zn	30.9	29.4 ± 1.2	95
Se	0.054	0.052 ± 0.005	96
Cr	1.99	1.93 ± 0.15	97
Al	598	568.1 ± 23.6	95
Ni	1.59	1.53 ± 0.13	96

*n* = 4.

**Tab. 2.** Trace element contents in analysed powdered beverage samples from Turkey.

Samples		Cu [ mg.kg <sup>-1</sup> ]	Mn [ mg.kg <sup>-1</sup> ]	Fe [ mg.kg <sup>-1</sup> ]	Zn [ mg.kg <sup>-1</sup> ]	Se [ mg.kg <sup>-1</sup> ]	Cr <sup>a</sup> [ μg.kg <sup>-1</sup> ]	Al <sup>a</sup> [ μg.kg <sup>-1</sup> ]	Ni <sup>a</sup> [ μg.kg <sup>-1</sup> ]
Brand A	Lemon	0.81 ± 0.07	0.44 ± 0.03	2.91 ± 0.25	1.12 ± 0.10	0.47 ± 0.04	37.5 ± 2.1	80.1 ± 5.2	162 ± 10
	Peach	0.98 ± 0.06	0.84 ± 0.07	3.20 ± 0.30	1.03 ± 0.10	0.13 ± 0.01	3.21 ± 1.3	26.3 ± 2.1	148 ± 12
	Orange	1.33 ± 0.12	0.86 ± 0.08	3.28 ± 0.27	1.48 ± 0.13	0.18 ± 0.01	48.4 ± 3.5	27.6 ± 2.4	217 ± 15
	Sour cherry	0.10 ± 0.01	1.90 ± 0.15	2.55 ± 0.24	0.22 ± 0.02	0.11 ± 0.01	21.9 ± 2.1	50.8 ± 4.3	34.6 ± 2.3
Brand B	Apple	0.10 ± 0.01	1.82 ± 0.17	1.41 ± 0.12	1.02 ± 0.10	0.11 ± 0.01	4.76 ± 3.5	42.5 ± 2.4	19.5 ± 1.1
	Mandarin	1.09 ± 0.10	1.03 ± 0.10	2.40 ± 0.21	3.56 ± 0.28	0.18 ± 0.02	45.7 ± 4.1	33.5 ± 2.6	102 ± 8
	Lemon	0.45 ± 0.04	0.30 ± 0.03	0.39 ± 0.03	0.58 ± 0.05	0.17 ± 0.01	6.28 ± 5.2	35.5 ± 1.9	121 ± 7
	Tropic	0.62 ± 0.05	0.92 ± 0.07	1.47 ± 0.10	0.48 ± 0.04	0.15 ± 0.01	17.4 ± 1.4	44.6 ± 3.6	27.9 ± 2.3
	Peach	0.95 ± 0.08	0.62 ± 0.05	2.18 ± 0.16	1.71 ± 0.16	0.16 ± 0.01	35.7 ± 3.2	31.1 ± 1.5	121 ± 9
Brand C	Sour cherry	0.10 ± 0.01	0.29 ± 0.02	1.67 ± 0.15	1.21 ± 0.11	0.23 ± 0.02	21.7 ± 2.1	34.2 ± 2.5	91.2 ± 7.5
	Orange	1.21 ± 0.11	0.38 ± 0.03	2.83 ± 0.25	4.54 ± 0.33	0.24 ± 0.02	71.5 ± 5.5	29.6 ± 2.1	11.0 ± 1.1
	Lemon	0.47 ± 0.03	0.22 ± 0.02	1.66 ± 0.13	0.91 ± 0.07	0.25 ± 0.02	47.4 ± 4.2	22.8 ± 1.7	6.12 ± 0.5
Brand D	Orange	0.35 ± 0.03	0.55 ± 0.05	0.76 ± 0.07	1.34 ± 0.11	0.20 ± 0.02	18.4 ± 1.4	33.9 ± 1.9	63.1 ± 5.6
	Mandarin	0.33 ± 0.02	0.53 ± 0.04	2.04 ± 0.12	1.63 ± 0.15	0.30 ± 0.03	35.6 ± 3.2	24.8 ± 2.1	90.3 ± 7.1
	Sour cherry	0.40 ± 0.03	0.95 ± 0.08	2.81 ± 0.26	2.69 ± 0.22	0.15 ± 0.01	30.8 ± 2.5	32.4 ± 2.8	38.8 ± 3.7
	Mixture	0.47 ± 0.03	1.02 ± 0.10	4.19 ± 0.33	1.22 ± 0.10	0.17 ± 0.02	31.6 ± 2.1	20.9 ± 1.4	87.8 ± 7.3
	Strawberry	0.33 ± 0.03	0.37 ± 0.03	1.10 ± 0.10	1.06 ± 0.10	0.22 ± 0.02	18.1 ± 1.6	21.8 ± 1.6	134 ± 11
	Lemon	0.46 ± 0.04	0.77 ± 0.06	1.81 ± 0.16	0.53 ± 0.05	0.23 ± 0.02	48.3 ± 3.5	31.7 ± 1.8	138 ± 12
	Peach	0.33 ± 0.03	0.52 ± 0.05	0.90 ± 0.08	1.10 ± 0.10	0.17 ± 0.01	24.7 ± 2.2	39.9 ± 3.6	57.2 ± 5.3

