Making Ink—An Ancient Process

Introduction

All ink has two basic ingredients—a liquid and a source of pigment for color. The ancient Egyptians and Chinese used a very simple ink composed of soot mixed in water or vegetable oils. By the 9th century A.D., medieval Europeans were making ink using a slightly more complex process of mixing tannic acid (extracted from oak galls) with an iron salt. This was called iron-gall ink. In this activity, a simpler version of iron-gall ink is made using ordinary tea bags.

Science Concepts

- Solutions
- Suspensions
- Physical and chemical changes

Materials

Acacia (gum arabic), 1–2 g
Hydrogen peroxide, H_2O_2, 3%, 0.5 mL
Steel wool, 2 × 2 cm, 0.5–0.7 g
Vinegar, white, 50 mL
Balance, 0.1 precision
Beakers, 250-mL, borosilicate glass, 3
Beaker, 100-mL
Beaker tongs or hot vessel gripping device
Filter paper (size to fit funnel)
Funnel, utility
Graduated cylinders, 25-mL, 2
Heat resistant pad, ceramic
Hot plate or Bunsen burner setup
Paper, notebook (chromatography or construction paper optional)
Pipet, Beral-type, graduated
Plastic coffee stirrer
Scissors
Stirring rod
Support ring with clamp
Support stand
Tap water, 100 mL
Tea bag, black pekoe

Safety Precautions

A small portion of the population finds acacia to be an allergen. While a 3% solution of hydrogen peroxide is very weak, it is an oxidizer and a skin and eye irritant. Avoid contact of all chemicals with eyes and skin. The iron(III) tannate solution will stain clothing. Exercise caution when using a hot plate or Bunsen burner. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Please review current Material Safety Data Sheets for additional safety, handling, and disposal information.

Preparation

Concentrated tea solution: Measure 100 mL of tap water into a 250-mL beaker. Heat to boiling using a hot plate or Bunsen burner. Remove beaker from heat with beaker tongs or hot vessel gripping device and place the beaker on a heat-resistant pad. Add a tea bag to the hot water and let it steep for at least 5 minutes. Remove and discard bag. The concentrated tea solution is rich in tannic acid.

Iron(II) ion solution: Measure 50 mL of vinegar into a 250-mL beaker and add a small piece of steel wool (about 2 × 2 cm or 0.5–0.75 g). Heat to boiling on a hot plate or with a Bunsen burner. Reduce heat and simmer for 7 minutes. Remove the beaker from heat with beaker tongs and place the beaker on a heat-resistant pad. Allow the solution to cool for a few minutes. Set up a filtration system with the support stand and ring. Remove the piece of steel wool with beaker tongs and discard. Filter the solution through a funnel lined with folded filter paper. Collect the filtrate in a clean, 250-mL beaker and allow to cool to room temperature. The filtrate contains dissolved iron(II) ions.

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Procedure

1. Measure 0.5 mL of 3% hydrogen peroxide with the graduated, Beral-type pipet. Add the hydrogen peroxide to the cooled iron solution.
2. Observe any changes in the color or appearance of the iron solution upon addition of hydrogen peroxide. Iron(III) ions have been produced.
3. Measure 10 mL of the strong tea using a graduated cylinder. Pour into a clean, 100-mL beaker.
4. Measure 10 mL of the iron(III) solution in a clean graduated cylinder. Add to the tea in the 100-mL beaker. Stir with a stirring rod. Observe the color and appearance of the iron(III) tannate ink mixture.
5. Add 1–2 g of acacia to thicken the ink and to keep the pigment in suspension.
6. Cut one end of a plastic coffee stirrer at an angle with scissors to use as a "quill." Dip the stirrer into the ink and write your name or a short message on a sheet of notebook or construction paper.

Disposal

Please consult your current *Flinn Scientific Catalog/Reference Manual* for general guidelines and specific procedures governing the disposal of laboratory waste. The iron mixture made in this demonstration may be disposed of down the drain with plenty of excess water according to Flinn Suggested Disposal Method #26b.

Tips

- Iron(II) ions will be produced by adding steel wool to vinegar and letting it sit for 2–3 days. No boiling is necessary in this case. Bubbles from hydrogen gas being produced can be observed.
- The more ink deposited on the paper, the darker the writing will be, but the longer the ink will take to dry. Using a more absorbent paper, such as construction paper or chromatography paper, will help speed the drying process.
- A micro tip Beral-type pipet may also be used as a quill.
- This would be an excellent and easy activity to do as a science component for an integrated unit on medieval or early American history.
- Making Ink is available from Flinn Scientific as a student laboratory kit, "Drawing Like DaVinci—A Recipe for Medieval Ink" (Catalog No. AP7158).

Discussion

Iron-gall ink has been used for centuries. Leonardo da Vinci (1452–1519) wrote his notes with it, Johann Sebastian Bach (1685–1750) composed with it, and the United States Constitution (1787) was drafted with it.

Oak galls are growths on tree leaves and stems caused by the egg-laying activities of gall wasps or gallflies. To make ink, the galls were collected and crushed, producing gallotannic acid. This was then mixed with water to form gallic acid. Iron(II) sulfate was mixed with water and added to the gallic acid to create the pigment, and finally gum arabic (acacia) was mixed in to thicken the ink and keep the pigment in suspension.

Since parchment (processed animal skin) was preferred by medieval scribes, the iron-gall ink worked better than carbon inks which smeared. The iron-gall ink actually "ate into" the paper by reacting with the collagen (a fibrous protein) in the parchment Some ancient documents actually have holes in them but can still be read by "reading the holes." Preservationists who work with ancient manuscripts deacidify such documents first and then remove any excess iron(II) compound in order to keep the writings from being destroyed completely.

In the ink-making reactions in this demonstration, hydrogen ions from the vinegar react with the iron in the steel wool to produce iron(II) ions and hydrogen gas. See Equation 1.

$$2H^+(aq) + Fe(s) \rightarrow Fe^{2+}(aq) + H_2(g) \qquad \textit{Equation 1}$$

The iron(II) ions (Fe^{2+}) are then oxidized by reaction with hydrogen peroxide to form iron(III) ions (Fe^{3+}). See Equation 2.

$$2Fe^{2+}(aq) + H_2O_2(aq) + 2H^+(aq) \rightarrow 2Fe^{3+}(aq) + 2H_2O(l) \qquad \textit{Equation 2}$$

Finally, the iron(III) ions react with tannic acid in the tea to form the black pigment, iron(III) tannate, which is insoluble in water.

Connecting to the National Standards

This laboratory activity relates to the following National Science Education Standards (1996):

Unifying Concepts and Processes: Grades K–12
- Systems, order, and organization
- Evidence, models, and explanation
- Constancy, change, and measurement

Content Standards: Grades 5–8
- Content Standard B: Physical Science, properties and changes of properties in matter
- Contents Standard G: History and Nature of Science, history of science

Content Standards: Grades 9–12
- Content Standard B: Physical Science, structure of atoms, structure and properties of matter, chemical reactions
- Content Standard G: History and Nature of Science, historical perspectives

Reference

This activity was adapted from "An Iron-Clad Recipe for Ancient Ink," *ChemMatters*, October, 2001.

Materials for *Making Ink—An Ancient Process* are available from Flinn Scientific, Inc.

Catalog No.	Description
A0001	Acacia, 100 g
H0009	Hydrogen peroxide, 3%
S0128	Steel wool
V0005	Vinegar, white
AP7158	Drawing Like DaVinci—A Recipe for Medieval Ink

Consult your *Flinn Scientific Catalog/Reference Manual* for current prices.