Assessment of Trace Elements in Roasted Oats and Flaxseeds by the Means of ICP-MS

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ABSTRACT

Oats and flaxseeds are uniquely nutritious foods. They have been well recognised as functional food with nutraceutical properties, offering health benefits due to their high levels of bioactive and antioxidant compounds. Interestingly, roasting of oats and flaxseeds helps to increase their functional properties and digestibility. While their nutraceutical and nutritional features have been studied, the possibility of authenticating them with respect to processing has not been explored. In this work, we have studied the micro-elemental composition of roasted oats and flaxseeds using microwave digestion and ICP-MS as a chemical descriptor. The study mainly focuses on the tracing of elements such as calcium, magnesium, sodium, phosphorus, zinc and iron for authentication.

Keywords: oats, flaxseeds, elements, ICP-MS, digestibility.

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1. Introduction

Minerals are essential for normal human physiological functions. However, humans require minerals in small quantities, especially compared to macronutrients like carbohydrates and protein. [1] Minerals can be grouped into two categories: trace minerals and major minerals. The major minerals in a diet include calcium, magnesium, sodium, sulfur, phosphorus, potassium, and chloride.

Trace or minor minerals include iron, iodine, zinc, fluoride, selenium, copper, chromium, magnesium, and myrobalan. In a diet, the requirement for major minerals is nearly 100 milligrams or more per day, while less than 100 milligrams per day of trace minerals is sufficient for normal human functions. [2] Iron is a key component of haemoglobin, which is responsible for transporting oxygen, and myoglobin. Furthermore, iron is part of many enzymes needed for metabolic processes. It influences the composition of gut microbiota, which obtains

energy from food. Normal intestinal microbiota converts ellagic acid to urolithin A, thus increasing dietary iron availability. However, free iron in the gut can form free radicals and cause oxidative stress, but the presence of bifidobacteria helps reduce free radicals and supports gut health. Calcium is a major structural element of bones and teeth. Additionally, it plays a vital role in various physiological processes, including nerve conduction, muscle contraction, blood clotting, inflammatory responses, and hormonal secretion. Magnesium is also a component of bones and participates in nerve transmission, muscle contraction, and ribosomal activities. It is the second most abundant positively charged mineral that helps bacteria to survive at higher temperatures in the gut, such as Lactobacillus rhamnosus GG, Lactobacillus casei Zhang, Lactobacillus Plantarum P-8 and supports the heat shock survival mechanism. Likewise, phosphorus aids in forming bones, teeth, nucleic acids, and energy-carrying molecules such as ATP. It also helps maintain the body's acid-base balance. Moreover, zinc is another important element involved in nucleic acid metabolism and signal transduction. [1] Even a mild deficiency of zinc can profoundly impact growth and development, especially during the developing stages of life when immune differentiation and maturation are at their peak. In addition, an animal study concluded that a deficiency of zinc may affect gut health by increasing GI inflammation and overall decline in GI health. [2] Sodium helps in maintaining osmotic balance and water distribution across different body fluid compartments. It plays a key role in maintaining pH, the function of the heart and muscles, and electron transport reactions, and also acts as a cofactor for many enzymes. [3]

In recent years, there has been growing interest in the functional foods that provide health benefits beyond their basic nutrition. Oats and flaxseeds are well recognised for their nutritional and functional properties. Oats are a rich source of beta-glucan and dietary fibre, contributing to improving gut health and protecting against colon cancer. [4] On the other hand, flaxseeds are a rich source of omega-3 fatty acids and have a high content of protein and dietary fibre. [5] However, their mineral composition in the roasted form is not well known. However, it is observed that milling of grains helps to improve their digestibility. [6]

Inductively coupled plasma mass spectrometry (ICP-MS) is a highly sensitive, reliable, robust analytical technique for the determination and quantification of elemental composition. It is also used to measure the concentration and isotopic abundances of various matrices. [7]

The study mainly focuses on describing the quantification and tracing the mineral content in roasted oats and flaxseeds using microwave digestion and ICP-MS. There are six components of ICP-MS. It consists of a sample introduction system, an ion source, the electrostatic lenses, an interface, mass spectrometry, and a detector. [8]

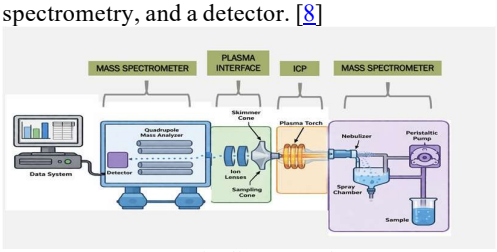


Figure 1: Systematic diagram of ICP-MS components

Materials and Methods

2.1 Chemicals

To prepare the standard solution and sample, ultrapure water (15 ml) was obtained from Milli-Q (18.2 MΩ·cm) and a Q water purification unit (Institute of Technology, Central Instrument Facility, Banaras Hindu University, Varanasi, In-dia). Ten millilitres of HNO3 were used for the analysis of the microelements in the sample.