n = 4.

tal contamination. For instance, in the application of agricultural practices, copper-based pesticides are in common use in farms in some countries [8]. FAO/WHO has set a limit for heavy metal intake based on body weight. For an average adult (60 kg body weight), the provisional tolerable daily intake (PTDI) for iron, copper and zinc are 48 mg, 3 mg and 60 mg, respectively [16]. The lower manganese content was found as 0.22 mg.kg<sup>-1</sup> in lemon (brand C) while the highest manganese content was 1.90 mg.kg<sup>-1</sup> in sour cherry (Brand A). There is limited information about manganese contents of powdered beverage. There is no information about maximum manganese levels in powdered beverage samples in Turkish standards [15]. The US National Academy of Sciences recommends 2.5–5 mg per day manganese [17] and, WHO recommends 2–9 mg per day for an adult [18].

The lowest and highest iron levels in powdered beverage samples were found as 0.39 mg.kg<sup>-1</sup> in lemon (brand B) and 4.19 mg.kg<sup>-1</sup> in the mixture powdered beverage samples (brand D). The maximum iron level permitted for food is 15 mg.kg<sup>-1</sup> according to Turkish Food Codex [15]. Iron levels in analysed powdered beverage samples were found to be lower than legal limits. It is known that adequate iron in diet is very important for decreas-

ing the incidence of anemia. Iron deficiency occurs when the demand for iron is high, e.g. in growth, high menstrual loss and pregnancy, and when the intake is quantitatively inadequate or contains elements that render iron unavailable for absorption [19]. Poor bioavailability is considered to be an important factor leading to iron deficiency in many countries.

The lowest and highest zinc levels in powdered beverage samples were found as 0.22 mg.kg<sup>-1</sup> in sour cherry (brand A) and 4.54 mg.kg<sup>-1</sup> in orange (brand C). Zinc contents have been reported in the literature in the range of 0.16–9.00 mg.kg<sup>-1</sup> in Nigerian foods [8]. The maximum zinc level permitted for food is 5 mg.kg<sup>-1</sup> according to Turkish Food Codex [15]. Zinc levels in the analysed powdered beverage samples were found to be lower than legal limits. Zinc is known to be involved in several metabolic pathways in humans and zinc deficiency can lead to loss of appetite, growth retardation, skin changes and immunological abnormalities. Zinc is widespread in living organisms due to its biological significance.

Powdered beverage samples in this study were very rich in selenium. The minimum and maximum selenium levels were found as 0.11 mg.kg<sup>-1</sup> in sour cherry and apple samples (brand A and B)

and  $0.47 \text{ mg.kg}^{-1}$  in lemon samples (brand A). Selenium contents have been reported in the range of  $7.6\text{--}610.7 \text{ }\mu\text{g.kg}^{-1}$  [20];  $1.4\text{--}7.9 \text{ }\mu\text{g.kg}^{-1}$  [21]. No maximum selenium level in powdered beverage samples is set by Turkish standards [15]. The adequate daily dietary selenium intake ranges from 50 to  $200 \text{ }\mu\text{g}$ , with an average value of  $55 \text{ }\mu\text{g}$  for adult humans [22]. Selenium is recognized as an essential micronutrient in animals and humans. Moreover, it plays important biological roles as an antioxidant, as a regulator of thyroid hormone metabolism or as an anti-carcinogenic agent. Insufficient contents of selenium in food may cause anomalies in living organisms, while high contents are toxic.

