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Determination of contamination of Nickel in Vanaspati (vegetable fats) by EDXRF

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Abstract- A simple method for the quantitative determination of Nickel in Vanaspati by EDXRF is described. The nickel spiked fat was taken in platinum crucible and heated at 800^0C in muffle furnace with sodium carbonate thereafter the ash was dissolved in HCl:HNO_3 (1:3). The quantitative determination of Nickel was carried out by Energy Dispersive X ray Fluorescence Spectrophotometer after optimization of the instrumental parameters. Results from spike and recovery experiments at levels of 20 and 50 ug g–1 are 95–110%.

Introduction

Vanaspati (vegetable fats) are generally made up of different oils like palm oil, sesame oil. The presence of metals in edible oils may be due to different factors: natural contamination, introduction of the metals during the refining process and contact with the storage material. Metals like Cu, Fe are essential for human body where as metal like Nickel is toxic even at concentration levels forward and rapid pretreatment systems for oils is dilution of μg g–1,[1,2] these oxidation processes may generate peroxides, aldehydes, ketones, acids, epoxides and other compounds which may produce pathological effects in the digestive system and also react with food components (proteins and pigments), thus sensitizing the action of some carcinogenics.

There are methods available in different literatures dealing with the determination of these metals in vegetable fats at ppm levels using UV Visible spectrophotometer and graphite furnace Atomic Absorption spectrophotometer[3and4]. Also methods like voltammetry[5] atomic absorption [6-12] and atomic emission[13] are available to determine metals in edible oils. Vegetable oil pretreatment procedures which destroy the organic material are calcination, extraction, solubilization and dilution. The dissolution procedure by microwave digestion at atmospheric pressure has been applied with ICP-AES and GFAAS[13]. A method was developed for the semi quantitative simultaneous analysis of Ba, Cd, Co, Cr, Cu, Mn, Ni, Tl, Pb, U, V and Zn in olive oil by ICP-MS by the formation of aqueous emulsions[14]. There are some problems with plasma stability and there may be carbon deposition on the metallic cones of the interface as vegetable oil contains organic matter. Ion chromatography is the method which is developed for determination of metals in vegetable oils after treatment with peroxide and pyrolysed by UV photosensitisation[15].

The simple method has been developed for determination of nickel in vanaspati (vegetable fat) by EDXRF after heating

it, at 800^{0}C in muffle furnace with sodium carbonate thereafter the ash was dissolved in HCl:HNO_3 (1:3).

Experimental

Instrumentation

All experiments were performed Energy Dispersive X-ray Fluorescence Spectrophotometer (ED2000 of Oxford instruments UK). Muffle furnace (Hecatech India) of capacity 1000^{0}C was used. Water bath was also used to melt fat.

Reagents

Analytical grade HCl (Merck UK) and HNO_3 (Merck UK) was used. Aqueous stock solutions of metals (100 μg mL–1) (Merck, UK) were used. With these solution multi elemental solutions of 2, 4, 6, 8, 10 μg mL–1 were prepared and used as a calibration solution for quantitation mode. Sodium carbonate of A.R. grade (Merck UK) was used as fluxing agent. Deionized water (Milli-Q grade) was used through out. The vanaspati fat in this case was used as Dalda. Nickel sulphate of A.R grade was used to spike 20 and 50 $\mu g\ g^{-1}$ of Nickel in vanaspati fat.

Sample preparations

All glassware was thoroughly cleaned and stored in 20% nitric acid prior to use. It was homogenized with distilled water immediately before use. Sample solutions were prepared gravimetrically; portions of around 1 g of spiked fat were weighed in a platinum crucible and was kept on water bath and mixed with sodium carbonate. The crucible was kept in muffle furnace at 800^{o}C for 2 hrs. The ash was dissolved in 5 ml HNO3: HCl (3:1) solution and followed by diluted with 5 ml deionised water. For the recovery study the spiked samples were prepared with Nickel so that the final concentrations were 20 and 50 $\mu g\ g^{-1}$ of Nickel in vanaspati fat.

Procedure

Standards of Nickel of different concentration 2, 4, 6, 8, 10 μg mL–1 were taken in the sample holder and analysed by EDXRF, from the counts per second at Kα line and concentration of Nickel calibration curve was plotted. Similarly from counts per second of spiked fat and linear equation the experimental value of spiked fat was calculated.

Results and discussions

XRF (X-ray fluorescence spectrometry) is a non-destructive analytical technique used to identify and determine the concentrations of elements present in solid, powdered and liquid samples. XRF is capable of measuring elements from beryllium (Be) to uranium (U) and beyond at trace levels often below one part per million and up to 100%.

Energy dispersive x-ray fluorescence (EDXRF) relies on the detector and detector electronics to resolve spectral peaks due to different energy X-rays with high-resolution detectors, like lithium drifted silicon, Si (Li). The XRF spectrometer measures the individual component wavelengths of the fluorescent emission produced by a sample when irradiated with X-rays.

