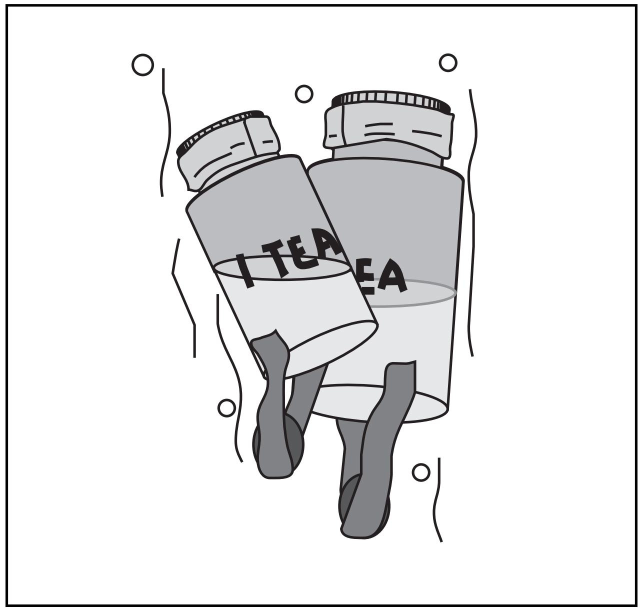
Zeolite Crystal Growth



Zeolite crystals are being grown in a hot water bath.

Objective:

- To grow zeolite crystals and investigate how gravity affects their growth.

Science Standards:

Science as Inquiry
Physical Science
Unifying Concepts and Processes
Change, Constancy, & Measurement
Science in Personal and Social Perspectives

Science Process Skills:

Observing
Communicating
Measuring
Collecting Data
Controlling Variables
Investigating

Mathematics Standards:

Measurement

Activity Management:

The preparation of zeolite crystals, although not difficult, is an involved process. A number of different chemicals must be carefully weighed and mixed. You may wish to prepare the chemicals yourself or assign some of your more advanced students to the task. Refer to the materials and tools list on the next page for a detailed list of what is required.

This activity involves maintaining a hot water bath continuously for up to 8 days. If you do not have the facilities to do this, you can conduct the experiment for just the 0 and 1 TEA (triethanolamine) samples described below. Crystals may also be formed if the hot water bath is turned off at the end of the school day and turned on the succeeding day. Crystallization times will vary under this circumstance, and close monitoring of the formation of the crystalline precipitate will be necessary.

Following the growth of zeolite crystals, small samples can be distributed to student groups for microscopic study.

Procedure:

1. While wearing hand and eye protection, weigh 0.15 grams of sodium hydroxide and place it in a 60 ml, high-density polyethylene bottle. Add 60 ml of distilled water to the bottle and cap it. Shake the bottle vigorously until the solids are completely dissolved. Prepare a second bottle identical to the first.
2. Add 3.50 grams of sodium

Materials and Tools

Sodium aluminate $NaAlO_2$ FW=81.97
Sodium metasilicate anhydrous, purum, Na_2O_3Si, FW=122.06
Sodium hydroxide pellets, 97+%, average composition NaOH, FW=40
Triethanolamine (TEA), 98% $(HOHCH_2)_3N$, FW=149.19
Distilled water
1000 ml Pyrex® glass beaker
Aluminum foil
Metric thermometer with range up to 100°C
Laboratory hot plate
2-60 ml high-density polyethylene bottles with caps
4-30 ml high-density polyethyene bottles with caps
Plastic gloves
Goggles
Glass microscope slides
Permanent marker pen for marking on bottles
Waterproof tape
Lead fishing sinkers
Tongs
Eyedropper
Optical microscope, 400X

metasilicate to one of the bottles and again cap it and shake it until all the solids are dissolved. Mark this bottle "silica solution." To the second bottle, add 5.6 grams of sodium aluminate and cap it and shake it until all the solids are dissolved. Mark this bottle "alumina solution."

