Atomic Absorption Analysis of Lead Leached from Ceramic Dinnerware

Introduction / Justification

During the United States Depression Era, there was a large production of inexpensive ceramic dinnerware. The dinnerware was produced by many companies including California Pottery and H.A. Bauer Pottery Company. The most famous was Fiesta Ware by Homer Laughlin Company of West Virginia which comes in red, blue, green, yellow, and ivory. [1] The glazes used on the pottery were originally prepared from uranium ore, causing the dinnerware to be radioactive. The uranium ore (U-238) decomposes through alpha and beta emissions until it reaches the stable isotope of Pb-206. [1] Today, in rural Mexican and Latin American communities Lead oxide (PbO) is added to the glaze as a fluxing agent. This decreases the starting temperature of the fusion process during firing from about 1670°C to 500°C. The Lead oxide also helps create a desirable shiny, smooth surface.[2]

The use of uranium and lead oxide poses a serious health risk because lead can leach from the glaze through an acidic medium, such as vinegar (acetic acid) or salsa (pH = 4.8). [1, 2, 3] Lead is extremely toxic at low levels, especially to children, and can cause many health problems including: a decrease in heme synthesis, increase in blood pressure, anemia, seizures, renal dysfunction or failure, and encephalopathy.[2] Lead poses even more of a danger in children, causing impaired growth and cognitive decline at low level exposure. [2]

Although, the pottery doesn't pose as large of a threat today, especially in the U.S., as it used to, it still is a hazard especially if imported from Mexico or Latin America. To help with safety and import, the U.S. Food and Drug Administration (FDA) has set regulatory action levels and analytical tests for ceramic ware. The action levels are: 3.0μg/ml (ppm) of leaching solution for flatware (plates), 2.0ppm for small hollowware (other than mugs/cups), 1.0ppm for large hollowware (other than pitchers), and 0.5ppm for mugs, cups, and pitchers.[4]

The purpose of this experiment is to determine if Western Oregon University's collection of plates and bowls from California Pottery and H.A. Bauer Pottery Company tests positive for lead using a quick color test. If the ceramic ware tests positive for lead, then the objective is to determine the concentration lead leached into solution, using standard FDA analytical atomic absorption spectrometric (AAS) methods and determine if it meets or exceeds FDA regulations.

Literature Review

Since lead is both an environmental and health concern, lead analyses are fairly common. Many different mediums

and methods are used based on what is being tested for lead – soil, water, paint, ceramic ware, etc. A few of the methods found for ceramic ware include inductively coupled plasma – mass spectrometry (ICP-MS), which can be highly informative and sensitive [2], and diffuse reflectance infrared Fourier transform spectroscopy (DRIFT), which can give characterization of the lead compounds present.[2] Two common tests found are the quick color test and AAS. The quick color test is a screening test to see if lead is present.[2, 5] The second, which is standardized by the FDA, uses atomic absorption spectrometry (AAS) (flame and graphite furnace) which can detect quantities as low as 1ppm for flame AAS and lower using a graphite furnace.[3, 6]

Method

Quick Color Test

A test to begin with is called the quick color or spot test. This test can help quickly identify glazed ceramics which release excessive lead. This method is chosen to start with because it is a screening test and can therefore save time and materials by excluding pieces that do not need to be tested. Gould found that, with extensive testing, the spot test "correctly categorized 100% of the sets of undecorated ware" [5] that were tested. Since all of WOU's ceramic ware is undecorated this test will work well. Gould also noted that the spot test is not sensitive to cadmium which can also be present.[5]

Materials[5]: Citric acid solution (1.3%) 1.33g citric acid in 100mL water, chromogen solution (0.2%) 0.005g rhodizonic acid dipotassium salt in 25ml water, filter paper (2 x 2 cm) Whatman chromatographic paper.

Sampling [5]: Slip of paper is wetted with approximately 75µL of citric acid solution, the paper must be soaked with no excess solution. The paper is placed into total contact with the surface of the ceramic ware for 30min or until dry. The slip is then removed and spotted with 5µL of chromogen solution. Lead is present if a rose/red color immediately appears indicating further analysis should be performed.

Comments: 4% acetic acid was tried and worked; however, there was a noted time delay of 0.5 to 3min for color to appear.[5] The minimum amount of lead tested that produced a visible color was 0.25µg/cm^2. [5] Another method of performing the color test is to treat the filter paper with 2%(w/v) rhodizonic acid solution, allow it to dry and dip it into a solution of 2% acetic acid that has been allowed to leach lead for 30s, a rose/red color will appear if lead is present.[2]

Atomic Absorption Spectrometry

The method chosen for this experiment to determine the amount of lead in each item is the standard FDA flame

AAS method. Since this method is designed and developed for determining lead levels from ceramic ware it seemed most appropriate.

Materials: 4% acetic acid (vinegar), distilled water, 0.02% detergent solution (1mL dish soap mixed with 5.0L water), atomic absorption grade acetylene for the AAS.

Apparatus: Ceramic bowls and plates to be tested, contamination-free laboratory beaker or dish (method blank), Atomic Absorption Spectrometer set to a wavelength of 217.0nm for solutions containing either high or low concentrations of lead or to a wavelength of 283.3nm for solutions containing high concentrations of lead.[6,7]

Sampling [6,7]: Wash selected ceramic ware and contamination-free laboratory beaker or dish, to be used as a method blank, for 30 seconds in a 0.02% detergent solution and gently dry with a soft cloth. Rinse with copious amounts of distilled water. Air-dry in a dust-free environment.

Fill dishes with 4% acetic acid to within 6-7 mm of the edge of the vessel. Record amount used for each dish. Cover each item to minimize evaporation and outside contamination. Leach dishes for 24 hours at room temperature. If evaporation occurs, add more vinegar until it is 6-7 mm from the edge of the vessel. Gently stir and transfer solution to plastic container using a pipette, do not pour. Store each solution in a sealed container in total darkness and analyze within one day for best results.

Analysis [6,7]: For a calibration curve prepare calibration solutions of lead in 4% acetic acid that produce responses of approximately 0.000, 0.050, 0.100, 0.200, and 0.350-0.400 Abs, each one is run three times. Between each aspiration or run of each calibration solution and the unknown (leached) solutions the AA needs to be re-zeroed so the solvent (4% acetic acid) is aspirated again and the AA is zeroed.

The leached solutions are then analyzed; if the absorbance is above the calibration range of 0.350-0400 then the solution needs to be diluted with solvent until a test solution produces an instrument response in the working range, 0.050 to 0.350-0.400 Abs. Use this response and dilution factor to calculate approximate concentration in each sub-sample leach solution. First, verify the calibration and absence of carry-over by running the method blank and the solvent (0ppm). If the concentrations are within ±5%, continue with analysis. Run each sample three times making sure that it is within the working response range and zeroing the AA in between each run.

Potential Problems [6]: Outside contamination from dust, supplies, and laboratory glassware can cause high results. To prevent or minimize contamination; work areas and labware must be kept clean using acid-cleaning techniques for glassware and if possible use plastic labware. Also, "nonspecific absorption and scattering of light due to concomitant species in leach solutions" [6] will give high results. To correct for this, instrument background correction needs to be used.

Budget / Timeline

Reagents for this lab should be readily available or easy to obtain and WOU already owns an atomic absorption spectrophotometer. Depending on whether or not the quick spot test is used the AA method will take 24hours prior to scheduled lab time to conduct and the solutions can be stored until lab. If all students are kept in one group the entire analysis should be able to be completed in one 4 hour lab period. For the quick spot test up to 30min is needed to screen the ceramic ware before leaching begins.

References

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