The standard Nickel of different concentrations were prepared and analysed by EDXRF at Kα line, from the count per seconds (table 1) of different concentrations the calibration curve was plotted as shown in figure 1. The equation of linear regression curve was obtained y = 31.9* x + 7.4. From the equation of linear regression the concentration of spiked vanspati sample was calculated. The detection limit was calculated by 3 times standard deviation of the blanks. Ten measurements of the blank were made in order to calculate the

deviation. The detection limit is in the range of 2- 8 $\frac{\mu g}{ml}$.

The concentration nickel in vanaspati fat was 0.01 μ g g–1 produced by Dalda. Results from the spike and recovery experiments are as follow the average recovery of triplicate samples for 20 $\mu g\ g^{-1}$ was 20.9 $\mu g\ g^{-1}$, 50 $\mu g\ g^{-1}$ was 50.97 $\mu g\ g^{-1}$ with relative standard deviation +/- 2 $\mu g\ g^{-1}$ after considering dilution factor 10.

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Table 1-

Sr No	Concentration of Nickel	Counts per seconds
1	2 µg ml-1	71
2	4 µg ml-1	128
3	6 µg ml-1	212
5	8 µg ml-1	256
6	10 µg ml-1	327
7	Nickel Spiked fat (1g fat in 10 ml)	74.3
8	Nickel Spiked fat (1 g Fat in 10 ml)	170

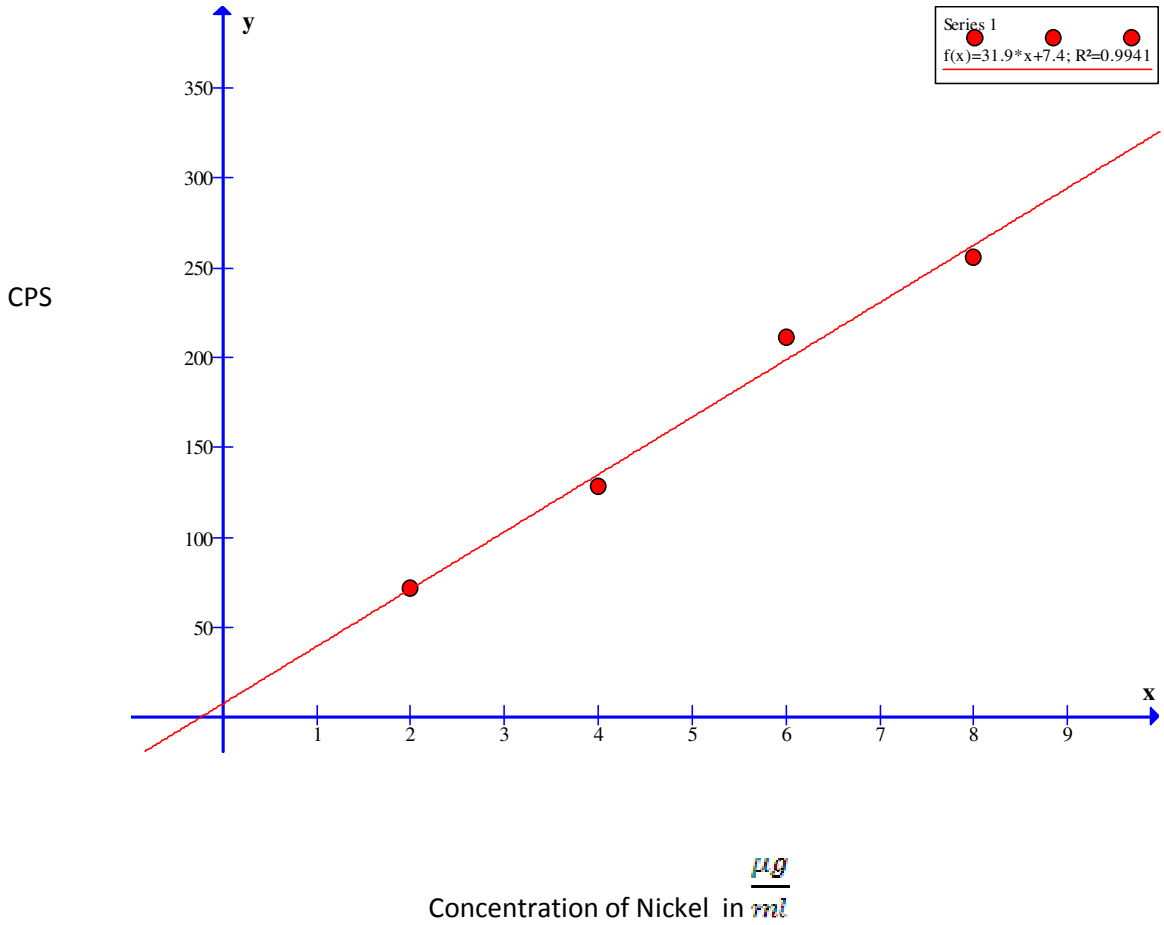

Fig. 1- Calibration curve of concentration of Nickel VsCounts per seconds