Determination of Lead in Alcoholic Beverages by FAAS: A Sustainable Approach with Hydrophobic Deep Eutectic Solvents

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Highlights

- Improved sensitivity for Pb determination by FAAS.
- Matrix effects were suppressed through the sample preparation step.
- A 20-fold preconcentration factor was achieved.

Abstract

In 2023, 17.3 billion liters of beer, 400 million liters of wine, and 1.4 billion liters of spirits were produced. The Brazilian Health Regulatory Agency (ANVISA), through Resolution RDC No. 42 (Aug 29, 2013), regulates the maximum limits for inorganic contaminants in food and beverages, establishing a tolerance limit for lead (Pb) in alcoholic beverages at 200 μg L^{-1}.(1) In this context, the present work aims to develop an analytical method using flame atomic absorption spectrometry (FAAS) combined with sample preparation that employs hydrophobic deep eutectic solvents (HDES) for the determination of lead in alcoholic beverages. Pb is a toxic metal widely used in the steel industry, particularly in low-melting-point welds, which can lead to its presence in alcoholic beverages due to contact with pipes and storage materials. When present in concentrations above the established limit, lead can bioaccumulate in the body, causing a range of health complications.(2) HDES are a class of solvents investigated for their innovative properties and studied as alternatives to traditional techniques that use environmentally harmful substances such as concentrated acids and toxic organic solvents. Their use is particularly advantageous in liquid-liquid extractions due to their hydrophobicity, which facilitates phase separation, and their high affinity for analytes, resulting in efficient extractions. These properties enable the preconcentration of analytes using HDES, improving the method's sensitivity and allowing the determination of lead by FAAS, considering the limit of quantification (LOQ) of 1012 μg L^{-1}, which is higher than the tolerable limit. For the analysis of lead in hydroethanolic samples using FAAS, a method was developed in which the sample is diluted fourfold to mitigate matrix effects. Then, 0.5 to 1.5 mL of HDES is added, and the mixture is stirred with a vortex for 15 to 300 seconds. The mixture is then centrifuged, and the extracting phase is separated. An aliquot of 0.5 mL of 5% v v^{-1} HNO_3 is added to the extraction phase, vortexed again, and separated by centrifugation. Finally, the aliquot of HNO_3 is recovered, and this eluent, with a 20-fold preconcentration factor, is introduced into the FAAS for analysis. Throughout the study, the extraction parameters were optimized, achieving an average recovery of 85% with a standard deviation of 8.5%. Additionally, the potential for solvent recycling and the application of the method to real samples are being investigated to ensure the reliability of the developed method.

[1] RDC Resolution No 42, august 29, 2013, Brazilian Health Regulatory Agency, Brazil, 2013.
[2] R. B. Martin, *Encyclopedia of Inorganic Chemistry*, John Wiley & Sons, Ltd., Chichester, UK, 2006.

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