REVIEW ON ESTIOMATION OF ZINC IN VARIOUS FOOD BY DIFFERENT ANALYTICAL METHODS

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Abstract- Zinc is essential mineral compound found in various foods. Various dairy products, cereals, vegetables, etc are rich sources of zinc. Zinc is 24th most abundant in earth crust. Deficiency of zinc can lead to various diseases and its higher amount can also be toxic. This review gives a brief overview of analytical methods for the Zinc in various food samples. This review not only focuses on conventional methods but also newer analytical methods. The different techniques for zinc estimation are UV visible spectroscopy, atomic emission spectroscopy, ICPES, fluorimetry, Voltammetry, Different Chromatographic methods like ion chromatography, size exclusion chromatography. etc. Their reliability, accuracy, reproducibility, better result are also mentioned in the review.

Keywords: Zinc, Analytical methods, food samples, Spectroscopy, chromatography

1.INTRODUCTION

Zinc is the essential mineral is naturally present in some food added to others and available as dietary supplement. Zinc is also in some cold lozenges, over the counter drugs sold as cold remedies, and some denture adhesive cream. $^{(1),(2),(3)}$

Zinc promotes health growth and development during pregnancy, infancy, childhood and adolescence and is involved in the sense of taste. $^{(1),(3),(4)}$

1.1sources of zinc

The richest food sources of zinc include meat, fish and seafood, oysters contain more zinc serving than any other food Eggs, beans, dairy products, nuts and whole grains also contains zinc.

But the bioavailability of zinc is lower than obtained from animal source because of presence of phytates. Phytates is a form of storage of phosphorus in plants and bind to some minerals such as zinc in intestine and insoluble complex that inhibit zinc absorption Fruits and vegetables contain a very little zinc

Breakfast cereals which are often fortified contains zinc. (1),(4)

1.2 dietary supplements

Zinc is available in supplements containing only zinc; supplements containing zinc in combination with other ingredients; and in many multivitamin/multimineral products. Supplements can contain any of a variety of forms of zinc, including zinc sulphate, zinc acetate, and zinc gluconate. The Supplement Facts panel on a dietary supplement label declares the amount of elemental zinc in the product, not the weight of the entire zinc containing compound. ^{(1)(2),(3)}

Absorption of zinc from supplements containing zinc citrate or zinc gluconate is similar, at approximately 61% in young adults; the absorption from supplements containing zinc oxide is 50%. However, the iron added to enriched or fortified foods does not interfere with zinc absorption. $^{(3),(7),(8)}$

2. ZINC DEFICIENCY

Zinc deficiency is commonly seen in developing regions that is attributable to malnutrition; however, in developed regions, it is found to be associated with aging and many chronic illnesses. Zinc deficiency can be acquired or inherited. Patients with an acquired form of zinc deficiency usually have a combination of various factors, such as:

- Nutritional: lack of meat intake, excess phytates (present in legumes, seeds, soy products, and whole grains), or oxalates (found in spinach, okra, nuts, and tea)
- Chronic illnesses: the presence of chronic illnesses (chronic gastrointestinal diseases, diabetes, liver disease, sickle cell disease, kidney disease, excess alcohol consumption, HIV infection) or chronic or infections.
- Skin manifestations Skin conditions associated with zinc deficiency include acrodermatitis enteropathy, cheilitis, and dermatitis.

Prolonged and severe deficiency of zinc can lead to many complications, such as:

- Growth failure Untreated zinc deficiency is often linked with permanently stunted growth and development.
- Hypogonadism
- Recurrent infections Zinc deficiency can aggravate both acute and chronic infections and these infections, in turn, can exacerbate zinc deficiency themselves.
- Diarrhoea

- Delayed wound healing
- Low bone mineral density The effect of the deficiency of zinc on bone density is not well understood. There is limited evidence adding calcium to zinc supplementation is more beneficial than giving calcium alone. ^{(6),(7),(8)}
- 2.1 recommended daily elemental intake is:⁽⁷⁾
- 3 mg/day for children less than 4 years
- 5 mg/day for children between 4 and 8 years
- 8 mg/day for children between 9 and 13 years
- 9 mg/day for women (non-pregnant and non-lactating)
- 11 mg/day for men
- 11 to 12 mg/day in pregnant and lactating women
- 2.2 formulations of zinc supplements include:⁽⁸⁾
- Zinc sulfate
- Zinc acetate
- Zinc aspartate
- Zinc orotate
- Zinc gluconate
- Vitamin A deficiency

