

Student 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

Supplies:

Ceramic pot pieces
Scalpel
Micro well plate (or test tubes) – transparent containers are best.
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 goggles and gloves at all times. Do not pour any of these substances down the drain.

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.
 - i. 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.
 - i. 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.
 - i. 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.
 - i. 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 $\mu\text{S}/\text{cm}$.
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:

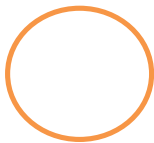
- Part I clean up: Use distilled water to remove contents of the micro well plate and collect for proper disposal.
- Part II clean up: All liquids can be poured down the drain. Be sure to run hot water behind the salt solutions.

Student Name _____ Date _____ Period _____

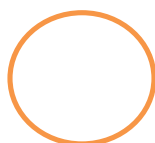
**Salts & Ceramics
Answer Sheet**

Part I Tests: (Write the word positive or negative in the circle)

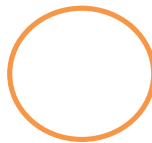
Well #1(Chloride)



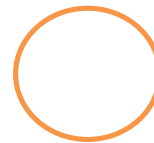
Well #3(sulfate)



Well #5(magnesium)



Well #7(nitrate)



Part II Treatment:

Bath I	Bath II	Bath III	Bath IV	Bath V	Bath VI

Graph the above data. Use the graph to interpret what happens as the ceramic piece is placed in successive water baths.

Analysis Questions:

1. Why must you use distilled water instead of tap water in the water baths?

2. Predict the formula(s) and name the salt(s) that may be present in the water baths.

3. Where might there be examples of spalling due to salt damage around your home or school?
