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# Determination of different trace and essential element in lemon grass samples by x-ray fluorescence spectroscopy technique

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**Abstract:** The quantitative determination of essential and trace elements via advanced X-ray fluorescence (XRF) Spectroscopic technique, in this response three different lemon grass samples were collected from different fields, among the analysed sample the highest amount of potassium (K) 53.40%, calcium (Ca) 26.19%, and silicon (Si) 10.01% were determined, while the second highest amount of sodium (Na) and magnesium (Mg) were found 2.57% and 2.05% respectively, the remaining were at below 2%. The standard deviations of samples are below than 5%. The validity and accuracy of the optimized procedure was checked by analysis of the certified reference materials (CRM)/literature values by XRF technique.

**Keywords: lemon grass, elements, moisture, ash, XRF**

## Introduction

Composition of food facts is an essential contrivance in many areas of nutrition and food science. The accuracy of nutrient intakes and adequacies depends on food composition data and the insinuation of improving these has been stressed out (Dwyer, 1994; Helsing, 1994; Scrimshaw, 1997). Lemongrass is a perennial herb, commercially cultivated in Guatemala, India, China, Paraguay, Sri Lanka and also cultivated in some areas of Pakistan (Bhan et al., 2005; Hassan et al., 2007; Ibrahim et al., 2009). The plants need a warm, humid climate. Lemon grass grows well in sandy soils with adequate drainage. The propagation is by root or plant division. The plant grows in dense clumps up to 2 meters in diameter and has leaves up to 1 meter long. Lemongrass is used as a fragrance and flavoring for a wide variety of ailments in folk medicine (Negrelle and Gomes, 2007). Freshly cut and partially dried leaves are used medicinally and are the source of the essential oil and also used as lemon tea. Limited studies have demonstrated antifungal and insecticide efficacy, as well as potential anticarcinogenic activity (Onawunmi et al., 1984; Zeng et al., 1993).

X-ray fluorescence (XRF) spectroscopy was proved as one of the simplest, accurate and most economic analytical methods that provide fundamental significant facility for the elemental determination of many types of material, particularly in the investigation of metals for research in geochemistry, biological and food samples. The XRF technique is non-destructive and reliable and requires no or very little sample preparation also suitable for solid, liquid and powdered samples (John et al., 2001). The XRF technique being multi-elemental, fast and cost-effective has the advantage over other techniques such as atomic absorption spectroscopy (AAS), inductively coupled plasma spectroscopy (ICPS) and neutron activation analysis (NAA). Unfortunately these techniques require in general liquid samples, therefore the samples have to be digested, nowadays mostly by utilization of a microwave digestion system. The digestion procedure has to ensure the complete dissolution of the sample. This process is not only time-consuming and costly but also unavoidable dilution of the sample leads to a drastic rise in detection limits (Sun and Ko, 2004). In addition, it provides a comparatively uniform detection limit across a large portion of the Periodic Table and is applicable to a wide range of concentrations, from a 100% to parts per million (ppm) (Jenkins, 1999). The purpose of this study covers the performance of the XRF for determination of essential and trace elements in lemon grass samples. The values found in this study will be keeping informed of the Pakistani food composition tables.

## Materials and Methods

Three samples of lemon grass were collected from the local fields of Karachi, Pakistan. Each sample was

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analyzed three times (triplicate) and samples were coded as LG-01, LG-02 and LG-03. The preference of the samples was based on the increasing consumption of lemon grass in tea preparation.

*Sample preparation*

In the solid sample preparation, grinding is major step up to obtain fine powder by magnetic grinder and polished to assure surface homogeneity. Weighed out about 7 grams sample by adding three pallets of binder material then pressed under the 20 tons pressure for 5 to 10 minutes for making pallets form. The samples were further analyzed by XRF spectrometer.

*Instrumentation*

Performed measurements done by using S4 PIONEER spectrometer. A closely coupled optical path helps provide high intensities and low detection limits for all elements. Automatic computer control of the generator allows the kV and mA settings to be adjusted automatically for each element. The optimization settings of voltage and milliampere provide furthermost sensitivity for all elements. The lower atomic number elements are typically analysed using low kV and high mA settings, while the higher atomic number elements are analyzed with high kV and lower mA settings. Operation and data reduction for the S4 PIONEER were easily handled with the Bruker AXS SPECTRA$^{plus}$ software package.

*Moisture content determination*

Initially weigh out fresh green leaves of lemon grass sample, kept in oven at 80°C for 72 hour, after drying of samples the moisture minus from the initial weight, which data shows as a mean percentage of triplicates.

*Ash percentage*

Oven dried leaves was taken in crucibles and kept in furnace Gold Star- 7122, furnace temperature gradually increase up to 450°C and sample kept for 2 hours, the obtained weight of samples were calculated from the initial weight for the ash % age.

