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ORIGINAL ARTICLE

Comparative efficacy of two standard methods for determination of iron and zinc in fruits, pulses and cereals

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Abstract Micronutrients are essential elements needed in small amounts for adequate human nutrition and include the elements iron and zinc. Both of these minerals are essential to human well-being and an adequate supply of iron and zinc help to prevent iron deficiency anaemia and zinc deficiency, two prevalent health concerns of the developing world. The levels of zinc and, iron were measured in the Banana, Papaya, Rice, Finger millet, Soybean and Urdbean. Standard Atomic absorption spectroscopy (AAS) method was also applied to all the samples for zinc and iron analysis and compared with inductively coupled plasma mass spectroscopy (ICP-MS). It was observed that there was no matrix interference affecting the determination of both elements interested in all the samples analyzed. Average concentration relative standard deviation and standard deviation were used for the statistical evaluation of the results for both elements. Correlation coefficient was used as statistical model to compare both the techniques.

Keywords ICP-MS, AAS · Zinc · Iron · Banana · Papaya · Rice · Finger millet · Soybean and Urdbean

Introduction

Nutritional quality is an important characteristic of food crop varieties that determines their functional value in the human diet. Micronutrient concentration in turn is an important component of nutritional quality especially for staple crops in developing countries (Frossard et al. 2000). Among micronutrient Fe and Zn are an essential mineral, vital to human metabolism, growth and immune function (Aggett and Comerford 1995). Iron (Fe) and zinc (Zn) are involved in the function of several enzymes and are essential for maintaining health throughout life (Uauy et al. 1998). Iron deficiency is the most prevalent single nutritional deficiency in the world and is the main cause of anemia in infants, children, adolescents, and women of childbearing age (DeMaeyer and Adiels-Tegman 1985). Zinc deficiency may be widespread in developing countries, but it is under-recognized due to lack of sensitive biomarkers of Zn status. In addition, there is scarce information on Fe and Zn intakes in our population. Accurate estimation of both the nutrient is very much essential and its value in same food material is varying with method adopted. For the elemental analysis there are a number techniques we could use, including spectrometric, flame and furnace AA (Atomic Absorption) and ICP-MS (ICP Mass Spectrometry). Selection of appropriate method to quantify the nutrient is an important task. Inductively coupled plasma-mass spectrometry (ICP-MS) is a multi-element analysis technique, which provides high selectivity and lower detection limits than other techniques, such as inductively coupled plasma optical emission spectrometry (ICP-OES), AAS and spectrometry (Tyler et al. 2002). These characteristics make ICP-MS an excellent tool for detailed characterisation of the elemental composition of numerous samples. When the highest quality of results is needed, the isotope dilution mode of analysis is the choice, whereas the quantitative mode of analysis is the default strategy (Laborda et al. 2001). The quantitative mode of analysis in ICP-MS requires external calibration with standards of each element to be determined. This strategy is time consuming and it is not easy to have a complete set of the multi-element standards required for the calibration. A third option, the semi-quantitative mode of analysis, is a versatile application of ICP-MS that it is claimed to allow the determination of about 80 elements with

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errors <20 % for most elements (Laborda et al. 2001; Soldevilla et al. 1998; Amarasiriwardena et al. 1997). This methodology has been successively applied to samples of different nature and origin, like biological (Amarasiriwardena et al. 1997; Krushevska et al. 1996; Alonso et al. 1997), environmental (Alonso et al. 1997), industrial (Hu et al. 1997), food (Castillo et al. 1999; Jakubowski et al. 1999; Castineira et al. 2001) and plastics (Fordham et al. 1995) samples. The semi-quantitative analysis software available for commercial ICP-MS instrumentation (e.g. TotalQuant II from Perkin-Elmer) has facilitated the rapid acquisition of analytical data by correcting automatically isobaric and molecular interferences as well as relative isotope abundances. This type of analysis is based on a pre-calibrated internal response (defined as ions per second per concentration unit) for all elements, which can be update with a single-point calibration (Amarasiriwardena et al. 1997)

In present study we have compared inductively coupled plasma mass spectrometry (ICP-MS), a latest technique can detect the elements at ppt level with atomic absorption spectrometry (AAS) for Fe and Zn determination in six different food material like Banana, Papaya, Rice, Finger millet, Soybean and Urdbean.