The lowest chromium contents were  $3.21 \text{ }\mu\text{g.kg}^{-1}$  in peach (brand A), while the highest chromium contents were  $71.5 \text{ }\mu\text{g.kg}^{-1}$  in orange (brand C). Chromium contents have been reported in the literature in the range of  $0.01\text{--}0.09 \text{ mg.kg}^{-1}$  [23]. No maximum chromium level in powdered beverage samples is set by Turkish standards [15]. Chromium is an essential mineral to humans and has been found important for carbohydrate, lipid and protein metabolisms. The recommended daily intake is  $50\text{--}200 \text{ }\mu\text{g}$  [24].

The minimum and maximum aluminium contents in samples were found to be  $20.9 \text{ }\mu\text{g.kg}^{-1}$  in mixture powdered beverage samples (brand D) and  $80.1 \text{ }\mu\text{g.kg}^{-1}$  in lemon samples (brand A). No maximum aluminium level in powdered beverage samples is set by Turkish standards [15]. Aluminium is not considered an essential element in humans. Exposure to aluminium has been implicated in a number of human pathologies including encephalopathy/dialysis dementia, Parkinson disease and Alzheimer's disease [25]. The permissible aluminium dose for an adult is quite high ( $60 \text{ mg}$  per day) [26]. Aluminium is present in food naturally, or it is added via food additives, or Al is used in food preparation and storage [9].

The lowest and highest nickel levels in powdered beverage samples were found to be  $6.12 \text{ }\mu\text{g.kg}^{-1}$  in lemon (brand C) and  $217 \text{ }\mu\text{g.kg}^{-1}$  in orange (brand A). Nickel contents have been reported in the literature in the range of  $0.02\text{--}0.18 \text{ mg.kg}^{-1}$  [27]. No maximum nickel level in powdered beverage samples is set by Turkish standards. WHO recommends  $100\text{--}300 \text{ }\mu\text{g}$  nickel for daily intake [18].

## CONCLUSION

Certain trace metals are very important for human biology. Foods such as fruits, fruit juices and powdered beverage are in general a good dietary

resource, especially in major elements. On the other hand, these are also media through which humans are exposed to various toxic metals. The sources of these metals in powdered beverage could be traced back to sample preparation and package process or environmental metal pollution. The study shows that Cu, Fe and Zn levels in powdered beverages from Turkey were lower than the legal limits.

## ACKNOWLEDGMENTS

The authors are grateful for the financial support of the Unit of the Scientific Research Projects of Gaziosmanpasa University and the Unit of the Scientific Research Projects of Erciyes University.

## REFERENCES

1. Smith, F. E. – Arsenault E. A: Microwave-assisted sample preparation in analytical chemistry. *Talanta*, **43**, 1996, pp. 1207–1268.
2. Tredez, Q.: Design and manufacture of a heavy metal sensor, school of applied sciences. [MSc Individual Project Thesis.] Cranfield: Cranfield University, 2007. 90 pp.
3. Sysalova, J. – Szakova, J.: Mobility assessment and validation of toxic elements in tunnel dust samples–Subway and road using sequential chemical extraction and ICP–OES/GF AAS measurement. *Environmental Research*, **101**, 2006, pp. 287–293.
4. Chen, S. S. – Chen, C. M. – Cheng, C. C. – Chou, S. S.: Determination of copper in edible oils by direct graphite furnace atomic absorption spectrometry. *Journal of Food and Drug Analysis*, **7**, 1999, pp. 207–214.
5. Biego, G. H. – Joyeux, M. – Hartemann, P. – Debry, G.: Daily intake of essential minerals and metallic micropollutants from foods in France. *The Science of the Total Environment*, **217**, 1998, pp. 27–36.
6. Bahemuka, T. E. – Mubofu, E. B.: Heavy metals in edible green vegetables grown along the sites of the Sinza and Msimbazi rivers in Dar es Salaam, Tanzania. *Food Chemistry*, **66**, 1999, pp. 63–66.
7. Kolayli, S. – Kongur, N. – Gundogdu, A. – Kemer, B. – Duran, C. – Aliyazicioglu, R.: Mineral composition of selected honeys from Turkey. *Asian Journal of Chemistry*, **20**, 2008, pp. 2421–2425.
8. Onianwa, P. C. – Adeyemo, A. O. – Idowu, O. E. – Ogabiela, E. E.: Copper and zinc contents of Nigerian foods and estimates of the adult dietary intakes. *Food Chemistry*, **72**, 2001, pp. 89–95.
9. Saiyed, S. M. – Yokel, R. A.: Aluminum content of some foods and food products in the USA, with aluminum food additives. *Food Additives and Contaminants*, **22**, 2005, pp. 234–244.
10. Radwan, M. A. – Salama, A. K.: Market basket survey for some heavy metals in Egyptian fruits and vegetables. *Food and Chemical Toxicology*, **44**, 2006, pp. 1273–1278.