Patients require dietary counselling regarding food rich in zinc. In addition to supplementation, patients deficient in zinc can consider eating more:

- Red meat
- Poultry
- Wheat germ
- Wild rice

Vegetarians may find it more challenging to obtain sufficient dietary zinc. For these patients, options for zinc sources include baked beans, peas, cashews, and almonds.^{(7),(8)}

2.3health risks of excessive zinc

If used for weeks, doses of 50 mg zinc or more—typically from supplements or excessive use of denture adhesive creams that contain zinc—can interfere with copper absorption (which can cause low copper status), reduce immune function, and lower HDL cholesterol levels. The amount of zinc obtained from food is rarely as high as 50 mg, so the zinc in foods is unlikely to cause zinc toxicity. Very high doses of zinc from supplements (142 mg/day) might also interfere with magnesium absorption and disrupt magnesium balance.^{(6),(7)}

3. ANALYTICAL METHODS FOR THE ESTIMATION OF ZINC IN FOOD

3.1 Atomic Absorption Spectroscopy ⁽⁹⁾

This method for the estimation of zinc uses bis-azo dye, 2,6-bis(1-hydroxy-2-naphthylazo) pyridine as spectrophotometric reagent.



Figure :3.1.1 Structure of PBN

Zinc recommended procedure for analysis: -

Zinc (II) in a solution to an aliquot containing zinc (II) ions between $5.0-25\mu g$, add 2 mL of $1.0 \times 10^{-3} mol/L$ PBN solution, 1 mL of borate buffer solution (pH7.8), 1 mL of

thiosemicarbazide (TSC) and make up the volume to 25 mL, maintaining 50%(v/v) ethanol concentration in the final solution. Record the absorbance at 560 nm against are agent blank prepared under similar conditions.

3.2. Atomic Flouroscence Spectroscopy⁽¹⁵⁾

Reagents: -

A stock solution of zinc (1000 μ gml⁻¹) was prepared by dissolving 0.1000 g of pure zinc in 5ml of 6 mol l⁻¹ hydrochloric acid and being made up to 100 ml with deionized water. The working solutions were prepared by diluting appropriate aliquots from the stock solution. Potassium tetra hydroborate solution (20 g l⁻¹) was prepared daily fresh by dissolving KBH4 powder in water, stabilized in 5 g l⁻¹potassium hydroxide. CTAB solution (1 × 10⁻² mol l⁻¹) was prepared by dissolving the surfactant in water by gentle warming.

The other surfactants of SLS and SDBS assessed were prepared in a similar way. All reagents used were of analytical reagent grade and deionized water was used throughout.



Figure: 3.2.1 schematic diagram of intermittent flow system

Procedure: -

Approximate 0.5 g sample of the reference material was accurately weighed and placed into a digest flask with 4ml nitric acid and 1ml sulfuric acid. The digest was carried out under conventional pressure until the solution appeared clear. The resultant solution was transferred into a 100 ml volumetric flask, followed by neutralization with potassium hydroxide solution and finally diluted to volume with water for determination. Hydride generation was carried out with mixing the sample solution, containing 0.35 mol l^{-1} hydrochloric acid and 0.8 mg l^{-1} nickel ion, for determination with 20 g l^{-1} KBH4 using an intermittent flow system described.

3.3. Flame Atomic Absorption Spectroscopy (21)

The reagent used is 1-(2-pyridylazo)-2-naphthol (PAN) and the micellar phase is obtained using the non-ionic surfactant octylphenoxypolyethoxyethanol (Triton X-114) and centrifugation. The optimization step was performed using Box–Behnken design for three factors: solution pH, reagent concentration and buffer concentration. A multiple response function was established in order to get an experimental condition for simultaneous extraction of copper and zinc.