Material and method

Sampling

About 18 samples of banana and 36 samples of papaya fruits were collected from Navsari agricultural university farm and well managed farm of south Gujarat, India. Samples were physically cleaned by rinsing with deionised water to remove dirt and other extraneous filth. Fresh pulp of banana and papaya were used for analysis. Whereas 15 samples of rice, 60 samples of finger millet, 27 samples of black gram and 27 samples of soybean were collected from Navsari agricultural university farm and well managed farm of south Gujarat, India. Seed samples were dried and powdered to analyzed zinc and iron content.

Sample analysis

About 0.5 g of sample was weighed using a Teflon vessel, and then a mixture of concentrated HNO_3 and $HCLO_4$ (10:4) was added. The digestion vessel was closed and heated in the microwave oven. The obtained solutions were allowed to cool at room temperature, and then were filtered by Whatman No. 1 (110 mm pores size) filter paper into a 25 mL in volumetric flask and make the volume using double distilled deionized water. These extract were used for determination of Fe and Zn through AAS (Electronics Corporation of India Ltd. AAS 4141) and ICP-MS (Perkin Elmer series Elan-900) at food quality testing laboratory, Navsari agricultural university, Navsari-396450 (Gujarat), India.

Result

Iron and zinc content of rice

Iron content was found to be 66.55 ppm to 71.37 ppm in rice samples analyzed by ICP-MS and 66.45 ppm to 72.24 ppm (range) by AAS. The average iron content in all rice samples was found to be nearly similar 69.20 ppm by ICP-MS and 69.55 ppm by AAS. Zinc content was found to be 30.95 ppm to 49.1 ppm in rice samples analyzed by ICP-MS and 31.52 ppm to 48.8 ppm by AAS. The average zinc content in all rice samples was found to be 37.02 ppm by ICP-MS and 38.32 by AAS (Table 1).

Iron and zinc content of finger millet

The iron and zinc content of finger millets is presented in Table 2. The results showed that all samples contained detectable amounts of iron and zinc. Iron content varied between samples from 60.0 ppm to 95.9 by ICP-MS and 59.4 ppm to 99.3 ppm by AAS. The average iron content in all the finger millets samples was found to be nearly similar 73.22 ppm by ICP-MS and 73.72 ppm by AAS. Zinc content was found to be 16.1 ppm to 23.1 ppm in finger millets samples analyzed by ICP-MS and 16.7 ppm to 23.9 ppm by AAS. The average zinc content in all the finger millets samples was found to be 18.69 ppm by ICP-MS and 19.3 by AAS.

Iron and zinc content of papaya

Iron content was found to be 43.6 ppm to 70.5 ppm in papaya samples analyzed by ICP-MS and 37.5 ppm to 71.8 ppm by AAS. The average iron content in all papaya samples was found to be 51.04 ppm by ICP-MS and 52.04 ppm by AAS. Zinc content was found to be 6.9 ppm to 12.4 ppm in papaya samples analyzed by ICP-MS and 7.8 ppm to12.6 ppm by AAS. The average zinc content in all papaya samples was found to be 9.45 ppm by ICP-MS and 10.07 by AAS (Table 3).

Iron and zinc content of banana

The iron and zinc content of banana is presented in Table 4. The results showed that all samples contained detectable amounts of iron and zinc. Iron content was found to be from 16.8 ppm to 26.7 ppm by ICP-MS and 17.5 ppm to 27.9 ppm by AAS in all the banana samples analyzed. The average iron content in all the banana samples was found to be nearly similar 22.43 ppm by ICP-MS and 23.05 ppm by AAS. Zinc