11. Sönmez, N. – Alizadeh, H. H. A. – Öztürk R. – Acar, A. İ.: Some physical properties of gilaburu seed. *Tarım Bilimleri Dergisi*, 13, 2007, pp. 308–311.
12. Soylak, M. – Colak, H. – Tuzen, M. – Turkoglu, O. – Elci, L.: Comparison of digestion procedures on commercial powdered soup samples for the determination of trace metal contents by atomic absorption spectrometry. *Journal of Food and Drug Analysis*, 14, 2006, pp. 62–67.
13. Tuzen, M. – Sesli, E. – Soylak, M.: Trace metal levels of mushroom samples from East Black Sea region of Turkey. *Food Control*, 18, 2007, pp. 806–810.
14. Ferreira, K. S. – Gomes, J. C. – Chaves, J. B. P.: Copper content of commonly consumed food in Brazil. *Food Chemistry*, 92, 2005, pp. 29–32.
15. Turkish Food Codex 2002/63. Regulation of setting maximum levels for certain contaminants in foodstuffs. *Official Gazette*, September 23, 2002, Issue 24885. 13 pp.
16. Joint FAO/WHO Expert committee on food additives 53rd meeting, Rome, 1–10 June, 1999. Summary and conclusions. FAO/WHO, 1999. 21 pp.
17. Recommended dietary allowances. 10th ed. Washington : National Academy of Sciences, 1980. 285 pp. ISBN 978–0–309–04633–6.
18. Quality directive of potable water. 2nd ed. Geneva : World Health Organization, 1994. 197 pp.
19. Lynch, S. R. – Baynes, R. D.: Deliberations and evaluations of the approaches, endpoints and paradigms for iron dietary recommendations. *Journal of Nutrition*, 126, 1996, pp. 2404–2409.
20. Klavec, T. – Mandic, M. L. – Grgic, J. – Primorac, L. – Perl, A. – Krstanovic, V.: Selenium in selected foods grown or purchased in eastern Croatia. *Food Chemistry*, 85, 2004, pp. 445–452.
21. Pappa, E. C. – Pappas, A. C. – Surai, P. F.: Selenium content in selected foods from the Greek market and estimation of the daily intake. *Science of the Total Environment*, 372, 2006, pp. 100–108.
22. Dietary reference intakes for Vitamin C, Vitamin E, selenium, and carotenoids. Washington, D. C. : The National Academies Press, 2000. 509 pp. ISBN 978–0–309–06935–9.
23. Bratakos, M. S. – Lazos, E. S. – Bratakos, S. M.: Chromium content of selected Greek foods. *The Science of the Total Environment*, 290, 2002, pp. 47–58.
24. Peter, F. M. (Ed.): Recommended dietary allowance (RDA). 10th ed. Washington : National Academic Press, 1989. 285 pp. ISBN 0–309–04633–5.
25. Narin, I. – Tuzen, M. – Soylak, M.: Aluminium determination in environmental samples by graphite furnace atomic absorption spectrometry after solid phase extraction on amberlite XAD–1180/ pyro-catechol violet chelating resin. *Talanta*, 63, 2004, pp. 411–418.
26. Evaluation of certain food additives and contaminants (Thirty-third Report of the Joint FAO/WHO Expert Committee on Food Additives). WHO Technical Report Series, No. 776. Geneva : World Health Organization, 1989. 64 pp.
27. Onianwa, P. C. – Lawal, J. A. – Ogunkeye, A. A. – Orejimi, B. M.: Cadmium and nickel composition of Nigerian foods. *Journal of Food Composition and Analysis*, 13, 2000, pp. 961–969.

Received 23 July 2008; revised 22 August 2008; accepted 2 September 2008.