

Application of atomic spectroscopy for trace element analysis of fruit juices: a review

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Abstract. Trace elements are crucial for human nutrition, requiring their precise analysis in fruit juices to ensure product quality and assess contamination risks. Atomic spectroscopy techniques including inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma optical emission spectrometry (ICP-OES), graphite furnace atomic absorption spectrometry (GFAAS), flame atomic absorption spectrometry (FAAS), atomic fluorescence spectrometry (AFS), X-ray fluorescence spectrometry (XRF), and glow discharge optical emission spectrometry (GD-OES) are sensitive, selective and versatile tools for trace element analysis of various solid and solution samples. Matrix modifiers, sample introduction and sample preparation methods are pivotal for improving the accuracy and mitigating matrix interferences. Further advancements in instrumentation are essential. This review provides a comprehensive overview of these techniques, highlighting their principles, advantages, limitations and future research directions in fruit juice analysis. Its global applications, focusing on As, Cd, Co, and Pb, along with sample preparation methods, element concentrations, detection limits, and recovery values, have been explored.

1 Introduction

Fruits are crucial to the human diet, offering essential macro and micronutrients, phytochemicals, and dietary fibre vital for overall health. Regardless of age, fresh and commercially available fruit juices often contribute to the daily nutritional intake. In addition to naturally present trace elements, fruits and fruit produced throughout the supply chain are highly susceptible to contamination by trace elements, including heavy metals.

Metal analysis studies on fruit juices have been based on Al, As, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Se, Sn, and Zn including both nutritive and hazardous elements. Although most trace elements are beneficial (Cu, Cr, Co, Fe, Mn, Se, Zn), contamination may lead to intolerable levels in a fruit serving or containing excess amounts to the risk of trace metal element accumulation in human body tissues. Due to the small difference between allowable and

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harmful levels of trace elements, assessing possible key contaminant levels with good accuracy in fruit juices is vital for ensuring consumer safety and product confidence.

Among numerous methods of trace element analysis at industrial and laboratory levels, atomic spectroscopy techniques have gained widespread popularity in the food industry due to their feasibility, accuracy, robustness, and analytical speed. This review aims to enhance the understanding of atomic spectroscopy methods in trace element analysis of fruit juices aiding in method selection and advancing comprehension in this field.

2 Materials and methods

2.1 Atomic spectroscopic techniques for trace element analysis of fruit juices

2.1.1 Inductively coupled plasma optical emission spectroscopy and inductively coupled plasma mass spectroscopy

Inductively Coupled Plasma (ICP) forms by passing an inert gas, usually argon, through concentric quartz tubing surrounded by a radiofrequency coil. A Tesla-spark used to ignite the plasma, which ionises the argon gas and creates a self-sustaining low-temperature plasma (4000-9000 K) that facilitates efficient atomisation, excitation and ionisation of the elements in the sample and photons with distinct wavelengths, which is unique to the specific element [1-3].

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) records the UV-VIS light radiation emitted by certain chemical elements introduced into ICP [1]. For elemental analysis, this method facilitates the direct injection of liquid and aerosol samples into the instrument, but solid samples require extraction or acid digestion before sample introduction [4] or alternative sample introduction methods such as laser ablation (LA) or electrothermal vaporisation (ETV). Fruit juice contains an average level of soluble solids, mainly sugars with a 5-20 Brix value [5]. Owing to solid components, direct solution analysis is difficult, especially for ICP-OES, ICP-MS, and FAAS such as analytical instruments utilising nebulization systems. Consequently, samples were subjected to simple procedures before the analyses as shown in Table 1. In addition, other issues associated with ICP-OES include lower detection limits with solid samples, spectral interferences, high capital investment and high operational costs [1, 3]. These limitations are addressed to some extent by modern techniques, ultrasonic nebulisers, automated background correction software algorithms (ABCA), or alternative methods such as laser induced breakdown spectroscopy (LIBS), and microwave induced plasma (MIP) technology [3]. ICP-OES is a user-friendly, multi-element technique with a wide analytical range that does not require advanced analytical skills from the operator.

