DYES AND DYEING

Dyeing of textiles has been practiced for thousands of years with the first written record of the use of dyestuff dated at 2600 BC in China. All dyes were natural substances obtained from plant, animal or mineral sources. In 1856, William Henry Perkin, while searching for a cure for malaria, discovered the first synthetic dye, Mauve. The mauve dye was a brilliant fuchsia color, but faded easily. Since that time, a great number of synthetic dyes have been manufactured and their resistance to running and fading has been almost eliminated. Almost all garments purchased today are dyes with synthetic dyes.

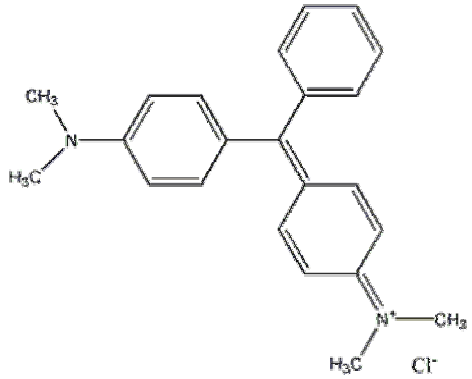
Dyes used for fabric such as cotton, wool, and silk are complex organic molecules that contain what is known as a chromophore group, that is, they contain some type of conjugated, alternating double and single bonds in part of the molecule. These molecules can absorb certain wavelengths of visible light and reflect the remaining light and, thus, give a fabric its color.

Not only do the dyes have polar or ionic groups, but fabrics such as cotton and wool also contain polar groups such as –OH (hydroxyl) and –NH (amide) which help the dye attach to the fabric.

Sometimes chemical bonds are formed between the dye and the fabric molecules which hold the two together. Another process involves the use of *a mordant,* which serves as sort of an intermediary that bonds the dye and the fabric. If the dye molecules attach firmly to the fabric, the color will be "fast", that is, it does not run when wet or washed, after the initial rinsing of excess dye.

This experiment will deal with four types of dyeing processes, direct dyes, mordant dyes, developed dyes, and vat dyes, along with an example of how dyes can be used to identify different fabrics.

Direct Dyes are molecules that adhere to the fabric molecules without help from other chemicals. In this experiment we will use a dye known as malachite green to illustrate this process.

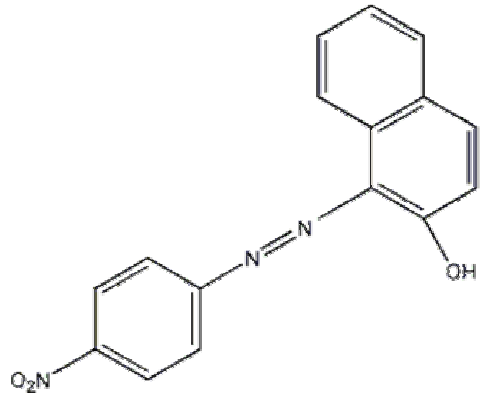


Malachite green (blue-green)

Mordant Dyes are dyes that do not adhere to fabrics directly. These need a chemical intermediate, known as a mordant, to attach themselves to the fabric. In this process, the mordant is applied to the fabric and then the dye is applied, which then bonds to the mordant. In this experiment, we will use tannic acid as a mordant to dye a sample of cotton with malachite green dye.

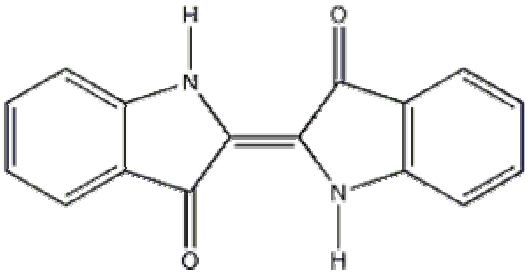
Developed Dyes are those where the dye-forming chemical reaction is carried out on the fabric. This process is commonly done with diazo dyes which are characterized by containing a -N=N- structure in the molecule. In this

experiment, we will use a dye known as para red which will be formed on the cotton fabric from the reaction between *p*-nitroaniline and 2-naphthol.