2.2 Sample Collection

Flaxseeds and rolled oats were purchased from an online store (named Manna Oats and Bliss of Earth flaxseeds). These were 100 mg samples of roasted and ground oats and flaxseeds used for the analysis.

2.3 Sample Treatment

The pre-treatment of the samples involved roasting oats and flaxseeds at low to medium heat for 10 minutes. After roasting and cooling at room temperature, the samples were ground using a mixer grinder and then packed into airtight packets for further laboratory testing.

2.4 Sample Preparation

For microwave digestion and ICP-MS analysis, 100 mg of the sample was weighed and placed in a clean, dry microwave digester vessel. Then, 5 mL of HNO3 was added to convert the solid samples into a liquid state. The mixture was left to sit for 10-15 minutes to allow initial gas escape, reducing the risk of pressure spikes during heating. The vessel cap was then securely closed and placed in the microwave digestion system.

Typically, the microwave digestion was set at 180°C for 25 minutes and maintained at the same temperature for an additional 20 minutes. After cooling, the digested samples were transferred into vials for ICP-MS analysis.

Calculations for standard preparation:

- Sample weight = 100 mg
- Digestion = 5 mL HNO3
- Dilution = +15 mL water
- Final volume = 20 mL (0.02 L)

For ICP-MS instrument setup:

Argon and helium gases were used primarily. Argon, exhaust, and chiller systems were turned on at least 15–30 minutes before warm-up and thermal stability. For plasma ignition, the instrument was operated at a 6000–10,000 K plasma torch. Helium was also used to minimise polyatomic interference. The liquid sample was introduced into the nebuliser, where a high-velocity argon gas broke the sample into a fine mist or aerosol. This aerosol entered a spray chamber that filtered out larger droplets, sending the smaller droplets into the plasma. At the base of the argon plasma torch, the solvent evaporates instantly due to the high heat, converting into microscopic solid particles. The plasma atomises the molecules into atoms, which then react with high-energy electrons to produce singly charged positive ions. These ions are channelled into the high-vacuum interface of the mass spectrometer. The mass analyser then separates the ions based on their mass-to-charge (m/z) ratio, detects these ions, and the software calculates the mineral concentrations in the sample.

Parameters	Analytical values
Nebulizer gas flow	0.90 L/min
Radio frequency powder	1100 W
Plasma gas flow (Argon)	50 L/min
Reaction gas (Helium)	50 L/min

Table 1: System operating parameters for ICP-MS.

2.1 Validation Parameters.

The ICP-MS mineral analysis procedure was validated for reliability and consistency. Calibration was conducted using standard solutions at concentrations of 0.05 ppm, 0.1 ppm, 0.5 ppm, and 1 ppm to ensure accurate element quantification. The instrument response showed a linear relationship within this concentration range. Precision was evaluated by performing triplicate measurements, with results expressed as relative standard deviation (RSD). RSD values were less than 5% for most elements, indicating good repeatability and precision.

2.6 Statistical Approach

Descriptive statistics, including mean, RSD, and standard deviation, were derived from ICP-MS data. The method's reproducibility was confirmed by RSD values below 5%, indicating consistent measurements.

2. Results

The result of the mineral composition in the roasted oats and flaxseeds has been summarised in Tables 2 and 3. The outcome was expressed as mean, relative standard deviation and standard deviation in tables 4 and 5. The results obtained from ICP-MS readings of Conc were in the form of ppm; hence, for further calculations, they were converted into mg/L in solution. The formula used for conversion was mg/kg = C*V/W. Here = C stands for conc (PPM), V stands for final volume, and W stands for weight of the sample in kg. (W = 100 mg = 0.1 gm = 0.0001 kg.) Hence, the calculated values of ICPMS for mineral elements present in oats are given below in Table 2.