Table 1. Characteristics of Atomic Spectroscopy methods in assessing As, Cd, Co, and Pb in fruit juices/beverages.

Metal	Detection Method	Country	Sample Preparation Method	LOD ($\mu\text{g/L}$)	Concentration range (mg/L)	Recovery (%)	Ref.
As	HGAAS	Portugal	MAWD		<LOD-0.1338	84-111	[6]
	ICP-MS	Turkey	MAWD	0.26	<LOD-0.036	88-118	[7]
	GFAAS	Hungary	DA +MM	5	<LOD	61.2 \pm 8.0	[8]
	ICP-OES	Iran	MAWD	0.21	0.00046-0.00747	98	[9]
	ICP-MS	US	WD in hot block		0.00055-0.0126		[10]
	ICP-OES	Brazil	MAWD	12	<LOD	87-94	[11]
	TXRF	Brazil			ND-4.463		[12]

iAs	HG-AFS	China	Filtration + DLLME	1.2	ND-60	92-102	[13]
Cd	FAAS	Egypt	Dry ashing + WD	20	ND		[14]
	GFAAS	Portugal	MAWD		<LOD-0.0034	84-111	[6]
	ICP-MS	Turkey	MAWD	0.05	<LOD-0.008	90-102	[7]
	GFAAS	Hungary	DA + MM	0.03	0.00005-0.0165	96.2±3.1	[8]
	ICP-OES	Iran	MAWD	0.03	0.00012-0.00130	93	[9]
	ICP-OES	Serbia	Dry ashing	328	0.0221-0.1212		[15]
	FAAS	Turkey	DMSP	0.19		91.1-93.5	[16]
	ICP-MS	US	WD in hot block		0.000004-0.016		[10]
	ICP-OES	Yemen	Dry ashing	1.3	ND-0.03		[17]
Co	ICP-OES	Brazil	MAWD	1.2	<LOD	95-97	[11]
	ICP-MS	Turkey	MAWD	0.03	<LOD-1.6	85-95	[7]
	ICP-OES	Serbia	Dry ashing	82.62	0.0111-0.3296		[15]
	ICP-MS	US	WD in hot block		0.00003-0.410		[10]
	ICP-OES	Brazil	MAWD	2.8	<LOD	93-95	[11]
	TXRF	Brazil			ND-59.85		[12]
Pb	FAAS	Egypt	WD	100	ND		[14]
	GFAAS	Portugal	MAWD		<LOD-0.00851	84-111	[6]
	ICP-MS	Turkey	MAWD	0.14	0.0001-0.042	102-109	[7]
	ICP-OES	Iran	MAWD	0.92	0.00088-0.0039	97	[9]
	GFAAS	Hungary	DA	0.8	0.006-0.090	98±2.3	[8]
	GFAAS	Thailand	MAWD + Solvent terminated DLLME	0.025	0.2-2.79	86.3-115	[18]
	ICP-MS	US	WD in hot block		0.0000014-0.006		[10]
	ICP-OES	Yemen	Dry ashing	23.3	ND-8.07		[17]
	ICP-OES	Brazil	MAWD	3.4	<LOD-0.0134	89-91	[11]

Notes: WD: Wet Digestion, MAWD: Microwave Assisted Wet Digestion, DA: Direct Analysis, DLLME: Dispersive Liquid-Liquid Microextraction, DMSP: Dispersive micro-solid-phase extraction, iAs; Inorganic arsenic, LOD: Limit of detection, ND: Not detected, MM: Matrix modifiers.