Para red (bright red)

Vat Dyes are insoluble in their colored form. They are reduced by another chemical and converted to a soluble form. The reduced dye is applied to the fabric, and then exposed to the air which oxidizes he dye back to its colored form. In this experiment indigo dye will be used to dye a sample of cotton fabric. Indigo is the dye used to make blue jeans blue.



Indigo

Materials Needed

6 beakers, 400 mL
beaker, 20 mL or 50 mL
Erlenmeyer flask, 125 mL, with stopper
3 stirring rods
graduated cylinders, 10 mL and 100 mL
tongs or forceps
beaker tongs
metal spatula
malachite green
p-nitroaniline
tannic acid
sodium nitrite, $NaNO_2$
2-naphthol
indigo powder
TIS Identification stain No. 1
TIS Identification stain No. 3A
iron(II) sulfate, $FeSO_4$
sodium hydrosulfite, $Na_2S_2O_4 \cdot 2H_2O$

hydrochloric acid, HCl, concentrated
sodium hydroxide, NaOH, pellets
5% sodium hydroxide solution, NaOH
10% acetic acid solution, $HC_2H_3O_2$
ammonia, NH_3, concentrated
Thermometer, -10 to 120°C
ice
cotton, strips, approximately 2 cm x 5 cm
wool, strips, approximately 2 cm x 5 cm
silk, strips, approximately 2 cm x 5 cm
multifiber fabric, strips, approximately 2 cm x 5 cm
rubber or plastic gloves

Safety Precautions

Wear safety goggles at all times in the laboratory

Wear rubber or plastic gloved to protect your hands from the dyes.

The dyes used in this experiment should be handled with the utmost care since they will dye the skin and clothing. Be careful to keep the dye powders and solutions off the desk tops. Clean up any spills immediately.

Most dye materials are irritants to the skin, eyes, and respiratory system and may be toxic by inhalation and ingestion. In the event of skin contact, wash the affected area well with water.

Hydrochloric acid is corrosive and can cause severe burns to the skin along with irritating fumes. Work with the concentrated solution under a hood. In the event of skin contact, wash the affected area well with water. If the skin is broken or blistered, seek medical assistance. If irritated by the vapors, get fresh air immediately.

Sodium hydroxide is caustic and can cause severe burns to the skin. Do not handle sodium hydroxide pellets with your hands. In the event of skin contact, wash the affected area well with water until there is no soapy feel. If the skin is broken or blistered, seek medical assistance.

Sodium nitrite is an irritant to the skin, eyes, and respiratory system. It is toxic by ingestion. In the event of skin contact, wash the affected area well with water. Sodium nitrite can also be an explosion hazard. Do not heat this compound dry or in contact with any flammable material.

Ammonia vapors are irritants to the eyes and respiratory system. Work with the concentrated solution under a hood. In the event of skin contact, wash the affected area well with water. If irritated by the vapors, get fresh air immediately.

Disposal and Clean up

Dispose of all materials in the specific containers provided in the laboratory.

Dye stains can be removed from glassware by applying a paste of powdered bleach and water, then rinsing. The paste will also remove stains from the hands, which should be washed off with soap and water.

Procedure

Direct Dyeing

Measure 0.1 g of malachite green.

Dissolve the malachite green in 200 ml of distilled or deionized water in a 400-mL beaker.

Heat the solution to boiling and place one strip each of cotton, wool, silk, and multifiber fabric in the solution. Allow the cloth strips to remain in the hot solution for 2 to 3 minutes.

Remove the strips of cloth with forceps or tongs and rinse thoroughly with water. Are the colors "fast"? Record your results.

Save the solution for the next dyeing procedure.

Mordant Dyeing

Measure 0.2 g of tannic acid.

Dissolve the tannic acid in 100 ml of distilled or deionized water in a 400-mL beaker.

Heat the solution to boiling and place a strip of cotton and multifiber fabric in the solution. Allow the cloth strips to remain in the hot solution for 1 to 2 minutes.

Remove the cloth strips from the tannic acid solution, place them on a paper towel and press the excess solution from them, then immerse in the hot malachite green solution saved from the first procedure. Allow the cloth strips to remain in the hot malachite green solution for 2 minutes.

Remove the cloth strips with forceps or tongs and rinse thoroughly with water. Are the colors "fast"? Record your results.