Table 2. Mineral composition of roasted oats (mg/100g):

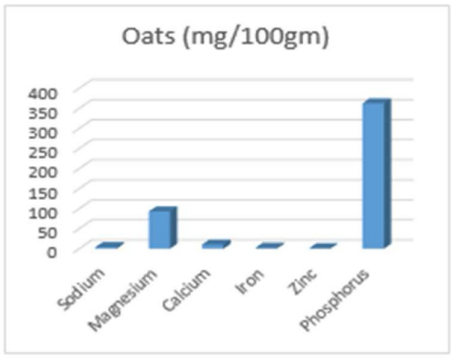
Elements (mineral)	ICP-MS Conc. (ppm)	Oats (mg/100gm)
Sodium	**0.205**	**4.10**
Magnesium	**4.645**	**92.90**
Calcium	**0.519**	**10.38**
Iron	**0.134**	**2.68**
Zinc	**0.063**	**1.26**
Phosphorus	**18.136**	**362.72**

Table 3. Mineral composition of roasted flaxseeds (mg/100g)

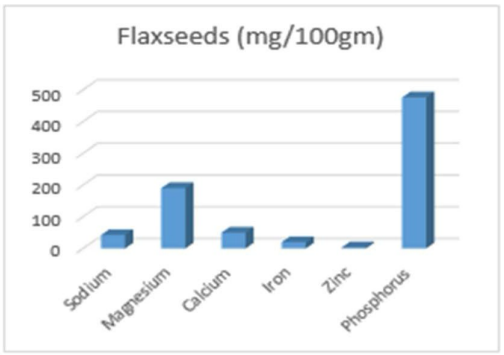
Elements (mineral)	ICP-MS Conc. (ppm)	Flaxseeds (mg/100gm)
Sodium	2.124	42.48
Magnesium	9.518	190.36
Calcium	2.494	49.88
Iron	0.992	19.84
Zinc	0.173	3.46
Phosphorus	23.805	476.10

Table 4: Descriptive statistics for roasted oats (mg/100g):

Elements	Mean (mg/100g)	RSD (%)	SD
Sodium	42.48	1.4	0.59
Magnesium	190.36	3.8	7.23
Calcium	49.88	0.8	0.40
Iron	19.84	0.9	0.18
Zinc	3.46	2.2	0.08
Phosphorus	476.10	5.1	24.28



(a)



(b)

Figure 2. Mineral composition of flaxseeds and oats (mg/100g)

Element	Mean (mg/100g)	RSD (%)	SD
Sodium	4.10	5.5	0.23
Magnesium	92.90	0.4	0.37
Calcium	10.38	2.7	0.28
Iron	2.68	0.8	0.02
Zinc	1.26	2.9	0.04
Phosphorus	362.72	4.1	14.87

Table 5: Descriptive statistics for roasted flaxseeds (mg/100g):

3. Discussion

The interpretation of ICP-MS analysis in the present study evaluates the mineral composition. It was observed that roasted oats contain 42.48 mg/100 gm of sodium, 190.36 mg/100 g of magnesium, 49.88 mg/100 g of iron, 3.46mg/100 g of zinc and 476.10mg/100 g of phosphorus. While the results indicate that flaxseeds contain 4.10mg/100 g of sodium, 92.90mg/100 g of magnesium, 10.38 mg/100 g of calcium, 2.68mg/100 g of iron, 1.26mg/100 g of zinc and 362.72 mg/100 g of phosphorus. However, a previous study indicated that fermented oats contain 2.1 mg of zinc, 25.48mg of magnesium, 0.772 mg of iron, 0.54 mg of sodium, 83.6mg of phosphorus, and 9.84mg of calcium per 100ml. [9] Another study revealed that sand roasting of oats affects the physiochemical properties of oat groat. The roasted oat flour showed increased antioxidant properties, reducing power, and chelating activity. However, sand roasting decreases the phenolic and flavonoid content in the oats. Moreover, sand roasting of oats leads to grain size expansion, lowering the length and breadth ratio and bulk density. [10]

