Teacher Guide

Salts & Ceramics Activity

Introduction:
Salt deposits can crystallize within porous materials such as ceramics. This process causes damage known as flaking or spalling, which can destroy object surfaces. Identifying the salts present in an artifact is vital for determining the course of treatment. If the salts are identified and characterized as soluble, one of the most common treatment procedures is to soak the artifact in distilled water. Conservators monitor the treatment progress by measuring the conductivity of the successive water baths. This experiment uses chemical spot tests to discover the chemical formula of a salty build-up on ceramic material. The second half of the experiment investigates conductivity and how the practice of bathing ceramics works.

Objectives:

- Identify the unknown ions present
- Predict ionic compound formulas
- Observe changes in conductivity levels
- Graph conductivity data

<u>9-12th grade Science Georgia Performance Standards</u>

SCSh3 Students will identify and investigate problems scientifically.

d. Graphically compare and analyze data points and/or summary statistics.

e. Develop reasonable conclusions based on data collection.

SB1. Students will analyze the nature of the relationships between structures and functions in living cells.

d. Explain the impact of water on life processes (i.e., osmosis, diffusion).

SC1. Students will analyze the nature of matter and its classifications.

b. Identify substances based on chemical and physical properties.

c. Predict formulas for stable ionic compounds (binary and tertiary) based on balance of charges.

Supplies:

Ceramic or Clay pot pieces
Scalpel
Micro well plate (or test tubes)
Black paper
White paper
Droppers or pipets
Distilled water
Acetic acid, dilute

Silver nitrate ($AgNO_3$), 0.2 M
Hydrochloric acid (HCl), 3 M
Barium chloride ($BaCl_2$), 2 M
Diphenylamine, 1% in sulfuric acid
Sodium hydroxide (NaOH), 5%
Nitrophenylazo solution
Large beaker (500 mL)
Conductivity meter *or* LabQuest interface with conductivity probe

Safety: $AgNO_3$, HCl, $BaCl_2$, diphenylamine in sulfuric acid, and nitrophenylazo solution are corrosive. Minimize contact with skin. Wear personal safety items, including gloves and goggles, at all times. Do not pour any of these substances down the drain.

Teacher Pre-Lab:

Part I. Preparing ceramic pot pieces

- A terracotta flower pot or tiles may be used as the ceramic. Pot tiles can be broken into several smaller pieces for individual or small group testing. Place pot or tile into a paper or plastic bag and hit with hammer on a hard table top or ground to create fragments.
- Partially submerge the fragments in a solution of 200 grams sodium chloride and/or magnesium sulfate in 1000 mL of distilled water for 3-5 days or until some crystallization is observed. Salt crystals will appear as a white powder on the water line of the pot.

  *** The amount of salt and water simply need to form a concentrated solution. Amounts used may vary based on the size pot or number of fragments used.**
- Remove the salty fragments and allow to dry for several days.
- Drying fragments in an oven can help crystals form more quickly.

  * **Students could complete this portion of the prelab and have a discussion on osmosis and diffusion.**

Part II. Prepared or Purchased Solutions

- 0.2 M silver nitrate ($AgNO_3$) aqueous solution:

  Dissolve 1.5g of solid $AgNO_3$ in 50mL of distilled water. Keep in a dark bottle or wrap with aluminum foil. Silver nitrate used in the experiment should be relatively fresh.
- 2M barium chloride ($BaCl_2$) aqueous solution 5% (w/v):

  Dissolve 2.5g of $BaCl_2$ in 50mL of distilled water.
- 3M hydrochloric acid (HCl) solution (1:3):

  Add 10mL concentrated HCL to 30mL of distilled water.
- Prepared diphenylamine, 1% in sulfuric acid can be purchased.
- Prepared nitrophenylazo solution can be purchased.