ICP-MS is an interactive combination of ICP and mass spectrometer that produces singly charged ions and separates and measures them based on the mass-to-charge ratio (m/z) [1]. ICP-MS offers more advantages over a few drawbacks such as extreme initial and operating costs, and low tolerance of high matrix components. Despite these drawbacks, it comprises multi-element capabilities with a wider working range without losing linearity, and better detection limits, lower or similar to GFAAS. Furthermore, ICP-MS facilitates the measurement of elemental isotopic concentrations and ratios, which are important for identifying the carcinogenicity or genotoxicity of elements present in fruit juice [1,19,20]. In addition, ICP-MS is more productive when coupled with hyphenated techniques, which combine separation techniques generally a chromatographic separation such as liquid chromatography (LC) and high-performance liquid chromatography (HPLC). Other than these commonly used LC and HPLC, ion chromatography and capillary electrophoresis (CE), and field flow fractionation (FFF) have also been used in studies. In this integrated setup, the fractionation device separates the elements and ICP-MS performs detection, thereby enhancing the accuracy, precision, selectivity and sensitivity of the method [21]. Another benefit of this coupling is that it bypasses the digestion step [22]. ICP-OES and ICP-MS use ICP the same plasma source. However, in ICP-OES the ions formed in the plasma are directly measured via optical detection, whereas in ICP-MS the ions are transferred into a mass spectrometer employing usually a radio-frequency quadrupole. ICP-OES is not as ideal as ICP-MS coupled with chromatographic techniques because the mass spectrometer provides more specific and sensitive hyphenation techniques than ICP-OES [23]. Additionally, chemical vapour generation (CVG) has been utilised as a sample introduction technique to

enhance sample introduction efficiency and eliminate matrix interference in atomic spectroscopy for trace element determination. CVG is commonly used in arsenic analysis, including hydride generation (HG), electrochemical HG, halide generation, and chelate generation. [24]. Photochemical vapour generation (PVG) is a promising CVG technique that was recently discovered [25]. This area should be considered in further studies with specific elements and different matrices, such as fruit juices.

2.1.2 Graphite furnace atomic absorption spectroscopy

Graphite furnace atomic absorption spectrometry (GFAAS) involves intake of an analyte into an electrically heated tube-shaped graphite furnace atomiser. This atomiser processes the sample through drying, pyrolysis and atomisation steps, where drying and pyrolysis remove the solvent and matrix salts as vapours. Subsequently, free atoms are produced in the optical path during atomisation. The instrument then measures the attenuation of the primary radiation beam by these atoms, based on light absorption [26-27]. GFAAS offers low detection limits in the $\mu\text{g/L}$ range for solutions and can analyse small samples typically 20-50 μL per cycle, with well-documented applications and moderate initial cost. Moreover, GFAAS facilitates a more reliable direct analysis of samples, including powdered solid samples without prior sample preparation. Matrix interferences in the direct analysis of fruit juices and fruit-based beverages are minimized by combining simple sample preparation procedures with appropriate sample introduction techniques, proper matrix modifiers and optimised heating programs [8, 28-30]. This is a prospective area for further study and development. Compared to other methods, the major drawbacks associated with GFAAS are lower sample throughput and limited working range [1].

The primary instrumental techniques for GFAAS are line source atomic absorption spectrometry (LS-AAS) and high-resolution continuum source atomic absorption spectrometry (HR-CS-AAS). Initially, LS-GFAAS was limited to analysing 2-6 elements simultaneously due to sensitivity reduction caused by optical beam splitting [31]. Recent advancements have enhanced the sensitivity of multielement analysis in GFAAS using an echelle polychromator, which has high spectral resolution and is powered by a high-pressure xenon arc lamp. Subsequently, comprehensive multielement analysis was performed using a laser-driven xenon arc lamp and linear charge coupled device (CCD) detector for true multielement analysis [32]. Furthermore, modifying the wavelength, applying less sensitive secondary spectral lines and incorporating appropriate chemical modifier combinations, such as operational practices, have contributed to the improvement of multielement analysis using GFAAS [8, 33, 34].

GFAAS is less susceptible to spectral and non-spectral interferences compared to FAAS, ICP-OES, and ICP-MS, owing to modern instruments equipped with transversely heated graphite tube atomisers, longitudinal Zeeman-effect background correction and L'vov-platform which minimise interferences and encourage complete atomization for accurate measurements [28, 31, 35]. Modern GFAAS instruments often use stabilised temperature platform furnace (STPF) concept, a widely used methodological advancement since the 1980s [36]. Such features make GFAAS suitable for analysing fruit juices, which have moderate solid content, although it has lower sample throughput than methods like ICP-MS and ICP-OES [1].