Another study reported the microelement composition in 10 different kinds of commercially available oat products. The amount of phosphorus (%) found in crushed oats was 60.9, instant oat bran flakes was 39.7, extruded crisps with bran was 51, oat bran was 41.1, oat groat was 55.1, instant oat flakes was 53.4, oat flakes was 44.4, oats flake polished was 45, extruded oat crips 53.6, and extruded oat and corn crisps was 53.9. The amount of magnesium (%) found in crushed oats was 38, instant oat bran flakes was 16.4, extruded crisps with bran was 22.5, oat bran was 21.5, oat groat was 39.8, instant oat flakes was 30.1, oat flakes was 18.5, oats flake polished was 23.3, extruded oat crips 25.9, and extruded oat and corn crisps was 27.9. The amount of calcium (%) found in crushed oats was 39.5, instant oat bran flakes was 29.8, extruded crisps with bran was 36.4, oat bran was 30, oat groat was 38.8, instant oat flakes was 38, oat flakes was 29.1, oats flake polished was 27, extruded oat crips 28, and extruded oat and corn crisps was 18.2. The amount of iron (%) found in crushed oats was 29, instant oat bran flakes was 10.8, extruded crisps with bran was 8.2, oat bran was 6.5, oat groat was 12.2, instant oat flakes was 10.9, oat flakes was 7, oats flake polished was 9.1, extruded oat crips 15.2, and extruded oat and corn crisps was 8.3. The amount of zinc (%) found in crushed oats was 17.2, instant oat bran flakes was 3.9, extruded crisps with bran was 4.3, oat bran was 0.4, oat groat was 2.8, instant oat flakes was 4.8, oat flakes was -1.9, oats flake polished was -2.9, extruded oat crips -11, and extruded oat and corn crisps was 3.3. [11]

Further, a study done on six different varieties of flaxseeds taken from Punjab, Madhya Pradesh, and Himachal Pradesh, indicates that micro minerals such as zinc and magnesium contribute significantly to the total mineral content. [12]

Moreover, another study reported the mineral composition of flaxseeds and flax sprouts. The flax sprouts contain 0.70 % phosphorus, 0.78% potassium, 0.25% calcium, 0.41% mag-
Magnesium, 0.22% sulfur, 0.07% sodium, 44.39% zinc, 63.44% iron in 100g on a dry basis. While flax seeds contain 0.21 % phosphorus, 0.65% potassium, 0.40% calcium, 0.90% magnesium, 3.42% sulfur, 0.26% sodium, 17.67% zinc, 43.67% iron in 100 g on a dry basis. [13]

Another study demonstrates that the roasting of flaxseeds results in influencing the nutritional quality and oxidative stability of flaxseed oil. Roasting modifies the content of bioactive components, i.e., tocopherol decreases, whereas plastochromanol-8 and Millard reaction products increase. [14]

Similar studies showed the effect of roasting on the mineral profile of flax seeds. Roasting enhances the amount of phosphorus, sodium and potassium. However, it decreases the level of magnesium, calcium, iron and zinc. This research reported that unroasted flaxseeds contain phosphorus of 3125.26 mg/kg, sodium of 730.25 mg/kg, calcium of 998.69 mg/kg, magnesium of 3875.89 mg/kg, potassium of 701.58 mg/kg, iron of 100.58 mg/kg, and zinc of 45.36 mg/kg. while the roasted flaxseeds at 160 degrees Celsius for 5-15 minutes contain phosphorus of 3165.30-3235.14 mg/kg, sodium of 738.54-750.24 mg/kg, calcium of 975.23-950.74 mg/kg, magnesium of 3754.21-3564.21 mg/kg, potassium of 736.10-775.65 mg/kg, iron of 87.42-81.56 mg/kg, and zinc of 43.21-39.54 mg/kg. Moreover, the roasted flaxseeds on 180 degrees Celsius for 5-15 minutes contain phosphorus of 3178.58-3257.69 mg/kg, sodium of 742.57-765.28 mg/kg, calcium of 962.14-841.65 mg/kg, magnesium of 3710.24-3487.25 mg/kg, potassium of 742.31-789.35 mg/kg, iron of 85.67-78.21 mg/kg, and zinc of 42.51-38.31 mg/kg. [15]

4. Conclusions

The present study determines microelements composition by means of the ICP-MS technique and demonstrates that both samples are a rich source of essential minerals such as calcium, sodium, zinc, iron and phosphorus. These findings emphasise the nutritional profile of roasted oats and flaxseeds and their potential role as nutrient-dense functional foods. Regular inclusion of roasted oats and flaxseeds may help in fulfilling the micro- and macro-nutrient needs. Thus, promoting overall health and well-being.

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Abbreviations

The following abbreviations are used in this manuscript:

ICPMS	Inductively coupled plasma mass spectrometry
ATP	Andesine triphosphate RSD Relative Standard deviation

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