## Results and Discussions

The trace elements includes manganese (Mn), iron (Fe), cobalt (Co), copper (Cu) and zinc (Zn), all of these belong to the category of micronutrients, and need of human body in very small quantity (generally less than 100 mg/day), the elements considered as macronutrients, such as sodium (Na), calcium (Ca), magnesium (Mg), potassium (K) and chlorine (Cl), which are required in larger quantities. Trace elements are important components of biological structures; they can be toxic for human body or biological function. Metal ions has deep effect on abundance of each other, it was reported that the deficiency or excess level or improper diet may disturb the metabolism of essential trace (iodine, Fe, Zn, Ca, Mg, Mn) and toxic (Cu and Cd) elements, resulted in malfunctioning of the human body metabolism (Kandhro et al., 2008, 2009a, 2009b and 2010).

The results in Table 1 showed that the high mean levels of K, Ca and Silicon (Si) were found 54.02%, 25.87% and 9.02% respectively, while the second high amount of Na and Mg were observed 2.91% and 2.08% in analyzed lemon grass samples respectively. The levels of Fe, Phosphorous (P), sulfur (S) and aluminum (Al) were obtained > 1%, while others trace elements were show < 1% as given in Table 1.

The excess level of Cu may cause a Fe or Zn deficiency and Cu absorption may be decreased by excess dietary Fe or Zn (Kandhro et al., 2008; Kandhro et al., 2010). The deficiency of iodine might occur with high consumption of soybeans (or other beans) which is known to be high in Cu and other toxic elements (Kandhro et al., 2009a). Brzoska and Moniuszko-Jakoniuk (2000) have been studied that enhanced consumption of Zn and Fe may decrease Cd absorption from the digestive tract and its accumulation in the organism, and as a result, it may ameliorate the toxic effects of Cd. The Cu and Zn are known to be essential micronutrients, but both can be toxic depending upon the concentration (Onianwa et al., 1999). The low or high amount of dietary intake of Cu and Zn were based to create the various physiological and pathological diseases because of these trace element deficiency or toxicity (De Romana et al., 2005; Hurrell et al., 2004). The mean values of both elements in analyzed lemon grass sample were 0.15 % or 0.0015g $g^{-1}$ (Table 1). The Zn constitutes about 33µg $g^{-1}$ of an adult body mass and it is essential component of many enzymes implicated in several physiological functions, such as protein synthesis and energy metabolism (Onianwa et al., 2001). A normal adult human body contains about 1.5–2.0 µg $g^{-1}$ of Cu (Kies, 1989), required in hemoglobin synthesis and in the catalysis of metabolic oxidation (Underwood, 1977).

The Fe is most important essential trace element and its deficiency causes anemia in human and animals. The mean concentration of Fe was determined 1.49% in samples as shown in Table 1. The typical sources of heme Fe are hemoglobin and myoglobin which obtained from animals; however cereals, seeds of leguminous plants, fruits, vegetables and dairy products are of non-heme Fe sources (Fraga and Oteiza, 2002).

**Table 1.** X-ray fluoresces Spectroscopic (XRF) Elemental determination of different lemon grass tea samples (n =3×3=6)

| Elements (% age) | Sample 01 (n=3) | Sample 02 (n=3) | Sample 03 (n=3) | Mean ± STD |
|---|---|---|---|---|
| K | 53.40 | 54.65 | 54.00 | 54.02 ± 0.62 |
| Ca | 26.19 | 24.77 | 26.64 | 25.87 ± 0.98 |
| Si | 10.01 | 9.45 | 7.61 | 9.02 ± 1.26 |
| Na | 2.57 | 3.32 | 2.85 | 2.91 ± 0.38 |
| Mg | 2.05 | 2.04 | 2.14 | 2.08 ± 0.05 |
| Fe | 1.54 | 1.28 | 1.67 | 1.49 ± 0.20 |
| P | 1.28 | 1.53 | 1.90 | 1.57 ± 0.31 |
| S | 1.03 | 0.77 | 0.71 | 0.84 ± 0.17 |
| Al | 0.77 | 1.28 | 1.43 | 1.16 ± 0.34 |
| Sr | 0.51 | 0.26 | 0.48 | 0.41 ± 0.14 |
| Br | 0.21 | 0.15 | 0.21 | 0.19 ± 0.03 |
| Mn | 0.15 | 0.13 | 0.12 | 0.13 ± 0.02 |
| Cu | 0.15 | 0.18 | 0.12 | 0.15 ± 0.03 |
| Zn | 0.13 | 0.20 | 0.12 | 0.15 ± 0.05 |

Key = [a]Average value ± confidence interval (*P* = 0.05)

