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Application News

NO. SP-11-ADI-067

ICPMS-2030

Estimation of toxic elements in jam, juice and concentrates using Shimadzu Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Introduction

Commercially available fruit jams, juices and concentrates contains different physiologically and nutritionally important compounds, e.g. bioactive phytochemicals (phenolic, flavonoids, carotenoids) and vital nutrients (proteins, carbohydrates, and vitamins). An increased popularity and demand for these food items is due to increased awareness about healthcare and average life expectancy in society. All these reasons make fruit juices very widely consumed in both developed and developing countries, and by different age groups. Apart from nutritional and functional compounds, fruit juices contain many major (macro elements Ca, K, Mg), minor (microelements Cu, Fe, Mn, Zn) and trace elements (As, Cd, Cr, Pb, Hg, Se).
Copper plays an important role in hemoglobin synthesis. Zinc is essential for many enzymes involved in several physiological functions such as protein synthesis and energy metabolism. Lead is highly toxic element that accumulates in biological systems and it leads to deficits in psychological functions such as intelligence and learning ability in humans. Even though macronutrients are required for many biological functions, their excess presence in human body leads to health hazards. The main sources of the elements in such samples may be water, fruit, soil, manufacturing process, container and environmental contamination due to fertilizers, pesticides, raw materials, etc.
Considering the nutritional value associated with essential elements indispensable for life maintenance on one hand, and the health hazard of toxic elements on another hand, elemental analysis of these food items is of importance. It becomes particularly important for fruit juices, due to high seasonal variability in their elemental composition. Their regular examination with respect to elemental content, can bring valuable information about their suitability for consuming. Reliable information on the elemental content of jam, fruit juice, and concentrates can help producers to improve the overall quality of these products by identifying sources of contamination with toxic elements. It is also important for consumers in view of the nutritional value of jam, fruit juices and concentrates. The present work aimed to develop an ICP-MS method for determination of toxic elements in jam, juice and concentrates samples as per FSSAI limitations.

Experimental

The samples of jam, juice and concentrates were purchased from local market. The limits of elements as per FSSAI guidelines are shown in Table 1[1-2].

Table 1: FSSAI limits in ppm

Elements	FSSAI Limits (ppm)
Arsenic (As)	0.1*
Cadmium (Cd)	0.1*
Copper (Cu)	7
Mercury (Hg)	1
Lead (Pb)	1
Tin (Sn)	250
Zinc (Zn)	5

***Due to nonavailabilty of the FSSAI limits, they were taken arbitrarily.**

Sample Preparation

About 500 mg of sample was weighed in microwave vessels. Samples were kept for 10 minutes pre-digestion after carefully adding 3 mL of suprapure nitric acid (HNO_3) and 2 mL of ultra pure water. The vessels were then heated in microwave digestor system under controlled temperature program (Table 2).
After digestion, samples were left to cool to ambient temperature and filled up using ultra-pure water in a 50 mL volumetric flask. Pre-spiked recovery studies were carried out at LOQ, & 10 x LOQ levels by spiking samples with standard stock solution. The LOQ level was set as per with EC 836/2011[3

Table 2: Microwave digestion program

Steps	Ramp (min)	Temp (°C)	Hold time (min)
1	10	120	05
2	10	180	20

Table 3 shows limit of quantification (LOQ) which are achieved in the present work.

Table 3: LOQs (ppm) for Jam, Juice and Concentrates

Elements	LOQs (ppm)
Arsenic (As)	0.02
Cadmium (Cd)	0.02
Copper (Cu)	1.4
Mercury (Hg)	0.2
Lead (Pb)	0.2
Tin (Sn)	50
Zinc (Zn)	1

Calibration standard preparation

Sigma Aldrich 1000 ppm individual certified reference standards were used for preparation of intermediate stock solution. Calibration standard solutions were prepared by diluting intermediate stock solution. The concentrations of linearity standards are given in Table 4. Yttrium and Bismuth were used as internal standards. Internal standards were aspirated using online internal standard addition kit.

Analytical Conditions

A Shimadzu ICPMS-2030 coupled with auto sampler AS-10 (Figure 1) was used for analysis.

Figure 1.Shimadzu ICPMS-2030 with autosampler AS-10

The instrument configuration and operating parameters are summarized in Table 5.

