DETERMINATION OF NICKLE CONTENTS IN SELECTED VANASPATI GHEE THROUGH ATOMIC ABSORPTION SPECTROPHOTOMETER

Waqas Ahmad[1], Hameed Ur Rehman[1], Muhammad Aamir[1], Maria Urooj[1], Nayyar Hafiz[1], Zubia Masood[2], Mohib Ullah[1], Ijaz Ahmad[1]

[1]Department of Chemistry, Kohat University of Science & Technology (KUST), Kohat-26000 Khyber Pakhtunkhwa, Pakistan

[2]Department of Zoology, Sardar Bahadur Khan Women University Quetta

[*]E-mail: drijaz-chem@yahoo.com

Abstract

To convert vegetable edible oils into vanaspati ghee, nickel is used as a catalyst in the hydrogenation process. A simple and fast method for the trace level determination of nickel in ghee is reported. In this different methods were applied for the extraction of residual nickel from ghee samples. Using toluene, as organic solvents, an acid mixture was used for the extraction of nickel. Extracted nickel was quantified with atomic absorption. Among the organic solvents, toluene proved to be the best solvent mediating a 95% extraction of nickel from ghee samples. Nickel was extracted and determined in ten different brands of ghee and in all samples its amount was well above the permissible limit of WHO (0.2 µg/g). Other metals like lead, zinc, copper, and cadmium were also determined and their concentrations were found to be much below the WHO permissible limits.

Keywords: Hydrogenated edible oil; Nickel formats catalyst; Solvent extraction; Spectrophotometric quantification

Introduction:

The human body uses oils and fats in the diet for three purposes, as an energy source, as a structural component and to make powerful biological regulators. Oils and fats also play an important role in metabolic reactions in the human body. Oils and fats contain fatty acids, which are susceptible to attack by a number of agents e.g. light, oxygen, metals, etc. A heavy metal is a member of a loosely defined subset of elements that exhibit metallic properties. It mainly includes the transition metals, some metalloids, lanthanides, and actinides. Many different definitions have been proposed—some based on density, some on atomic number or atomic weight, and some on chemical properties or toxicity. The term *heavy metal* has been called a "misinterpretation" in an IUPAC technical report due to the contradictory definitions and its lack of a "coherent scientific basis".{1}

There is an alternative term *toxic metal*, for which no consensus of exact definition exists either. As discussed below, depending on context, heavy metal can include elements lighter than carbon and can exclude some of the heaviest metals. Heavy metals occur naturally in the ecosystem with large variations in concentration. In modern times, anthropogenic sources of heavy metals, i.e. pollution, have been introduced to the ecosystem. Waste-derived fuels are especially prone to contain heavy metals, so heavy metals are a concern in consideration of waste as fuel. {2}

Heavy metals include lead (Pb), cadmium (Cd), zinc (Zn), mercury (Hg), arsenic (As), silver (Ag) chromium (Cr), copper (Cu) iron (Fe), and the platinum group elements. Environment is defined as the totality of circumstances surrounding an organism or group of organisms especially, the combination of external physical conditions that affect and influence the growth, development and survival of organisms reducing the quality of life and may eventually cause death. Such a substance has to be present in the environment beyond a set or tolerance limit, which could be either a desirable or acceptable Limit. Ghee is composed almost entirely of fat; the nutrition facts label found on bottled cow's ghee produced in the USA indicates 8 mg of cholesterol per teaspoon. Studies on rats have shown that ghee helps to reduce serum cholesterol slightly but not significantly.{3} It is difficult to remove some metallic compounds completely during processing without incorporating post treatments, particularly, if the diameters of the particles are less than 2 micrometer. As the occurrence of trace metals is at the cutting edge both for oil quality and human health, so their analysis is of significance. Several atomic absorption spectrophotometric methods have been developed over the years for the determination of trace metal content in edible oil and fats. Most of the procedures involved time consuming and

tedious digestion and ashing of the oil and fat samples before their analysis. There is no universal ashing procedure for all metals because of their reactivity and volatility. Currently acid-extraction of various terrace metals from oil and ghee and then analysis for all trace metals in oil and ghee by atomic absorption methods, without their isolation has become an important tool for quality control of acid-extraction.

In the present study an attempt has been made to establish a precise and improved sample preparation method that could be used for the estimation of Nickel in ghee.

Material and Method

Sampling

Different brands of ghee samples were collected from the local market at different time intervals with a gap of at least two months.