**Table 2.** Moisture content of Lemon grass samples

| Samples | Mean fresh weight (MFW) g | Mean dry weight (MDW) g | (MFW) – (MDW) | Moisture (%age) ± STD |
|---|---|---|---|---|
| 01 | 50.12 | 36.52 | 13.6 | 27.13±0.65 |
| 02 | 50.12 | 35.81 | 14.31 | 28.55±0.84 |
| 03 | 50.11 | 37.14 | 12.97 | 25.88±0.58 |

**Table 3.** Ash %age of Lemon grass samples

| Sample | Mean Dry Weight (g) | Mean Ash (g) | Ash (%age) ± STD |
|---|---|---|---|
| 01 | 3.12 | 0.73 | 23.40±0.45 |
| 02 | 3.12 | 0.75 | 24.04±0.48 |
| 03 | 3.12 | 0.78 | 25.00±0.62 |

**Table 4.** Validation of the proposed method for determination of lemon grass against literature value (mg)

| Sample Lemon grass | Certified values/literature values (mg) | Observed values[a] | RSD[b] | % recovery[c] |
|---|---|---|---|---|
| K | 723.0 | 727.0 | 1.15 | 99.4 |
| Ca | 65.00 | 62.0 | 3.79 | 104.8 |
| Na | 6.00 | 5.7 | 13.06 | 105.3 |
| Mg | 60.0 | 61.0 | 2.4 | 98.4 |
| Fe | 8.17 | 8.12 | 13.42 | 100.6 |
| P | 101.0 | 99.0 | 19.75 | 102.0 |
| Mn | 5.224 | 5.45 | 15.38 | 95.9 |
| Cu | 0.266 | 0.315 | 20 | 84.4 |
| Zn | 2.23 | 2.43 | 33.33 | 91.8 |

*Literature values (http://food.vegtalk.org/products/lemon_grass_(citronella)_raw.html).
[a]Average value±confidence interval.
[b]RSD =Std Dev*100/mean value
[c]%Recovery =literature value*100/Found values.

Manganese was associated with amino acid, lipid and carbohydrate metabolism and development of bone in human body (Davis and Greger, 1992). The typical sources of dietary Mn include grain, rice, tea and nuts. The toxicity of Mn occurs when it is in excess; it can be cause of Parkinson-type syndrome (Aschner, 2000), the mean value of Mn was observed at 0.13% in lemon grass samples (Table 1).

Table 2 shows the mean percentage of moisture content of lemon grass samples. In 1998, the Brazilian government approved a legislation (RDC 519 of 06/26/1998) establishing the identity and quality of products made from plants to be used for infusions or decoctions. According to the regulation, dried leaves to be used for the preparation of tea should have at least of 12% (w/w) moisture (Barbosa et al., 2008). The moisture regulation is important in packaged foods, moisture levels within the food package environment limits moisture-mediated deprivation such as microbial spoilage, and preserves the appearance and flavor characteristics of food products to extend shelf life and protect brand integrity, moisture content ranges from 25.88 to 28.55%. High levels of humidity can make easy enzymatic processes that can humiliate the active principles, however the development of microorganisms under high-level moisture, which decreases the product therapeutic quality (Farias, 2003; Silva et al., 2005). The subject parameter of lemon grass sample is an important quality parameter, so the matter should be taken into consideration.

The ash contents of the samples in Table 3 shows, plants grown in humus rich soils or sand rich soil also significantly lowers the ash content of the grass, while Plants grown in heavy clay soils produce the highest ash content. The soil type of the growing location influences the ash content of red canary-grass (Burvall, 1997). The ash content is lowest in the stem and highest in the leaf sheaths and leaf blades (Pahkala and Pihala, 2000). The content in lemon grass samples was determined at the range of 23.40% to 25.00%.

The method was assured by the analysis of triplicate samples, certified reference material (literature values) in Table 4. While recoveries elements of were compared with literature reported data. The percentage recoveries of some different elements were calculated by the equation: %Recovery =literature value*100/Found values. The measured values are therefore not significantly different from the certified values (literature values). The validation data confirm that applied method could be used as a routine procedure for the determination of total different elements in lemon grass samples.

## Conclusion

Commonly consumed Pakistani lemon grass for tea making were analysed for the contents of K, Ca, Si, Na, Mg, Fe, P, S, Al, Sr, Br, Mn, Cu, Zn. The Data clearly shows the suitability of the Bruker AXS S4 PIONEER (XRF) spectrometer analysis of different essential and trace elements. Accurate, reliable and repeatable results are obtained in a measurement time of just 20 minutes per sample. The data shown here verified that XRF was able to provide the necessary levels of analytical accuracy for an effective, rapid and simple screen for elements in lemon grass samples. The use of this data to produce efficient food composition labels is a step towards making estimates of nutrient intakes and adequacies from food using up surveys more reliable.

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