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Study of The Degree of Contamination of Sunflower Oil Composition with Heavy Metal Ions

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Abstract: This article provides a scientific substantiation of modern laboratory approaches used to determine the degree of contamination of sunflower oil with heavy metal ions. The main focus is placed on the sample preparation stage, microwave digestion, the advantages of ICP-MS and ICP-OES methods, the alternative capabilities of graphite furnace atomic absorption spectrometry, quality control, method validation, and the procedure for recalculating results into mg/kg units. Taking into account the specific characteristics of the oily matrix, the article summarizes the principles of minimizing external contamination, using blank samples, evaluating recovery rates, and comparing results with current regulatory documents. In conclusion, closed-vessel microwave digestion followed by ICP-MS or ICP-OES analysis is recommended as the most reliable scientific approach for determining Pb, Cd, As, Hg, Cu, Fe, Mn, Ni, Cr, and Zn in sunflower oil.

Keywords: Sunflower oil, heavy metal ions, microwave digestion, ICP-MS, ICP-OES, GF-AAS, quality control, method validation.

INTRODUCTION:

Industrial Vegetable oils are an important source of energy and biologically active components in human nutrition, and their safety is one of the urgent directions of food chemistry and sanitary control. Since sunflower oil is a widely consumed product, the presence of heavy metal elements in its composition raises safety concerns related to technological processes, raw material quality, storage, and packaging conditions.

Toxic elements such as Pb, Cd, As, and Hg can negatively affect the human body even in trace amounts, while elements such as Cu, Fe, Mn, Ni, Cr, and Zn provide important information for assessing product oxidative stability, quality indicators, and sources of technological contamination at certain concentrations. Therefore, it is necessary to apply sensitive, selective, and reproducible analytical approaches for the determination of trace metals in oily matrices.

The purpose of this article is to systematize the methods used for determining heavy metal ions in sunflower oil, present their laboratory methodology in article format, and provide scientifically grounded recommendations for practical research.

RESEARCH OBJECT, MATERIALS, AND METHODS

As the object of study, sunflower oil samples from different manufacturers purchased from local retail outlets are considered. After sampling, they are stored in tightly sealed, light-protected, metal-free containers. Before analysis, the sample is thoroughly shaken to achieve homogeneity; if necessary, slight

heating, filtration, or separation of sediment is performed.

Analytical-grade concentrated nitric acid (HNO3), 30% hydrogen peroxide (H2O2), deionized water, multi-element standard solutions, and internal standard solutions are used in the analysis. The main equipment includes an analytical balance, a microwave digestion system, PTFE digestion vessels, volumetric flasks, automatic pipettes, and ICP-MS or ICP-OES spectrometers.

Selection of Methods and Their Scientific Basis

The most difficult stage in determining heavy metal ions in sunflower oil, which is an oily matrix, is sample preparation. Since the organic substances in the matrix may interfere with instrumental analysis and the metal content is usually very low, sensitivity and contamination control are priority criteria when selecting an analytical method.

Microwave digestion, being carried out in a closed vessel, reduces the risk of external contamination, more completely decomposes the organic matrix, and makes it possible to obtain a clear solution for subsequent ICP-MS or ICP-OES analysis. Due to multi-element analysis capability, low detection limits, and high reproducibility, this combination is the most appropriate approach for scientific research.

Table 1. Recommended Analytical Approaches

Method	Detectable Elements	Advantages	Application
Microwave digestion + ICP-MS	Pb, Cd, As, Hg, Cu, Fe, Mn, Ni, Cr, Zn, and others	High sensitivity, multi-element analysis, low detection limit	Main recommended scientific method
Microwave digestion + ICP-OES	Macro- and microelements, including Pb, Cd, Cu, Fe, Zn	Multi-element capability, relatively lower cost, convenient for routine use	For laboratories without ICP-MS
Direct GF-AAS	Mainly Pb and Cd	Sensitive, convenient for individual elements	Alternative method in laboratories without ICP systems
Direct dilution + HR ICP-OES	As, Cd, Hg, Pb, and some macroelements	Rapid preparation, low reagent consumption	For screening and technological control

Main Experimental Protocol

For analysis, 0.20–0.50 g of each sample is accurately weighed into a PTFE microwave digestion vessel. Then 2 mL of concentrated HNO3 and 1 mL of 30% H2O2 are added. To control the strong exothermic reaction, the vessel is left for pre-reaction for 10–15 minutes, after which it is tightly sealed and subjected to digestion in a microwave system under a stepwise heating program.

