CENE 486 Capstone Project—Adsorption of Copper and Lead from Mine Wastewater Using Mushrooms as a Bio sorbent

Environmental W.W. Engineering

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Procedure: XRF Detection Limit

Application: Determine Copper and Lead Detection Limit for Wastewater Sample Solution

Summary: The purpose of this procedure is to identify the XRF detection limits for lead and copper in liquid solutions. The copper chloride is a powder, while the lead acetate is a stock solution. Dilute solutions will be created for the copper chloride and lead acetate with a different series of steps.

1.0 Equipment

(a) 200 mL Beaker
(b) 1000 mL Beaker
(c) Analytical Scale
(d) Fume Hood
(e) Stir Rod
(f) XRF

2.0 Reagents and Materials

(a) Copper Chloride

(c) Lead Acetate Solution at 386 mg/mL

3.0 XRF Detection Limit for Copper Procedure

1. Add 100 mL of distilled water to a 200 mL beaker.

2. Measure out 0.21 g of copper chloride on a scale.

3. Add the copper chloride to the beaker under the fume hood and stir with a stir rod until the powder is fully mixed into the water.

4. Test the copper chloride solution with the XRF device.
    a. Add a small amount of the solution to the sample container
    b. Cover the sample with a specialized film and screw on the lid
    c. Place filled and sealed sample container in lead plated box to be measured by the XRF device
5. Test the copper chloride solution with the XRF device.
    a. Add a small amount of the solution to the sample container
    b. Cover the sample with a specialized film and screw on the lid
    c. Place filled and sealed sample container in lead plated box to be measured by the XRF device

6. If the concentration of the solution is within range for the XRF device
    a. If the solution is not within range because the concentration is too low, repeat Steps 2 through 5, adding 0.21 g of copper chloride to the solution repeatedly until the XRF device produces a reading.
    b. If the solution is within range, dilute the solution in half and retest until a non-detect is generated

7. Once a reading has been generated by the device, retest the same solution multiple times to check for consistency in the XRF device's ability to read the copper chloride in à liquid sample.

8. The lowest concentration of copper chloride that the XRF is capable of reading is the XRF detection limit for copper chloride.

4.0 XRF Detection Limit for Lead Procedure

1. 4.36 mL of the 360 g/L lead acetate solution will be added to a 1000 mL beaker where the rest of the container should be filled with DI water.
    a. This is stock solution is highly concentrated, therefore small amounts are needed to reach desired concentrations
    b. This amount will be used for dilutions if necessary

2. Test the lead acetate solution with the XRF
    a. Add a small amount of the solution to the sample container
    b. Cover the sample with a specialized film and screw on the lid

c. Place filled and sealed sample container in lead plated box to be measured by the XRF device

3. If the concentration of the solution is within range for the XRF device and produces a reading move on to Step 4 to dilute the solution. The solution should be diluted until the XRF device can no longer produce a reading, at this point the detection limit could be approximately the last viable reading.

4. Add 50 mL of distilled water to the beaker, stir the solution to mix, and repeat Step 3.