2.1.3 Flame atomic absorption spectroscopy

Flame Atomic Absorption Spectrometry (FAAS), uses premixed air/acetylene or nitrous oxide/acetylene flames to vaporise and atomise samples into free atomic vapour. Element concentrations are then measured based on the light absorbance of the ground-state atoms.

FAAS is noted for its simplicity, flexible handling, and lower costs than other techniques. However, its single-element analytical capability and lower sensitivity are due to analyte losses from nebulised aerosols before reaching the flame and short analyte residence time in the optical path [2, 37]. To enhance FAAS sensitivity, flame furnace atomic absorption spectrometry (FF-AAS) has been developed, incorporating beam injection, thermospray sample injection, and pneumatic nebulisation to reduce atom loss and extend residence times [25].

2.1.4 Atomic fluorescence spectroscopy

Atomic Fluorescence Spectroscopy (AFS) integrates principles from atomic absorption and emission techniques and offers a comprehensive approach to elemental analysis. This process begins by atomising a sample in a flame and then exciting these atoms with a focused light beam to measure subsequent absorption and emission to determine element concentration. AFS is considered a robust technique owing to its excellent sensitivity and selectivity even for arsenic and mercury. Furthermore, it supports speciation analysis when coupled with separation techniques. Lai et al. conducted a study on the speciation of As(III) and As(V) in fruit juices using ion-pairing dispersive liquid-liquid microextraction (DLLME) and hydride generation atomic fluorescence spectroscopy (see Table 1). They obtained promising results that proved the applicability of this method for arsenic analysis in most fruit juices except pear juice because it contains a significant amount of monomethyl arsenic acid which causes erroneous signals [13]. Furthermore, AFS is an inexpensive analytical technique compared to other common speciation instruments, ICP-MS and ICP-OES, however, it requires advanced operator skills compared to FAAS and ICP-OES.

2.1.5 X-ray fluorescence spectroscopy

In X-ray Fluorescence Spectrometry (XRF), samples are ionized by X-rays that cause electrons to move from inner to outer orbitals, emitting characteristic fluorescence with unique energy peaks of different intensities [38, 39]. XRF is non-destructive, rapid and requires minimal sample preparation, offering multielement analysis at low operational costs. This provides qualitative and quantitative measurements for a large set of elements ($Z > 11$). This method has been used for fruit juice and other food analyses in fewer studies [38]. XRF is sensitive to the surface roughness, necessitating homogenisation, drying, grinding, and pelletising of samples, when organic soluble solid substances in samples allow for carbonisation to form solid residues [40]. Therefore, XRF becomes destructive and time-consuming when sample preparation must be an option [38, 41, 42]. XRF includes wavelength dispersive (WD) and energy dispersive (ED) techniques [42]. Total Reflection Fluorescence Analysis (TXRF) is an energy dispersive XRF (EDXRF) technique that is particularly suitable for liquid samples such as fruit juices and is commonly used for trace element analysis [42, 43].

2.1.6 Glow discharge optical emission spectroscopy

Electrolyte cathode atmospheric glow discharge (ELCAD) with OES detection is a plasma spectroscopy technique for direct solution analysis [44]. It generates atmospheric pressure plasma discharge between an electrode and the sample surface, causing atoms to emit characteristic light at specific wavelengths. Gorska and Pohl (2021) utilised flowing liquid cathode GD-OES, a variant of ELCAD to analyse fruit juices for macro minerals Ca, K, Mg, and Na [45]. They chose the flow liquid cathode atmospheric pressure glow discharge (FLC APGD) because of its lower detection limits compared with other similar techniques. This

method involved simple sample preparation, including dilution and acidification with 0.1 mol/L HNO₃ without extensive digestion. The achieved LODs were comparable to or better than those obtained by ICP-OES, with similar precision [45]. Importantly, the instrument's small size, low electric power consumption and the unnecessary of noble gases make it more attractive. Although its sensitivity is lower than GFAAS and ICP-MS, its significant advantages in terms of time-saving, environmental friendliness, and cost effectiveness, open up opportunities for future research and application in trace metal analysis.