*** Micro well plates, small test tubes, or small watch glasses may be used for testing. Transparent containers are preferable. Containers should be clean and dry before use.**

Conductivity

- The LabQuest interface used with a compatible conductivity probe will collect and graph data. Alternatively, you can use a handheld conductivity meter and graph data manually. ***In both cases, the device needs to be sensitive up to 2,000 μS/cm.**

Procedures:

Part I

Tests:

1. Obtain a ceramic fragment.
2. To remove salt crystals from your ceramic, scrape the surface of the fragment gently with a scalpel and collect on filter paper or in a small dish.
3. To test for chloride anions (Cl^-):
    a. Place a few salt crystals into well #1 (or test tube #1).
    b. Add a few drops of distilled water in wells #1 and #2.
    c. Add a few drops of acetic acid in well #1 and #2.
    d. Add 1-2 drops of silver nitrate ($AgNO_3$) solution to well #1 and #2.
    e. Observe and record. Use well #2 for comparison to determine the results of the test. It may help to place black paper under the micro well plate.
    Positive: white precipitate or cloudy solution
4. To test for sulfate anions (SO_4^{2-}):
    a. Place a few salt crystals into well #3.
    b. Add a few drops of distilled water in wells #3 and #4.
    c. Add 2-3 drops of 3M HCL to well #3 and #4.
    d. Add 1 drop of 2M $BaCl_2$ to well #3 and #4.
    e. Observe and record. Use well #4 for comparison and determine the results of the test. Again, a black background may help for observations.
    Positive: white precipitate
5. To test for magnesium cations (Mg^{2+})
    a. Place a few salt crystals into well #5.
    b. Add a few drops of distilled water in wells #5 and #6.
    c. Add 5 drops of 5% NaOH to well #5 and #6.
    d. Add 1-2 drops of nitrophenylazo solution to well #5 and #6.
    e. Observe and record. Use well #6 for comparison and determine the results of the test. Placing a white background under the well may help for observations.
    Positive: blue crystalline precipitate in purple solution (precipitate crystals are small but still visible)

6. To test for nitrate anions (NO_3^-):
    a. Place a few salt crystals into well #7.
    b. Add 1 drop of solution of diphenylamine in sulfuric acid to well #7.
    c. Observe and record.
    Positive: dark blue color after several seconds
7. Using the results of your tests, determine the ions present and possible formulas of the salts present in the ceramic.

Part II

Treatment:

1. Obtain a beaker large enough to hold your ceramic fragment. Add a 100mL of distilled water to the empty beaker. To make sure your probe is accurately calibrated, measure the conductivity of the water. It should be 0 µS/cm.
2. Place your ceramic fragment in the beaker. Add more distilled water until the ceramic is completely submerged. The water level should be slightly higher than the top of the ceramic, to ensure that it will not be exposed after evaporation.
3. Allow the beaker to sit for 2 days. Measure and record the conductivity as Bath I.
4. Carefully remove your ceramic fragment and place it on a paper towel. Pour the water down the drain.
5. Carefully put the fragment back into the beaker and refill with fresh distilled water. Remember that the level of water should be high enough to cover the entire fragment.
6. Allow the beaker to sit for 2 more days. Measure and record the conductivity of the water as Bath II.
7. Repeat Steps 4 – 6 so that you have a total of 6 readings (two weeks).
8. Graph your data from the six readings.

Clean up:

- Part I clean up: Use distilled water to remove contents of the micro well plate and collect for proper disposal.
- Part II clean up: All liquids can be poured down the drain. Be sure to run hot water behind the salt solutions.

Selected Resources:

- *All the King's Horses: the Treatment of a Geometric Horse Pyxis* – A podcast which explains desalination through diffusion and the relationship between salinity and conductivity using an object from the Carlos Museum's Collection https://youtu.be/Mjje4UD1-io

- Johnson, J. (1998). Soluble Salts and Deterioration of Archaeological Materials. *onserveOGram*, (6/5). Retrieved July 28, 2021 from: http://www.nps.gov/museum/publications/conserveogram/06-05.pdf

- Odegaard, N., Carroll, S., & Zimmt, W. S. (2007). *Material Characterization Tests for Objects of Art* (2nd ed.). Archetype Books.

- Wheeler, G. S., & Wypyski, M. T. (1993). An Unusual Efflorescence on Greek Ceramics. *Studies in Conservation*, *38*(1), 55–62.