Sample preparation: -

A mass of approximately 100mg of homogenized samples in a blender was weighed and volumes of 2.0mL of concentrated HNO3 and 1.0mL of 30% (v/v) H2O2 were added to the samples. After that, acid digestion bombs (4746 Model, Parr Instrument Company, USA) were closed and kept for 12 h at 150 ± 10 °C, which was the optimized time to achieve the complete dissolution. After cooling down the solutions to room temperature, pH was adjusted to 8.6 with 1mol L⁻¹ NaOH before making up to 50.0 mL. All samples were analysed in triplicate.

3.4. Microwave induced Atomic Emission Spectroscopy (12)

Determination of zinc in bread samples

Procedure: -

One slice (approximately 25 g) from different types of bread samples obtained from markets were dried at 100 °C in an oven for 2 days. Samples were ground using agate mortar and blended thoroughly. 1.0 g of portions were weighed precisely and dissolved in a mixture of 3 mL of HNO3 (65% w/v) and 1 mL of H2O2 (35% w/v) at 100 °C for 2 h. Digests were then diluted appropriately and aspirated to the plasma. Blanks were subjected to the same procedure. Results were given as the average of at least 3 replicates

3.5. Solid Surface Molecular Fluorescence (14)

A new method for zinc pre-concentration/separation and determination by molecular fluorescence is proposed. The metal was complexed with o-phenanthroline and eosin at pH 7.5 in Tris; a piece of filter paper was used as a solid support and solid fluorescent emission measured using a conventional quartz cuvette.

Procedure for analysis: -

Sample and standards Zn (II) $(1.29 \times 10^{-3} \text{ to } 4.50 \,\mu\text{g L}^{-1}, \text{pH7.5} \text{ and } 1 \text{ mL}$ methanol were put in a crystallizer flask, and the mixture diluted to 10 mL with ultrapure water. Pieces of Blue-Ribbon filter paper (1 ×3 cm) were impregnated through contact with each solution for 1 min (n = 4). The filter papers were dried at room temperature and kept in a dried ambient (20 °C–25 °C) atmosphere until analysis. Sample filter paper were was arranged in a conventional quartz cell, adapted for solid support, and SSF determined at $\lambda_{em} = 440$ nm (emission), using $\lambda_{ext} = 370$ nm (excitation)

3.6. Inductively Coupled Plasma Mass Spectroscopy (11) Responses

Reagents: -

All reagents used were of analytical-reagent grade, except HNO3 and HCl which were previously purified in a quartz sub-boiling still (Kürner) before use. A clean laboratory and laminar-flow hood capable of producing class 100 were used for preparing solutions.

High purity de-ionized water obtained using a Milli-Q water purification system

Sample preparation: -

Samples (0.10–0.25 g) were accurately weight in a PFA digestion vessel, and then 4 ml of nitric acid 14 mol/L + 2 mL of 30% (v/v) H2O2 were added. The bomb was placed inside the microwave oven, and the decomposition was carried out according to the difference in temperature. After that, the digestate were left to cool and then the volume made up to 50 mL with Milli-Q water. Then, Rhodium was added as internal standard to a final concentration of $10 \,\mu g/L$

3.7. Size exclusion chromatography – Inductively coupled plasma mass spectroscopy (13)

New method was developed for determination of zinc from cabbage, broccoli and kale (family Brassicaceae, species oleracea) using solid phase extraction (SPE) and size-exclusion chromatography inductively coupled plasma mass spectrometry (SEC-ICP-MS). **Procedure:** -

Tris [2-Amino-2- (hydroxymethyl)-1,3-propanediol]/hydrochloric acid (Tris/HCl) or ammonium nitrate were used as extractants added to the freeze-dried samples, which were then sonicated and centrifuged.

An enzymatic mixture was added to the extracts and then incubated for 5h and 18 h prior to analysis by SEC-ICP-MS

3.8. Voltametry (10)

This analytical method involves determination of zinc by cathodic pulse differential voltammetry.

Principle: -

Homogenized food samples are dry ash at475°C, using H2S04 as an ashing aid. The resulting ash is dissolved in dilute, HN03 and diluted to volume, and an aliquot is analysed for Zn by CSDPV.

Procedure: -

Food samples are dry ash using a sulfuric acid ashing aid, dissolved in dilute nitric

acid, buffered at pH ~ 4.3 with an acetate buffer, and quantitatively analysed using the

technique of standard additions at a hanging mercury drop electrode.

