Balsamic Vinegar Reproducibility Study of Heavy Metal Quantification Method Using Spectral Analysis

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Abstract

This project seeks to examine the reproducibility of a previously established method used to quantify heavy metals in organic and inorganic samples. The established method was primarily used to test heavy metals in eye cataracts, but its applicability to other types of samples requires further testing. Balsamic vinegar has the potential to contain traces of heavy metals due to its long aging process involving wooden barrels. This preparation technique expands the possibility of contamination by heavy metals. Therefore, this study will examine heavy metal concentrations in balsamic vinegar samples using the spectroscopic analyte quantification method. Heavy metal analysis of samples is performed using Graphite Furnace Atomic Absorption Spectroscopy (GFAAS) and Total Reflection X-Ray Fluorescence (TXRF) Spectrometry. The heavy metals copper and magnesium were spiked in balsamic vinegar samples and underwent microwave-assisted acid digestion prior to spectrometric testing. Flame atomization was applied to magnesium, while copper underwent graphite furnace atomization. Testing resulted in high average percent recoveries for both metals. The next metal to be tested in this reproducibility study using balsamic vinegar is lead.

Introduction

- Atomization of an element at a high temperature determines the unknown elemental concentration in the sample by measuring the specific wavelength of light transmitted [1].
- Heavy metals can be potentially hazardous by acting as environmental pollutants and accumulating to toxic levels in living organisms [2].
- The previously established heavy metal quantification method has the potential to analyze metals in a diverse array of organic and nonorganic samples. This research will delve further into the reproducibility of said method and move towards the prospect of quickly and effectively quantifying heavy metal concentrations.

GFAA

TXRF

Methodology

- The method began with preparation of the balsamic vinegar samples for heavy metal analysis. 1 mL of balsamic vinegar samples were spiked with either 1 ppm Mg or 50 ppb Cu and diluted to 10 mL with 5 M HNO_3. The samples remained refrigerated until testing occurred.
- To perform GFAAS analysis, the balsamic vinegar samples underwent microwave assisted acid digestion. This technique destroys the sample matrix to allow for the concentration of species to be analyzed [3].

GFAAS Optimization Parameters for Cu Analysis

Condition being Optimized	Optimal Conditions
Step 1	60 C
Step 2	120 C
Step 3	250 C
Step 4,5,6	800 C
Step 7	2400 C
Step 8	2500 C

Method Parameters for Microwave-Assisted Acid Digestion (MAAD)

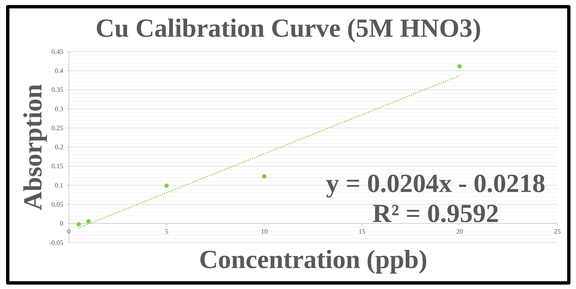
Stage	Power (W)	Ramp Time (min)	Pressure (bar)	Temperature (C)	Hold Time (min)
1	1200	15:00	20.68	100	5:00
2	1200	15:00	20.68	110	10:00

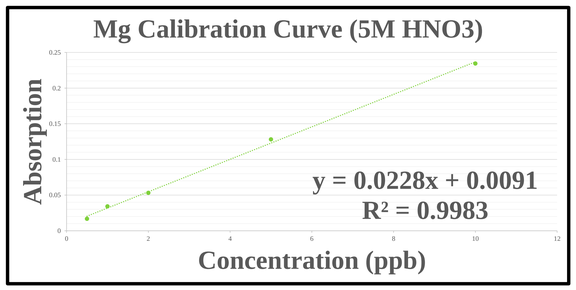
- The balsamic vinegar samples were transferred into digestion vessels and placed in their canisters. They were digested in a Microwave Assisted Acid Digestion (MAAD) instrument.
- Flame atomization analysis was performed on samples spiked with magnesium.
- Graphite furnace atomization analysis was performed on samples spiked with copper. The previously determined optimization parameters for copper GF testing were implemented.

Data Results

Metal Spiked	Amount Spiked	Average	SD	% RSD
Cu	50 ppb	52.60	7.098	13.49
Mg	1 ppm	1.202	0.1964	16.35

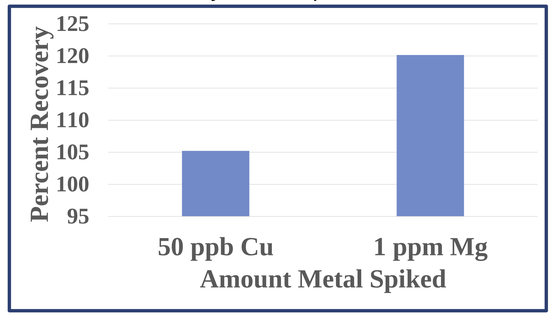
- For the heavy metals being tested, standards containing known concentrations of the metal are prepared and analyzed with atomic absorption spectroscopy (AAS) to compare against the unknowns with spiked concentrations of the metal. Comparing the spiked unknowns to the calibration curve is key in determining the concentrations of heavy metals present in the balsamic vinegar [4].
- The average recovered metal concentration for the samples tested is shown in the table above, along with SD and % RSD values within acceptable range to support reproducibility.
- Average recovered concentrations were averaged among the 15 balsamic vinegar samples analyzed for each metal.





Discussion

- The high average percent recoveries for copper and magnesium in balsamic vinegar using the established method demonstrate the reproducibility of this method to quantify heavy metals in organic and inorganic samples. The average percent recoveries are shown below for the two heavy metals used to spike balsamic vinegar samples.
- The next heavy metal to undergo reproducibility testing using balsamic vinegar is lead. The established method was previously confirmed for lead in *E. coli* and yeast samples.



Acknowledgements

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