Table 1 Analysis of iron and zinc in rice by ICP-MS and AAS

Name of sample	Iron		Zinc	
	Concentration (ICP-MS) ppm	Concentration (AAS) ppm	Concentration (ICP-MS) ppm	Concentration (AAS) ppm
Rice 1	68.84	68.12	49.1	48.8
Rice 2	71.37	72.24	37.4	38.5
Rice 3	71.37	72.19	36.1	36.7
Rice 4	67.89	68.75	30.95	31.52
Rice 5	66.55	66.45	31.56	32.12
Avg. Conc. Std. deviation	0.1	2.5	0.01	1.2
Avg. Conc. Relative std. deviation	1.0	5.9	0.9	3.8
Range	66.55–71.37	66.45–72.24	30.95–49.1	31.52–48.8
Mean	69.20	69.55	37.02	38.32
Correlation	0.969589		0.965291	

content was found to be 8.7 ppm to 12.1 ppm in banana samples analyzed by ICP-MS and 9.4 ppm to 12.9 ppm by AAS. The average zinc content in all the banana samples was found to be 10.58 ppm by ICP-MS and 11.6 by AAS.

Table 2 Analysis of iron and zinc in finger millet by ICP-MS and AAS

Name of sample	Iron		Zinc	
	Concentration (ICP-MS) ppm	Concentration (AAS) ppm	Concentration (ICP-MS) ppm	Concentration (AAS) ppm
Finger millet 1	95.9	99.0	23.1	23.9
Finger millet 2	93.2	95.3	20.7	21.3
Finger millet 3	64.5	66.6	20.5	21.2
Finger millet 4	60.8	62.6	19.6	20.1
Finger millet 5	87.3	90.6	19.3	19.9
Finger millet 6	97.1	97.8	19.3	19.8
Finger millet 7	80.9	81.0	19.6	20.2
Finger millet 8	66.7	67.3	19.1	18.9
Finger millet 9	72.6	72.1	17.3	17.8
Finger millet 10	60.0	59.4	18.6	19
Finger millet 11	69.3	70.5	19.9	20.2
Finger millet 12	65.7	66.9	17.6	18.3
Finger millet 13	71.4	72.5	18.3	18.9
Finger millet 14	63.8	64.6	18.6	19.4
Finger millet 15	60.9	62.4	17.3	17.9
Finger millet 16	76.5	64.4	17.6	18.9
Finger millet 17	64.4	66.2	17.3	18
Finger millet 18	69.8	70.6	16.1	16.7
Finger millet 19	80.6	82.4	16.2	17.1
Finger millet 20	63.0	62.3	17.8	18.5
Avg. Conc. SD.	0.1	3.3	0.1	8.6
Avg. Conc. RSD.	1.2	6.3	0.8	4.1
Range	60.0–95.9	59.4–99.3	16.1–23.1	16.7–23.9
Mean	73.22	73.72	18.69	19.3
Correlation	0.9688		0.985723	

Table 3 Analysis of iron and zinc in Papaya by ICP-MS and AAS

Name of sample (Papaya)	Fe (ppm)		Zn (ppm)	
	Concentration (ICP-MS) ppm	Concentration (AAS) ppm	Concentration (ICP-MS) ppm	Concentration (AAS) ppm
Papaya 1	47.0	48.2	9.2	9.9
Papaya 2	36.9	37.5	10.1	11.2
Papaya 3	43.6	45.3	12.4	12.9
Papaya 4	43.6	44.9	12.4	13.1
Papaya 5	53.7	54.8	11.5	12.6
Papaya 6	50.3	51.5	9.6	10.2
Papaya 7	51.2	51.9	7.1	7.9
Papaya 8	50.3	52.3	6.9	7.8
Papaya 9	70.5	71.8	8.3	8.9
Papaya 10	53.3	54.6	8.7	9.8
Papaya 11	50.3	51.4	9.3	10.1
Papaya 12	63.8	65.3	7.9	8.5
Avg. Conc. Std. deviation	0.7	10.2	0.5	3.4
Avg. Relative std. deviation	0.1	13	0.1	0.7
Range	43.6–70.5	37.5–71.8	6.9–12.4	7.8–12.6
Mean	51.04167	52.04167	9.45	10.075
Correlation	0.975332		0.94034	

Iron and zinc content of soybean

The soybean samples contain 71.8 ppm to 200.4 ppm iron and 40.9 ppm to 60.3 ppm zinc when analyzed by ICP-MS where as by AAS analysis it was found to be 78.6 ppm to 210.6 ppm iron, 39.8 ppm to 61.9 ppm zinc respectively. The average iron content found by ICP-MS was 123.73 ppm almost similar with, it was found to be 123.23 ppm by AAS in all the soybean samples. The average zinc content was found to be 50.7 ppm by ICP-MS and 51.1 ppm by AAS in all soybean samples (Table 5).