After digestion is completed, the vessels are cooled to room temperature, opened, and the resulting solution is quantitatively transferred to a 25.0 mL volumetric flask or another required volume and diluted to the mark with deionized water. The obtained clear solution is then used for instrumental analysis.

In instrumental analysis, external calibration is performed using a blank solution and at least a 3–5 point standard series. Where necessary, an internal standard is used. The ICP-MS method serves for determining very low concentrations, while the ICP-OES method is convenient for routine multi-element monitoring.

Alternative Methods

If an ICP system is not available in the laboratory, Pb and Cd can be determined by graphite furnace atomic absorption spectrometry. Although this method has high sensitivity for individual elements, its capacity for multi-element analysis is limited.

When rapid screening is required, the sample may be diluted with an appropriate organic solvent and measured by HR ICP-OES. However, in this approach the sample must be in a completely dissolved state; if solid particles, rust, or mineral residues are present, recovery values may decrease. Therefore, for final conclusions, a method based on microwave digestion remains superior.

Quality Control and Method Validation

In each analytical series, it is recommended to use a reagent blank, method blank, parallel sample, fortified sample, and, whenever possible, a certified reference material. The use of metal-free containers, a dust-free working environment, and clean pipettes plays an important role in minimizing external contamination.

The suitability of the method is assessed through the linearity of the calibration curve, stability of the blank background, recovery values, and the differences between repeated measurements. In practice, recovery is usually expected to be within the range of 80–120%, while repeatability must comply with the internal validation criteria of the laboratory.

Data Processing and Assessment of Contamination Level

If the instrument reports the concentration of an element in the solution in mg/L, the content in sunflower oil is recalculated into mg/kg using the following expression:

$$X\ (mg/kg) = (C \times V \times D) / m$$

where C is the concentration indicated by the instrument (mg/L), V is the final volume (L), D is the additional dilution coefficient, and m is the sample mass (kg). It is appropriate to express the results as the mean value of three parallel determinations ± standard deviation.

To assess the contamination level, the determined concentrations are compared with the relevant regulatory values, and the contamination coefficient can be expressed as K = Csample / Cstandard. If K < 1, the sample is within the permissible limit; if K = 1, it is in a borderline condition; and if K > 1, it is considered above the standard limit.

RESULTS AND DISCUSSION

The methodological analysis presented in the article shows that in determining heavy metal elements in oily matrices such as sunflower oil, the main source of uncertainty is more strongly related to the sample preparation stage than to instrumental measurement. Closed-vessel microwave digestion is advantageous because it reduces external contamination, almost completely decomposes the organic matrix, and provides a suitable solution for multi-element instrumental analysis.

The choice between ICP-MS and ICP-OES depends on laboratory capabilities and the expected concentration levels. ICP-MS is preferred for the determination of toxic elements at very low levels, whereas ICP-OES serves as a more economically convenient option for technological monitoring and broader practical application. GF-AAS, although limited in multi-element analysis capability, remains an effective alternative method for Pb and Cd.

At the same time, the high sensitivity of the instrumental method alone is not sufficient; results obtained without the use of blanks, recovery tests, and reference materials cannot be considered scientifically reliable enough. Therefore, methodological discipline and quality control requirements occupy a central place in assessing the degree of contamination of sunflower oil with heavy metal ions.

CONCLUSION

The most reliable approach for determining heavy metal ions in sunflower oil is closed-vessel microwave

digestion of the sample followed by ICP-MS or ICP-OES analysis. This approach is distinguished by its capability for multi-element analysis, low detection limits, high reproducibility, and effective control of external contamination.

In laboratories where ICP systems are not available, GF-AAS can be used for Pb and Cd, while HR ICP-OES based on direct dilution may be applied for rapid screening. However, for drawing final scientific conclusions, it is necessary to ensure complete quality control based on blank samples, parallel analyses, recovery tests, and reference materials.

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