Extraction Procedure

A modified version of the reported method{4} was used for the extraction of nickel from ghee samples, where as 20 g of ghee was heated to melt and then dissolved in 10 ml of toluene followed by the addition of 20 ml of acid mixture (20% HNO3 + 20% H2SO4). The mixture was shaken vigorously for ten minutes in a separating funnel. After the equilibration of the mixture, the acid layer was separated and collected in a china dish. To remove any un-extracted residual nickel, an acid mixture (10 ml) was added to the organic layer in the separating funnel, was shaken for 5 minutes and the acid layer was transferred and added to the previously collected extract. The acid extract was directly analyzed with AAS.

RESULTS AND DISCUSSIONS

Solvent Extraction of Nickel

After the conversion of vegetable edible oil into ghee, the spongy catalyst is separated from the product. However, due to overuse of the catalyst and poor filtration equipment, fine particles of the catalyst are retained in the product as nickel formats. In the solid state, nickel formats typically exist in the form of a dehydrate, Ni (HCOO) 2, 2H2O. On dissolution of melted ghee in organic solvent and then shaking it with aqueous acidic mixture, nickel formats are transferred from an organic into an aqueous medium and are analyzed.

Extraction Procedure

The extraction of nickel was carried out with several methods for comparison. The method developed by Price was used as a standard method in this regard.5 molten ghee of 20 g was dissolved in 10 ml of Toluene followed by addition of 15 ml HNO_3/H_2SO_4. The mixture was then vigorously shaken in a separating funnel for 30 minutes. Then 50 ml of distilled water was added to it and it was again shaken for 30 min and was allowed to stand. After equilibration of the two layers the inorganic layer was separated and analyzed for nickel (Table 1).

Choice of Organic Solvent

The original method of Price used carbon tetrachloride as organic solvent, {5} while *Souliotis* substituted benzene.{6} In this regard, using different acidic mixtures, and the extraction of nickel was checked using organic solvents such as toluene, carbon tetrachloride and benzene.{7} keeping in view our past experience, and toluene was used as organic solvent in the present work.

The extraction of nickel by different methods in this work indicates that toluene is an efficient solvent that provides better recovery than do benzene and carbon tetrachloride. The better efficiency of toluene can also be correlated with the polar nature of toluene as compared to benzene and carbon tetrachloride and also its complete immiscibility with water. Benzene and carbon tetrachloride are lightly miscible, i.e. 0.008 and 0.17 parts/100, respectively. 9-11 It is also clear from the results of different extraction methods that a 20% acid mixture of both H_2SO_4 and HNO_3 is more effective than concentrated acids for the same purpose. Residual nickel in hydrogenated vegetable oils has also been investigated by other workers but all these methods are tedious, time consuming and are not efficient in extracting the maximum amount of residual nickel, e.g. method of Price et al.3 requires treatment of sample with HNO_3 and Toluene in a separating funnel for ninety minutes. Similarly the method of the Association of Official Analytical Chemists10 produces extensive fumes and is difficult to be used for hydrogenated edible oils. Thus the method used for the extraction of nickel in this work represents the best compromise currently available for reliable, economical and efficient extraction of residual nickel in ghee samples. Table.1 presents the data of ten different brands of ghee. For each brand three samples were collected at an interval of two months. The data presented in this table is an

Average of three parallel determinations for the same sample. It is clear from the table in the analysis that the residual nickel content is well above the WHO permissible limit for nickel, i.e. 0.2 ppb for ingestible foods.6 After completing results for lot No. 1, the respective industries were informed about the observed level of nickel in their products and remedies like replacement of catalyst material and filtration cloth after a proper time, etc. were suggested. The response of the industries was cooperative in this regard. They took action to rectify the problem and afterwards there was an appreciable decrease in the residual contents of nickel in their samples. In