Iron and zinc content of urdbean

The iron and zinc content of urdbean is presented in Table 6. The results showed that all samples contained detectable amounts of iron and zinc. Iron content was found to be from 88.8 ppm to 221.9 ppm by ICP-MS and 86.1 ppm to 212.5 ppm by AAS in all the urdbean samples analyzed. The average iron content in all the urdbean samples was found to be nearly similar 142.2 ppm by ICP-MS and 143.5 ppm by AAS. Zinc content was found to be 32.9 ppm to 50.0 ppm in urdbean samples analyzed by ICP-MS and 34.1 ppm to 54.9 ppm by

Table 4 Analysis of iron and zinc in Banana by ICP-MS and AAS

Name of sample (Banana)	Fe (ppm)		Zn (ppm)	
	Concentration (ICP-MS) ppm	Concentration (AAS) ppm	Concentration (ICP-MS) ppm	Concentration (AAS) ppm
Banana 1	26.7	27.2	12.1	12.8
Banana 2	18.1	18.9	8.7	9.4
Banana 3	23.5	24.2	12.0	12.9
Banana 4	16.8	17.5	10.1	10.8
Banana 5	26.8	27.6	10.1	11.1
Banana 6	26.7	27.9	11.5	12.6
Avg. Conc. Std. deviation	0.5	8.5	0.6	4.2
Avg. Relative std. deviation	0.1	12	0.2	0.8
Range	16.8–26.7	17.5–27.9	8.7–12.1	9.4–12.9
Mean	22.43333	23.05	10.58333	11.6
Correlation	0.967581		0.968975	

Table 5 Analysis of iron and zinc in Soybean by ICP-MS and AAS

Name of sample (Soybean)	Fe (ppm)		Zn (ppm)	
	Concentration (ICP-MS) ppm	Concentration (AAS) ppm	Concentration (ICP-MS) ppm	Concentration (AAS) ppm
Soybean 1	199.7	210.6	56.3	58.6
Soybean 2	114.0	125.8	50.7	50.0
Soybean 3	200.4	186.5	60.2	61.3
Soybean 4	111.3	102.6	48.7	50.8
Soybean 5	88.9	80.1	52.4	48.9
Soybean 6	99.5	97.3	40.9	39.5
Soybean 7	81.7	85.4	46.5	49.7
Soybean 8	71.8	78.6	40.9	39.8
Soybean 9	146.3	142.2	60.3	61.9
Avg. Conc. Std. deviation	0.9	3.5	1.2	4.3
Avg. Relative std. deviation	0.1	1.3	0.1	2.3
Range	71.8–200.4	78.6–210.6	40.9–60.3	39.8–61.9
Mean	123.7333	123.2333	50.7	51.1
Correlation	0.981578		0.96789	

AAS. The average zinc content in all urdbean samples was found to be 43.1 ppm by ICP-MS and 43.7 by AAS.

Discussion

Iron and zinc contents of all samples

Iron and zinc content reported in this study reveals that iron and zinc content falls in the ranges reported in the previous studies. Anuradha et al. (2012) have analyzed 126 accessions of rice genotypes for iron and zinc concentration. They found Iron concentration ranged from 6.2 ppm to 71.6 ppm and zinc from 26.2 ppm to 67.3 ppm in their study which is similar to our study. Similarly Shashi et al. (2007) found 36.00 ppm to 73.00 ppm iron content and 18.00 ppm to 23.00 ppm zinc in finger millet genotype analyzed. In case of Iron and zinc content of banana, Mohpatara et al. (2010) were found 8.3 ppm iron and 2.3 ppm zinc in banana. Rani et al. (2008) reported 84.00 ppm to 112.00 ppm iron and 71.00 ppm to 79.00 ppm zinc in analyzed soybean genotype, supporting our result.