3.9. Extractive Spectroscopy⁽¹⁶⁾

N-ethyl-3-carbazolecarboxaldehyde-3-thiosemicarbazone (ECCT) is proposed as a new sensitive reagent for the extractive spectrophotometric determination of zinc (II). The ECCT forms yellow coloured species of zinc (II) at pH range 3.0–5.5 and the complex was extracted into benzene



Figure: 3.9.1 structure of ECCT

Procedure: -

To an aliquot of a working standard solution containing 12.5–150 lg zinc(II), were added pH 6.0 buffer (3 mL), 0.5% reagent solution (2 mL) and a salting-out agent, 0.1 mol/l magnesium sulphate (1 mL). The mixture was shaken two times with 10 mL portions of benzene each time for 1 min and allowed to stand for a few minutes. The two organic phases were collected into a 25 mL volumetric flask and made up to the mark with benzene. The absorbances of all the organic phases were measured at 420 nm against the reagent blank.

3.10. Fluorimetry ⁽¹⁷⁾

Dry dog food samples were acquired in local supermarkets. Each sample was digested induplicate, after a digestion pre-treatment procedure, where the samples were oven-dried at 65° C to constant weight and then ground in a 1mm sieve mill. Ground samples (*ca.* 500 mg) were solubilized by microwave-assisted acid digestion, using an MLS 1200 Mega high-performance microwave digestion unit equipped with an HPR-1000/10 S rotor.

Procedure: -

After weighing the sample using a plastic spatula, 3mL of HNO3 and 1mL of H2O2 were added to each polytetrafluoroethylene digestion vessel. The samples were subsequently submitted to a microwave heating program of 250W for 1 min, 0W for 1 min, 250W for 5 min, 400W for 5 min, and, finally, 650W for 5 min. The vessels were then allowed to cool to room temperature. thereafter, the content was transferred to 25mL polypropylene volumetric flasks and water was added to bring up to total volume. This digest was analysed by the developed fluorometric assay. A blank constituted by 500 μ L of water was included in each digestion run.

3.11. Ion chromatography (19)

Ion chromatography coupled with UV-vis detection is shown to be an appropriate technique for this objective.

Procedure: -

The method is based on the formation of mineral complexes by pyridine-2,6-dicarboxylic acid in the mobile phase. The complexes are then post column derivatized with 4-(2-pyridylazo) resorcinol (PAR), resulting in mineral-PAR complexes that are detected by UV-vis absorption at 500 nm. This facilitates the simultaneous separation and quantification of minerals in one chromatographic run. Within 16 min, Cu, Ni, Zn, Co, Mn, and Fe are analysed. When a 50 μ L injection volume is used, the average detection limit is 5 ppb in the injection liquid.

Different sample treatments were evaluated. The concentration of acid in the treated sample varied with the sample treatment, which may cause a limitation for the injection volume.

3.12. Microwave induced plasma optical emission spectroscopy ⁽²⁰⁾

Analytical method was developed for the estimation of zinc in fish muscle by microwave induced plasma optical emission spectroscopy after acid leaching

Procedure: -

For Zn, 5 g of homogenized sample was weighed on an analytical balance directly inside the Erlenmeyer used to conduct the digestion. Ten millilitres of HCl and 5 mL of HNO3 were added along with 1 mL of deionized water, to obtain a smoother reaction. The blank sample was prepared with the same amount of acids added to the samples. The samples were placed on a heating plate at 140 °C for 2 h. After the digestion, they were poured into a 50-mL gauge volumetric flask whose volume was made up with deionized water. For the CRM, half of the amounts were used for the sample, the acids, and the final volume. Concentrations of the analytes were determined with an emission wavelength of 213.857 nm for Zn. The pumping speed was 15 rpm and the nebulizer pressure was 240 kPa for Zn.

3.13. UV Visible Spectroscopy ⁽¹⁸⁾

By using dispersed TiO2 nanoparticles (TiO2-NPs) in a combined cloud point and solid phase extraction for the efficient preconcentration and determination of Zn2p in various samples.