3. Using a permanent marker pen, mark the four, 30 ml high-density polyethylene bottles with the following identifications: 0 TEA, 1 TEA, 5 TEA, and 10 TEA.
4. Place 0.85 grams of TEA into the bottle marked "1 TEA." Place 4.27 grams of TEA into the bottle marked "5 TEA." Place 8.55 grams of TEA into the bottle marked "10 TEA." Do not place any TEA into the bottle marked "0 TEA."
5. Add 10 ml of the alumina solution to each of the bottles. Also add 10 ml of the silica solution to each bottle.
6. Cap each bottle tightly and shake vigorously. Secure each cap with waterproof tape and tape a lead sinker to the bottom of each bottle. The sinker should weigh down the bottle so it will be fully immersed in the hot water.
7. Prepare a hot water bath by placing approximately 800 ml of water in a 1000 ml Pyrexs beaker. Place the four weighted bottles into the beaker. The bottles should be covered by the water. Cover the beaker with aluminum foil and punch a small hole in the foil to permit a metric thermometer to be inserted. Fix the thermometer in such a way as to prevent it from touching the bottom of the beaker. Place the beaker on a hot plate and heat it to between 8$ and 95° C. It will be necessary to maintain this temperature throughout the experiment. Although the aluminum foil will reduce evaporation, it will be necessary to periodically add hot (85 to 90° C) water to the beaker to keep the bottles covered.
8. After 1 day of heating, remove the bottle marked 0 TEA from the bath with a pair of tongs. Using an eyedropper, take a small sample of the white precipitate found on the bottom of the bottle. Place the sample on a glass microscope slide and examine for the presence of crystals under various magnifications. Make sketches or photograph any crystals found. Be sure to identify magnification of the sketches or photographs and estimate the actual sizes of the crystals. Determine the geometric form of the crystals. Look for crystals that have grown together.

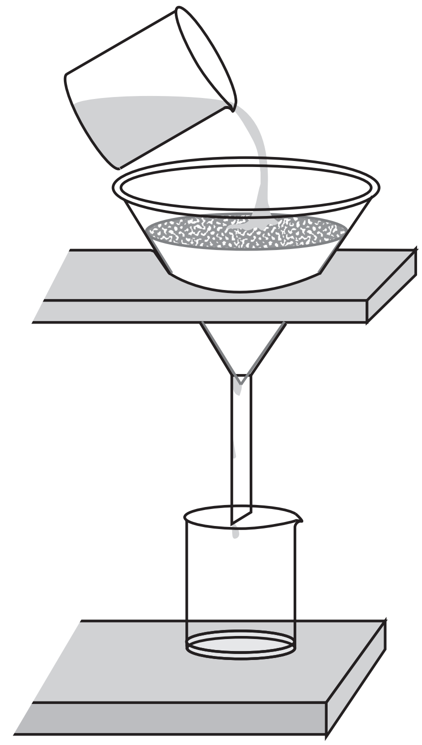
9. Repeat procedure 8 for the 1 TEA bottle after 2 days of heating. Repeat the procedure again for the 5 TEA bottle after 5 days and for the 10 TEA bottles after 8 days. Compare the size, shape, and intergrowth of the crystals formed in each of the bottles.

Assessment

Collect student sketches and written descriptions of the zeolite crystals.

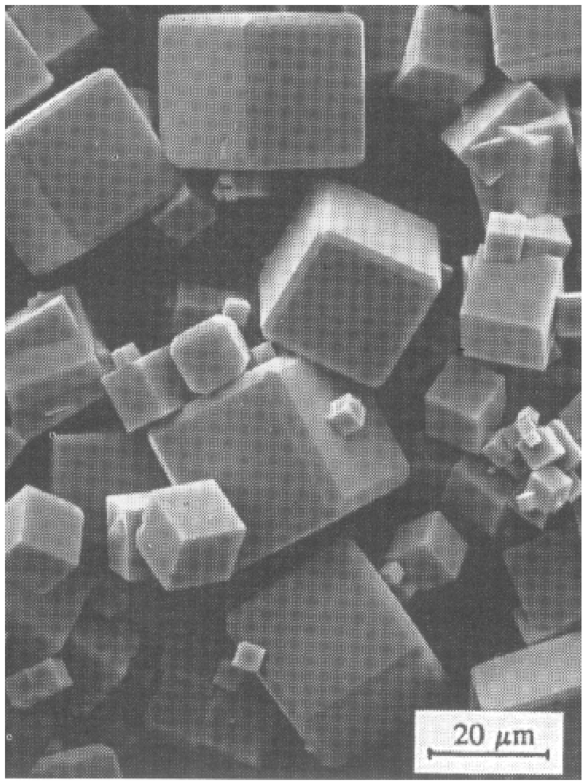
Extension:

1. Obtain zeolite filter granules from a pet shop. The granules are used for filtering ammonia from aquarium water. Set up a funnel with filter paper and fill it with the granules. Slowly pour a solution of water and household ammonia (ammonia without lemon or other masking scents) into the granules. Collect the liquid below and compare the odor of the filtered solution and the unfiltered solution. Try running the filtered solution through a second time and again compare the odors. Be sure to wear eye protection.