Table 6 Analysis of iron and zinc in Urdbean by ICP-MS and AAS

Name of sample (Urdbean)	Fe (ppm)		Zn (ppm)	
	Concentration (ICP-MS) ppm	Concentration (AAS) ppm	Concentration (ICP-MS) ppm	Concentration (AAS) ppm
Urdbean 1	204.2	212.5	36.4	38.5
Urdbean 2	90.3	95.3	38.9	37.4
Urdbean 3	96.2	95.8	48.6	47.6
Urdbean 4	208.9	211.6	42.9	45.8
Urdbean 5	144.4	159.2	48.4	48.2
Urdbean 6	132.1	139.7	48.1	46.2
Urdbean 7	93.5	90.9	42.0	40.6
Urdbean 8	221.9	200.6	50.0	54.9
Urdbean 9	88.8	86.1	32.9	34.1
Avg. Conc. Std. deviation	0.7	2.1	0.4	6.2
Avg. Relative std. deviation	0.1	5.3	0.1	1.9
Range	88.8–221.9	86.1–212.5	32.9–50.0	34.1–54.9
Mean	142.2	143.5	43.1	43.7
Correlation	0.983113		0.932699	

Comparison of ICP and AAS methods

The two methods used for mineral analysis were reliable and gave similar results as shown by low coefficients of variation and highly significant correlations between methods. In terms of repeatability of the iron analysis in six different matrices shown average concentration standard deviation and relative standard deviation 0.5 and 0.55 respectively for ICP-MS while 5.01 and 7.3 respectively for AAS method which revealed that ICP-MS is more precise technique than AAS. Similarly in zinc analysis average concentration standard deviation and relative standard deviation found to be 0.56 and 0.36 respectively for ICP-MS while 4.65 and 2.26 respectively for AAS method which revealed that ICP-MS is more precise technique than AAS. Relative standard deviation value found lower in ICP-MS than AAS might be due to ICP-MS is almost free from chemical interferences. The chemical bonds that still exist at below 3,000 °C are completely ruptured at above 6,000 °C. The high temperatures reached in plasma eliminate chemical interferences, which accounts (for the most part) for the better detection limits achieved for refractory elements. Chemical interferences are common in AAS but are less common or practically nonexistent in ICP-MS due to the relatively high temperature of the plasma, long residence time in the plasma, and inert atmosphere of the Argon plasma (Tyler et al. 2002). ICP-MS has linear dynamic range between 10^5 and 10^7 where as AAS has linear dynamic range up to 10^3 which revealed higher accuracy of ICP-MS at low concentration (Tyler et al. 2002). Correlations coefficient between Fe and Zn determination through the ICP and AAS methods were 0.9743 and 0.9601, respectively which revealed both the method are comparable. Previous publications have also shown significant positive correlation coefficients and linear regression analysis between the values of element determined through these two methods. (Bland and Altman 1986; Bland and Altman 1999; Kivisto 1993; Ottenbacher and Tomchek 1994; Rothwell 2000; Blair et al. 2009; Miksa et Al. 2005)

Economics comparison of both the method

The capital cost and running cost of an ICP-MS is quit high compared to AAS. Although high capital cost and running cost of ICP-MS this technique have many advantages when to go for multi elemental analysis will result comparable cost per sample. The real advantage of ICP-MS is the speed of analysis, the wider linear dynamic range, accurately and reproducibly results high sensitivity.

Conclusion

It is evident from this work presented that the both techniques are comparable for the analysis of iron and zinc. In the last section, we discussed both methods to determine iron and zinc, which clarifies that improving determination technique, should take into account many things like sensitivity, efficiency, cost, etc. By comparing ICP-MS and AAS, we determined that ICP-MS was better but more expensive, which helped determine that the most important thing to do in any analysis is to compare the different methods and then choose the best one of them that works.

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