Procedure: -

The colorimetric determination was carried out by performing the following steps. 5 mg of TiO2-NPs was added to 8 mL of 5% (v/v) of Triton X-100 as the non-ionic surfactant. The mixture was stirred for 1 min, causing further dispersion and suspension of the TiO2-NPs. Then an aliquot of zinc solution (so that its final concentration would be in the range of 0.5–90 mg L⁻¹), 1 mL of phthalate buffer (pH 7) and 1 mL of 0.5 mol L1 of NaCl, were added to the above mixture in a 50 mL volumetric flask and diluted to the mark with water. This solution was transferred to a 50 mL conical centrifuge tube and placed in a thermostat water bath at 80 °C for 40 min. Under this condition, the analyte was adsorbed on the TiO2-NPs and the phase separation occurred., the solution was cooled in an ice-bath for 5 min. The TiO2-NPs easily settled down in the surfactant phase and the aqueous phase was decanted. The enriched surfactant phase was separated from TiO2-NPs by centrifugation (3 min). Next, 500 μ L of dithizone (5.85 - 104 mol L⁻¹) was added to TiO2-NPs and shaken for about 2 min to desorb the Zn2b ions. Finally, the mixture was centrifuged (3 min), the eluate (Zn dithizonate) was collected in a 700 μ L glass cell and its absorbance was measured at λ /4530 nm by UV–vis spectrophotometer. A blank solution was also run under same conditions without adding any zinc ion.

4. RESULTS AND DISCUSSION

4.1. Atomic absorption spectroscopy ⁽⁹⁾

The ethanolic solution of PBN gave very deep blue to violet colour with zinc (II)

Sample	1	ash ml/g	Zn found in whole sample using PBN(ug)	Zn found in whole sample using AAS(u)	Range of Zn levels(mg/11g)
		, <u>B</u>	Sample asing P Dr (µg)	sample using mis(µ)	or (mg/100ml)
Milk samples					
Cow	4	100	279,271,276,277	280,273,276,274	0.271 0.279
Buffalo	4	100	225,220,223,228	220,221,223,226	0.220 0.228
Goat	4	100	201,206,204,201	202,205,204,201	0.201 0.206
Food samples					
Cicer arietinum	3	5	34.28,34.81,33.74	34.30,34.86,33.76	0.674 0.696
Oryza sativa (bran-rice)	4	2	89.30,92.02,92.70,91.28	89.30,92.75,91.28	4.463 4.635
Penniseliumtyphoidem (bajra)	4	5	53.29,53.83,53.56,54.10	53.31,53.80,53.50,54.0	1.065 1.082
Zea mays(maize)	4	4	27.32,27.58,27.85,28.38	27.36,27.89,27.60,28.32	0.683 0.709
Lens culinarist (mansur)	3	4	94.08,96.14,96.82,95.45	94.12,96.06,96.80,95.54	2.352 2.420
Triticum aestivum (wheat flour)	3	5	32.27,32.96,34.33	32.30,32.92,34.31	0.645 0.686
Milk powder	4	5	111.9,112.6,111.2,113.9	111.7,112.8,111.3,113.6	2.225 2.278
Tea samples					
Lipton	3	2	111.9,140.4,143.4	144.6,140.8,143.9	7.020 7.245
Brooke-Bond	3	2	128.5,132.9,135.9	128.8,133.0,136.0	6.425 6.795
Тај	3	2	80.60,79.68,82.17	80.70,77.75,82.28	3.884 4.108

Table 4.1.1-content of zinc in various food and milk samples by AAS

4.2. Atomic fluoroscence spectroscopy (15)

The results were found to be $45.3 \pm 1.0 \ \mu g \ g^{-1}$ (average value \pm standard deviation) for milk powder and $21.4 \pm 1.7 \ \mu g \ g^{-1}$ for wheat powder, respectively agreed with the certified values of $46.8 \pm 2.8 \ \mu g \ g^{-1}$ and $22.7 \pm 2.0 \ \mu g \ g^{-1}$ within the error of determination.