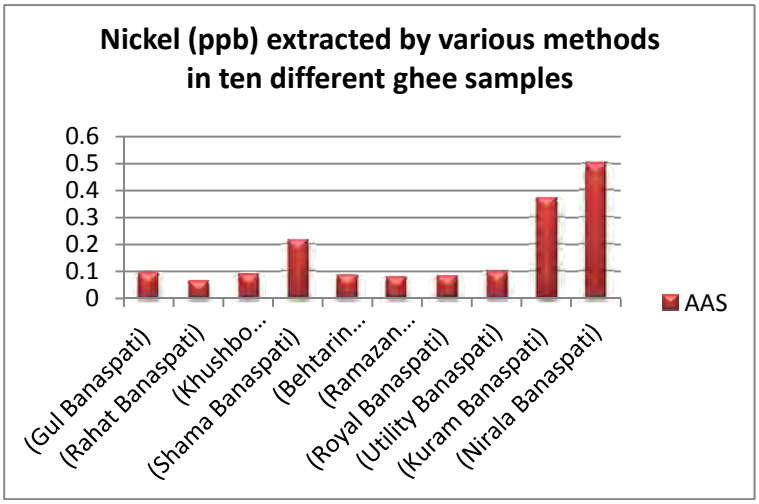
each lot the variations in the concentration of residual nickel is random; however, the decrease in the level of residual nickel in some samples is encouraging.

Comparison of Sample Preparation Methods for the Analysis of Trace Metals in Typical Oil Fats Products as Determined by AAS

[Ni] Metal	improve acid extraction	wet digestion
Tul. Ba	0.390 ˛ 0.006	0.389 ˛ 0.05	0.410 ˛ 0.04
Dal. Ba	0.289 ˛ 0.05	0.300 ˛ 0.03	0.310 ˛ 0.06
Sos.Co	0.038 ˛ 0.005	0.041 ˛ 0.004	0.041 ˛ 0.004
Hab. Co	0.059 ˛ 0.005 0	0.065± 0.003	. 0.064±0.006
Pkb.Ma	1.090 ˛ 0.04	1.096 ˛ 0.03	1.150 ˛ 0.05

Table 1. Nickel (ppb) extracted by various methods in ten different ghee samples

Extraction Method Technique	Method-I	AAS
Sample 1 (Gul Banaspati)	Toluene	0.0933 ± 0.005
Sample 2 (Rahat Banaspati)	Toluene	0.0648 ± 0.005
Sample 3 (Khushbo Banaspati)	Toluene	0.0897 ± 0.01
Sample 4 (Shama Banaspati)	Toluene	0.216 ± 0.03
Sample 5 (Behtarin Banaspati)	Toluene	0.0836 ± 0.006
Sample 6 (Ramazan Banaspati)	Toluene	0.080 ± 0.02
Sample 7 (Royal Banaspati)	Toluene	0.081 ± 0.04
Sample 8 (Utility Banaspati)	Toluene	0.096 ± 0.007
Sample 9 (Kuram Banaspati)	Toluene	0.373 ± 0.017
Sample 10 (Nirala Banaspati)	Toluene	0.499 ± 0.016



Graph.1 Show the nickel content in different type of ghee.

CONCLUSION

The extraction of nickel by different methods in this work indicates that toluene is an efficient solvent that provides better recovery than do benzene and carbon tetra chloride. Atomic absorption spectrophotometric and colorimetric methods proved to be quite sensitive for nickel determination and both have yielded almost the same results. This method is recommended for the oil and fat industries for quality control and better estimation of residual nickel in their products. The analysis proved to be a better way for correcting residual nickel content in the products as is clear from the cumulative results. However, a high level of nickel present in consumer products is undoubtedly a health hazard and strong efforts are required for the strict implementation of quality control regulations.

ACKNOWLEDGEMENTS:

Authors are grateful to the faculty members for their appreciativeness and making the facilities available for the research in , department of chemistry, Kohat University of science and technology, Kohat, Pakistan.

REFERENCES:

[1] John H. Duff us "Heavy metals" a meaningless term? (IUPAC Technical Report)" Pure and Applied Chemistry, (2002), Vol. 74, pp. 793–807
[2] Smouse, T. H. Factors affecting oil quality and stability in methods to assess oil quality and stability of oils and Fat containing food; (1994) pp 17-36.
[3] Matam Vijaya Kumara, MV; Kari Sambaiaha; Belur R. Lokesh "Hypocholesterolemic effect of anhydrous milk fat ghee is mediated by increasing the secretion of biliary lipids". The Journal of Nutritional Biochemistry. (2000) 11 (2): pp 69–75.
[4] Ilyas, M.; Hussain, M.; Khan, H.; Zaheer, I, Physical Chemistry pp (1994), 13, 123
[5] Price, W. J.; Roos, J. T. H.; Clay, A. F. Analyst, (1970), 95, 760.
[6] Souliotis, A. G. J., Anal. Chem. (1964), 36, 1385.
[7] Ilyas, M.; Hussain, M.; Khan, H.; Zaheer, I. Physical Chemistry. (1994), 13,123