Table 5: Instrumental parameters

Plasma torch	Mini torch (P/N:S211-94018)
Radiofrequency	1.2 kW
Sampling depth	5 mm
Plasma gas flow	10 L/min
Auxiliary gas flow	1.1 L/min
Carrier gas flow	0.7 L/min
Collision gas	Helium
Collision gas flow	6.0 mL/min
Cyclone Chamber temp.	5 °C

Table 4: Linearity standards (ppb) for Jam, Juice and concentrate samples

Elements	As	Cd	Hg	Pb	Cu	Sn	Zn
Calibration Std Level 1	0	0	0	0	0	0	0
Calibration Std Level 2	0.1	0.1	1	1	7	25	5
Calibration Std Level 3	0.2	0.2	2	2	14	50	10
Calibration Std Level 4	0.5	0.5	5	5	35	125	25
Calibration Std Level 5	1	1	10	10	70	250	50
Calibration Std Level 6	2	2	20	20	140	500	100
Calibration Std Level 7	2.5	2.5	25	25	175	625	125

Results and discussion

The calibration standard solutions showed good linearity (Figure 2) with correlation coefficient (r) ≥ 0.999 for all elements. The elemental content in all matrices was found be below LOQ. Table 5 shows the results obtained for % recoveries at LOQ and 10 x LOQ level. Using ICPMS-2030, excellent spike recoveries were achieved for most of the elements in spiked samples. All recoveries were within 80 to 120 % for all elements.

Conclusion

The macronutrient as well as toxic elements specified by FSSAI were measured in sub ppb to ppm range. The spiked recoveries shows reliability of the method. The results demonstrates suitability of the Shimadzu ICPMS-2030 for routine analysis of jam, juice and concentrate samples.

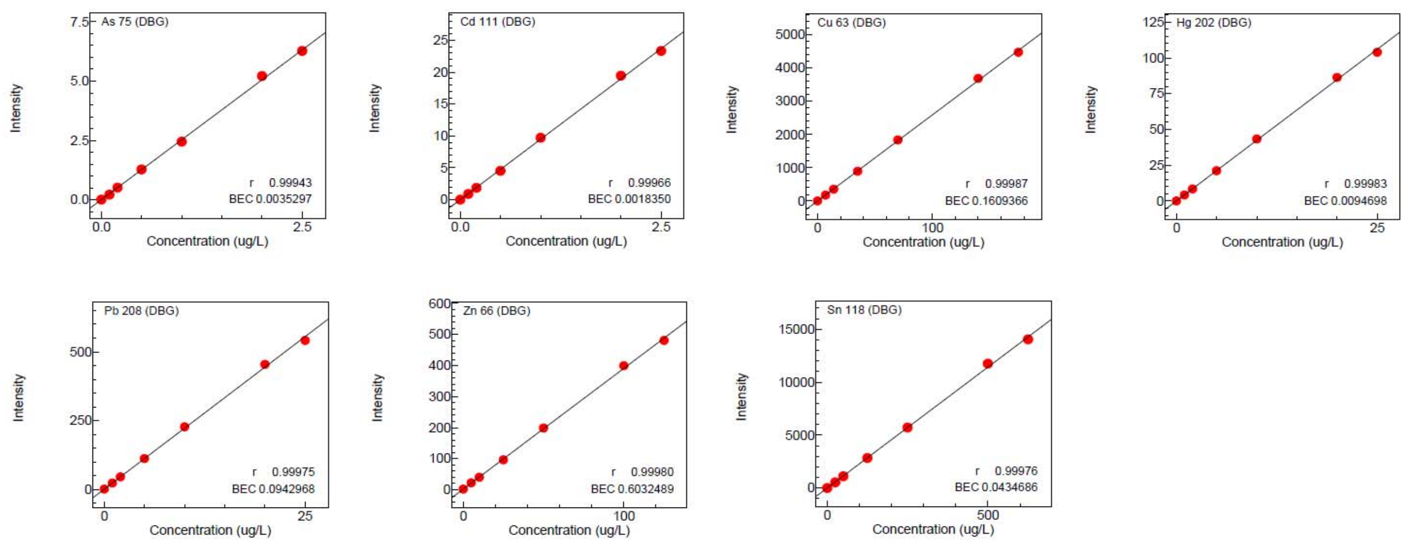

Figure 2. Linearity curves of targeted elements

Table 5: Average % recoveries obtained at LOQ and 10 x LOQ (n =4)

Elements	Jam		Juice		Concentrate	
	LOQ % Recovery	10 x LOQ % Recovery	LQQ % Recovery	10 x LOQ % Recovery	LOQ % Recovery	10 x LOQ % Recovery
As	91.8	91.7	92.5	95.2	102.3	99.8
Cd	97.4	95.4	92.3	93.8	99.1	95.6
Cu	94.9	92.7	95.2	92.3	97.3	91.7
Hg	101	97.4	97.4	96.2	103	102.4
Pb	99.5	94.4	103.3	99.6	109.9	107.7
Sn	96.1	100.9	96.1	97.5	98.6	104.7
Zn	107.1	94	96.8	94.9	108.6	96.7

References

[1] Food Safety and Standards (Contaminants, Toxins and Residues) Regulations, 2006

[2] Food Safety and Standards (Contaminants, Toxins and Residues) Regulations, 2011

[3] Commission regulation, EU No 836/2011

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