Figure:4.2.1 Effect of transfer line length on the signal of 100 ng ml⁻¹Zn(II) in 2.5 × 10–5 M CTAB media

4.3. Flame atomic absorption spectroscopy (21)

Under the optimized experimental conditions, the method allows the determination of zinc with a LOD of $0.15\mu g L^{-1}$ and precision as R.S.D. of 2.7 and 1.7% for concentrations of 10 and $50\mu g L^{-1}$, respectively. The enhancement factors obtained was 32 for zinc. The zinc content in the samples analysed varied from 8.7 to $22.9\mu g g - 1$

Samples	Zinc ($\mu g g^{-1}$)
Oats	22.9±1.4
Powdered chocolate	9.0±1.0
Corn flour	16.8±1.6
Wheat flour	8.7±1.1

 Table: 4.3.1 samples and zinc content by FAAS

4.4 Microwave induced plasma atomic emission spectroscopy (12)

All determinations were made by linear calibration technique using aqueous standards. The LOD value for zinc was found to be 3.00 ng mL⁻¹. No spectral interference was detected at the working wavelengths of the analytes.

4.5. Solid surface molecular fluorescence (14)

Under optimal conditions, the limits of detection and quantification were 0.36×10^{-3} and $1.29 \times 10^{-3} \mu g L^{-1}$, respectively, and the linear range from 1.29×10^{-3} to $4.50 \mu g L^{-1}$. It was applied to the determination of zinc in foods and tap water. The absence of filtration reduced the consumption of water and electricity.

4.6. Inductively coupled plasma mass spectroscopy (11)

The LOD of zinc was found to be 30ng/l.

Sample	Certified value of Zn (µg/g)	Found value Zn(µg/g)	
Wheat flour SRM 1567a	11.6±0.4	11.0±0.5	
Whole egg powder 8415	67.5±7.6	73±4	
Rice flour 1568a	19.4±0.5	20.1±0.6	
Bovine muscle powder 8414	142±14	155±6	
Typical diet 1548a	24.6±1.79	22.9±0.2	

 Table:4.6.1 certified value and found value of zinc in various samples by ICP-MS

4.7. Size exclusion chromatography-inductively coupled plasma mass spectroscopy (13)

Good coefficient of variation (CV) of the elution time (0.06-0.9%), concentration (4.7–16.9%) and molecular size (0.4–5.4%). The limit of detection (LOD) and the limit of quantitation (LOQ) were 0.9 μ g L⁻¹ and 2.8 μ g L⁻¹, respectively

4.8. Voltammetry (10)

Lab	Food samples(g)	Zinc recovery%
1.	(a) whole milk,20	82
		107
	(b) peas, 20	93
	_	84
	(c) wheat flour, 5	102
		75

	(a) hamburger, 10	95
2.	_	141
	(b) orange juice, 20	101
		97
	(c) raw potatoes	99
	_	101
3.	(a)processed cheese, 10	89
		112
	(b)granulated sugar, 10	102
		90
	(c)head lettuce, 20	70
		85

Table: 4.8.1 food samples and zinc content by voltammetry



Figure :4.8.2volta gram of zinc

The quantitation limit of zinc was found to be 50ng/l

4.9. Extractive spectroscopy (16)

The Zn (II)–ECCT complex shows maximum absorbance at 420 nm with molar absorptivity and Sandell's sensitivity being 1.55×10^4 lit mol⁻¹cm⁻¹ and 4.212×10^{-3} µg cm⁻², respectively. The system obeys Beer's law in the range of 0.4–6.0 mg/l, with an excellent linearity in terms of correlation coefficient value of 0.999. Most of the common metal ions generally found associated with zinc do not interfere. The repeatability of the method was checked by finding relative standard deviation (RSD).

4.10. Flourimetry (17)

The developed methodology provided a limit of detection of 1 μ gL⁻¹ in sample acid digests, with a working range of 10 to 200 μ g L⁻¹, corresponding to 100–2000 mg of Zn per kg of dry dog food samples. Intraday repeatability and inter day repeatability were assessed, with relative standard deviation values < 3.4% (100 μ g L⁻¹) and <11.7% (10 μ g L⁻¹).