Developed Dyeing

Measure 3 mL of concentrated hydrochloric acid. Add it to a 400-mL beaker. Place the beaker under a fume hood.

Measure 0.1 g of p-nitroaniline. Add it to the hydrochloric acid in the 400-mL beaker.

Slowly, with stirring, add 10 mL of distilled or deionized water to the mixture. Place the beaker on an ice bath and cool the solution to about 5°C.

Measure 0.1 g of sodium nitrite and dissolve it in 5 ml of distilled or deionized water in a small beaker. Add this solution to the cold p-nitroaniline solution.

Measure 0.1 g of 2-naphthol (β-naphthol). Add it to 5 ml of 5% sodium hydroxide solution.

Immerse a strip of cotton and multifiber fabric in the *naphthol* solution. Remove the cloth strips and press to remove excess solution. Now immerse the cloth strips in the cold solution previously prepared. After the dye (para red) forms, test for colorfast properties and record your observations.

Vat Dyeing

Measure 0.1 g indigo powder in a 125-mL Erlenmeyer flask.

Add 0.1 g of sodium hydrosulfite ($Na_2S_2O_4$), 2 pellets of sodium hydroxide, and 10 mL of water to the flask. Stopper and shake for 2 to 3 minutes. Dilute to about 50 ml with distilled or deionized water.

Holding a cotton strip with forceps, dip the cloth into the dye solution for about 20 seconds. Remove the cloth and expose to air for a few minutes. Record your observations

Holding a multifiber fabric strip with forceps, dip the cloth into the dye solution for about 20 seconds. Remove the cloth and expose to air for a few minutes. Record your observations.

Test both cloth strips for color fastness and record the results.

> ***Alternative Procedure***
> If sodium hydrosulfite is not available, a mixture of 0.2 g indigo, 1.0 g ferrous sulfate, 5 ml water, and 5 ml concentrated ammonium hydroxide can be used. Heat the solution to boiling for a few minutes, allow the ferric hydroxide to precipitate, pour off the solution, and use in place of the one described in the procedure above.

Identification of an Unknown Fabric.

Dyeing tests involve the use of T.I.S. Identification stains (available from Testfabrics, Inc.). These are special mixtures of dyes that color different fibers different colors used to identify an unknown fabric or fibers.

Fabric Identifaction using T.I.S. Stain No. 1

To identify fibers or cloth samples, prepare a 1% solution of T.I.S. Identification Stain No. 1. Heat the solution to boiling. Maintain a hot, but not actively boiling solution.

Wet samples of cotton, wool, silk, and multifiber fabric, with distilled or deionied water. Squeeze out the excess liquid and place the samples in the hot dye bath for 3 to 5 minutes.

Remove the samples and wash out any excess dye.

Compare the color of the cloth samples with the multifiber fabric.

Fabric Identifaction using T.I.S. Stain No. 3A

A second color test can be conducted using T.I.S. Identification Stain No. 3A.

Prepare a 0.05% solution of T.I.S. Identification Stain No. 3A. (0.05 g for each 100 mL water) Heat the solution to boiling and add 1 mL 10% acetic acid solution. Maintain a hot, but not actively boiling solution.

Wet samples of cotton, wool, silk, and multifiber fabric, with distilled or deionied water. Squeeze out the excess liquid and place the samples in the hot dye bath for 5 minutes.

Remove the samples and wash out any excess dye.

Compare the color of the cloth samples with the multifiber fabric.

DYES AND DYEING
Data and Results

Name _____ Course and Section _____

Partner _____ Date _____

Attach fabric samples

Dye	Fabric	Results
Malachite green	Cotton	
Malachite green	Wool	
Malachite green	Silk	
Malachite green	Multifiber fabric	
Malachite green with Tannic acid mordant	Cotton	
Malachite green with Tannic acid mordant	Multifiber fabric	
Para red	Cotton	
Para red	Multifiber fabric	
Indigo	Cotton	
Indigo	Multifiber fabric	

Identification of an Unknown Fabric

Fabric Identifaction using T.I.S. Stain No. 1

Compare the colors of the cotton, wool and silk with the multifiber fabric

Fabric Identifaction using T.I.S. Stain No. 3A

Compare the colors of the cotton, wool and silk with the multifiber fabric