2.2 Sample preparation

Table 1 presents common sample preparation techniques associated with atomic spectroscopy analysis of fruit juices. Because of the heterogeneous composition of fruit juices, centrifugation and filtration are important for eliminating the physical interferences from solid particles [4]. Matrix interferences arising from the complex nature of fruit juices may cause alterations in signal detection, with common issues including spectral overlap and ionization effects. The dry ashing method removes bound organic compounds by converting them into CO₂ and H₂O. Alternatively, wet digestion methods involving acid or acid mixtures in open vessels in block heaters or closed vessels with thermal or microwave heating help minimise element loss and mitigate high temperatures [7,10]. Despite these benefits, dry ashing and wet digestion are time intensive and pose contamination risks. Microwave-assisted wet digestion is a frequently used wet decomposition method owing to its efficiency, short run time and low acid use. Direct analysis techniques primarily favoured by GFAAS and XRF, often involve simple procedures such as filtration, centrifugation and dilution. Over time, extraction, separation and preconcentration techniques have evolved from conventional liquid-liquid extraction to solid phase extraction and microextraction, aligning with green chemistry principles for thorough sample analysis and quantification.

2.3 Method validation and quality assurance

Method validation evaluates the linearity, accuracy, precision, sensitivity and specificity. Linearity is assessed through calibration curves using standard solutions across expected concentration ranges in fruit juices, and linearity is verified using the correlation coefficient from regression analysis [7]. The accuracy is ensured by analysing standard reference materials and obtaining recovery values from spiking experiments, which enables an understanding of element loss during pretreatment and the matrix effect. Because of the absence of standard reference material for fruit juice, alternative matrices such as natural water have been used [7]. Furthermore, comparative analysis with other spectroscopic methods enhances accuracy validation [7, 11]. The sensitivity of an analytical method is determined by its Limit of Detection (LOD) and Limit of Quantification (LOQ) values. Table 1 shows how LOD values vary according to the element, analytical method, and sample preparation technique. As shown in Table 1, GFAAS and ICP-MS demonstrate the lowest LOD values, indicating higher sensitivity. The lowest LOD values in these studies were achieved using microwave-assisted wet digestion. Method validation is vital for ensuring the reliability and robustness of the analytical method.

2.4 Future perspectives and emerging trends

Future research on atomic spectroscopy for trace element analysis of fruit juices should focus on integrating emerging technologies such as nanotechnology and machine learning to improve sensitivity and data interpretation. Developing a non-destructive or direct analysis

method with automated sample preparation will reduce the risk of sample contamination. Improving speciation analysis to differentiate and quantify various forms of elements would provide more detailed insights into nutritional and toxicological impacts. Importantly, developing specific standard reference materials for fruit juices would enhance the reliability of trace element analyses. Additionally, reducing or eliminating hazardous chemicals in the analysis process aligns with green chemistry principles, decreasing environmental impact and improving worker safety. These advancements will ensure more accurate, efficient and environmentally friendly analyses.

3 Conclusion

Atomic spectroscopy plays a vital role in analysing trace elements in fruit juices, assessing their nutritional value, and detecting contamination. These methods including ICP-OES, ICP-MS, GFAAS, FAAS, AFS, XRF, and GD-OES provide various advantages in terms of sensitivity, selectivity, speciation analysis and multielement analysis capabilities. Matrix modifiers, sample introduction and sample preparation methods are important for overcoming matrix complexities and ensuring accurate measurements. Advancements in instrumentation and sample preparation techniques would promise further improvements in sensitivity, efficiency and eco-friendliness. This holistic approach contributes to the understanding of nutrition, food safety, and public health.

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