Figure: -4.10.1 Fluorescence intensity of FluoZin-1 probe at different pH values (phosphate buffer)

Sample	Proposed method result	Absolute deviation	RD (%)
А	393±14	36	10.2
В	472±13	51	12.1
С	336±10	-6	-1.8
D	433±25	55	14.6
Е	279±20	-7	-2.6

F	297±43	41	15.8
G	225±9	-18	-7.4
Н	228±19	-15	-6.2

Table: - 4.10.2 samples of food and their zinc content by fluorimetry

4.11. Ion chromatography (19)

Ion chromatography microwave and leaching assisted result

Sample	Microwave	Leaching
Animal blood	12.6±0.6	12.5±0.2
	n=8	n=4
Dona white flour	21.5±0.3	24.7±2.0
	n=3	n=3
Sorgham red flour	20.5±1.2	24.6±0.6
-	n=7	n=7

Table: -4.11.1 sample and zinc content by ion chromatography assisted by microwave and leaching

4.12. Microwave induced plasma optical emission spectroscopy (20)

The recovery of zinc was found to be 99.7% and RSD 4.4%

LIMITS	ZINC
Detection Limit	0.05
Quantitation Limit	0.15

Table: -4.12.1 zinc detection limit and quantitation limit by MIP-OES



Figure: -4.12.2 1946srm result control chart for zinc

4.13. UV Visible Spectroscopy⁽¹⁸⁾

The calibration graph was linear in the range of 0.5-90.0 mg L1 of Zn2b (r¹/₄0.9996). An enrichment factor of 80 was achieved and the limit of detection for Zn2b was 0.33 mg L⁻¹. The relative standard deviation (RSD) for eight replicate measurements of 10 mg L⁻¹ and 60 mg L⁻¹ of Zn2b was 1.8%

and 1.5% respectively. The proposed method was successfully applied to the quantitative determination of Zn2b in tap water, powder milk and Zinc sulfate tablet with satisfactory results.



Figure: -4.13.1Absorption spectra of(A)ZndithizonatestrippedfromTiO2-NPs and (B) the blank.



Figure: -4.13.2 Influence ofpHondeterminationof40 mg L⁻¹ of Zn2p with CPE-SPE using TiO2-NPs in micellar media



Figure: -4.13.3Influence of electrolyte(A)CaCl2, (B)KNO3 and (C)NaCl on determination of 40 mg L_1 of Zn2b with CPE–SPE usingTiO2-NPs in micellar media



Figure: -4.13.4UV-visible absorption spectra Zn dithizonate stripped fromTiO2-NPs at different concentrations and the corresponding calibration curve

5.CONCLUSION

The review is complete overview of different methods used for the estimation of zinc in various food samples. To conclude that Dry ash voltammetry-CSDPV (Cathode stripping differential pulse voltammetry provided reliable alternative compared to atomic absorption spectroscopy. ICP-MS effective recovery of analytes routinely scanned 8h/day. MIP-AES is a novel instrument uses only nitrogen extracted from air by nitrogen generation is an advantage compared to ICP and AAS.

Solid surface fluorescence there is absence of filtration step. SEC-ICP-MS is a reliable for complex matrices and show consistency in vegetables as well as soil.

FAAS is an inexpensive, precise having LOQ sufficient for determination. In the present review the AAS analysis is done using PBN reagent for trace zinc determination. Atomic fluorescence spectroscopy there is use of cetyltrimethylammonium bromide (CTAB) that provides organic media for hydride generation. Fluorimetry is low cost and fast technique. UV-Visible spectroscopy provides better relative specificity and high preconcentration of Zn^{+2} ion. These methods were evaluated based on accuracy, precision, relative standard deviation.

REFERENCES:

- 1. Office of Dietary Supplements, National Institute of Health, "Zinc-health professional fact sheet", August 2022, https://ods.od.nih.gov/factsheets/Zinc-HealthProfessional/#h3
- 2. Office of Dietary Supplements, National Institutes of Health," Dietary Supplement Label Database" 2021,
- https://ods.od.nih.gov/Research/Dietary_Supplement_Label_Database.aspx
- Brnić M, Wegmüller R, Melse-Boonstra A, Stomph T, Zeder C, Tay FM, Hurrell RF. Zinc absorption by adults is similar from intrinsically labeled zinc-biofortified rice and from rice fortified with labeled zinc sulfate. Journal of Nutrition. 2016;146(1):76-80.
- 4. EFSA Panel on Dietetic Products, Nutrition and Allergies (NDA). Scientific opinion on dietary reference values for zinc. Europeon Food Safety Authority Journal. 2014;12(10):3844.