Zeolites

Zeolites are crystals made up of the elements silicon, aluminum, and oxygen. The crystals consist of alternating arrays of silica (beach sand, SiO_2) and alumina (aluminum oxide, Al_20_3) and can take on many geometric forms such as cubes and tetrahedra. Internally, zeolites are rigid sponge-like structures with uniform but very small openings (e.g., 0.1 to 1.2 nanometers or 0.1 to 1.2 X 10^{-9} meters). Because of this property, these inorganic crystals are sometimes called "molecular sieves." For this reason, zeolites are employed in a variety of chemical processes. They allow only molecules of certain sizes to enter their pores while keeping molecules of larger sizes out. In a sense, zeolite crystals act like a spaghetti strainer that permits hot water to pass through while holding back the spaghetti. As a result ot tnis filtering action, zeolites enable chemists to manipulate molecules and process them individually.



The many chemical applications for zeolite crystals make them some of the most useful inorganic materials in the world. They are used as catalysts in a large number of chemical reactions. (A catalyst is a material that has a pronounced effect on the speed of a chemical reaction without being affected or consumed by the reaction.) Scientists use zeolite crystals to produce all the world's gasoline though a chemical process called catalytic cracking. Zeolite crystals are often used in filtration systems for large municipal aquariums to remove ammonia from the water. Because they are environmentally safe, zeolites have been used in laundry detergents to remove magnesium and calcium ions. This greatly improves detergent sudsing in mineral-rich "hard" water. Zeolites can also function as filters for removing low concentrations of heavy metal ions, such as Hg, Cd, and Pb, or radioactive materials from waste waters.

Although scientists have found many beneficial uses for zeolites, they have only an incomplete understanding of how these crystals nucleate (first form from solution) and grow (become larger). When zeolites nucleate from a water solution, their density (twice that of water) causes them to sink to the bottom of the special container (called an autoclave) they are growing in. This is a process called sedimentation, and it causes the crystals to fall on top of each other. As these crystals continue to grow after they have settled, some merge to produce a large number of small, intergrown zeolite crystals instead of larger, separate crystals.

Zeolite crystal growth research in the microgravity environment of Earth orbit is expected to yield important information for scientists that may enable them to produce better zeolite crystals on Earth. In microgravity, sedimentation is significantly reduced and so is gravity-driven convection.

Zeolite crystals grown in microgravity are often of better quality and larger in size than similar crystals grown in control experiments on Earth. Exactly how and why this happens is not fully understood by scientists. Zeolite crystal growth experiments on the Space Shuttle and on the future International Space Station should provide invaluable data on the nucleation and growth process of zeolites. Such an understanding may lead to new and more efficient uses of zeolite crystals.

Microscopic Observation of Zeolite Crystals

Name: ________________________

Instructions:

Observe through a microscope each zeolite crystal sample provided to you by your teacher. Sketch the samples in the circles provided and write a brief description of what you see.

Sample 1

_______________ TEA

Sample age _____________ day(s)

Description:

Magnification: ___________ X

Sample 2

_______________ TEA

Sample age _____________ day(s)

Description:

Magnification: ___________ X

Sample 3

__________________ TEA

Sample age ________________ day(s)

Description

Magnification: _____________ X

Sample 4

__________________ TEA

Sample age ________________ day(s)

Description

Magnification: _____________ X

QUESTIONS:

1. What geometric form (crystal habit) did the zeolite crystals assume as they grew? Was there more than one form present? How did the zeolite crystals appear when they grew into each other?

2. Can you detect any relationship between the length of time crystals were permitted to form, their size and their geometric perfection?

3. Would additional growing time yield larger crystals? Why or why not?