- 5. Kaur K, Gupta R, Saraf SA, Saraf SK. Zinc: the metal of life. Comprehensive Reviews Food Science and Food Safety. 2014;13(4):358-76.
- 6. Sanna A, Firinu D, Zavattari P, Valera P. Zinc status and autoimmunity: a systematic review and meta-analysis. Nutrients. 2018;10(1):68.
- 7. Gammoh NZ, Rink L. Zinc in infection and inflammation. Nutrients. 2017;9(6):624.
- 8. Drug profile, "Zinc compounds", September 2022. https://go.drugbank.com/categories/DBCAT001980
- 9. Sharma AK, Singh I. A rapid spectrophotometric method for trace determination of zinc. Food Analytical Methods. 2009;2(4):311-6.
- 10. Jones JW, Gajan RJ, Boyer KW, Fiorino JA. Dry ash-voltammetric determination of cadmium, copper, lead, and zinc in foods. Journal of the AOAC. 1977;60(4):826-32.
- 11. Nardi EP, Evangelista FS, Tormen L, Saint TD, Curtius AJ, de Souza SS, Barbosa Jr F. The use of inductively coupled plasma mass spectrometry (ICP-MS) for the determination of toxic and essential elements in different types of food samples. Food Chemistry. 2009;112(3):727-32.
- 12. Ozbek N, Akman S. Method development for the determination of calcium, copper, magnesium, manganese, iron, potassium, phosphorus and zinc in different types of breads by microwave induced plasma-atomic emission spectrometry. Food chemistry. 2016;200:245-8.
- 13. Dell'Aquila C. Development of a method for the detection of zinc in Brassica oleracea using solid phase extraction and sizeexclusion chromatography inductively coupled plasma mass spectrometry (SEC-ICP-MS). MethodsX. 2021;8:101428.
- 14. Talio MC, Acosta MG, Acosta M, Olsina R, Fernández LP. Novel method for determination of zinc traces in beverages and water samples by solid surface fluorescence using a conventional quartz cuvette. Food Chemistry. 2015;175:151-6.
- 15. Sun H, Suo R, Lu Y. Determination of zinc in food using atomic fluorescence spectrometry by hydride generation from organized media. Analytica Chimica Acta.2002;457(2):305-10.
- Reddy KJ, Kumar JR, Ramachandraiah C, Thriveni T, Reddy AV. Spectrophotometric determination of zinc in foods using Nethyl-3-carbazolecarboxaldehyde-3-thiosemicarbazone: evaluation of a new analytical reagent. Food chemistry. 2007;101(2):585-91.
- 17. Martins RC, Pereira AM, Matos E, Barreiros L, Fonseca AJ, Cabrita AR, Segundo MA. Miniaturized Fluorimetric Method for Quantification of Zinc in Dry Dog Food. Journal of Analytical Methods and Chemistry. 2020;2020.
- 18. Pourreza N, Naghdi T. Combined cloud point-solid phase extraction by dispersion of TiO2 nanoparticles in micellar media followed by semi-microvolume UV-vis spectrophotometric detection of zinc. Talanta. 2014;128:164-9.
- 19. Fredrikson M, Carlsson NG, Almgren A, Sandberg AS. Simultaneous and sensitive analysis of Cu, Ni, Zn, Co, Mn, and Fe in food and biological samples by ion chromatography. Journal of Agriculture and Food Chemistry. 2002;50(1):59-65.
- 20. Gallego R, Peñuela GA, Ramírez B. Method validation for the determination of mercury, cadmium, lead, arsenic, copper, iron, and zinc in fish through microwave-induced plasma optical emission spectrometry (MIP OES). Food Analytical Methods. 2017;10(10):3407-14.
- 21. Ferreira HS, Santos AC, Portugal LA, Costa AC, Miró M, Ferreira SL. Pre-concentration procedure for determination of copper and zinc in food samples by sequential multi-element flame atomic absorption spectrometry. Talanta. 2008;77(1):73-6.
- 22. Fulgoni III VL, Keast DR, Bailey RL, Dwyer J. Foods, fortificants, and supplements: where do Americans get their nutrients? Journal of Nutrition. 2011;141(10):1847-54.
- 23. US Department of Agriculture, Agriculture Research Service, "Food Data Central", 2019. https://fdc.nal.usda.gov/