Teacher's Preparatory Guide

Polyaniline Synthesized Conducting Polymer for Applicable Uses as Nanomaterials

Overview: This lesson introduces students to polyaniline, a conducting polymer. It demonstrates how nanofibers significantly improve the processing of polyaniline and its performance as a conventional application involving polymer interactions with its environment (i.e. inorganic and organic uses). The lesson shows just one example how polyaniline has great technological potential due to its physicochemical characteristics (i.e. lightweight, conductivity, mechanical flexibility), and for its inexpensive methods for synthesis.

Purpose: For this lesson, the instructor or the students will synthesize polyaniline which will be used with cotton cloth to create "gloves" that are conductive for use with a smartphone during the wintertime. Students will learn about this useful and inexpensive approach for the application of conducting polymers as smart multifunctional textiles for fashion and personal comfort products.

Time required: (2) - 50 minute class periods; an additional 50 minute class period could be utilized in order to further discuss the applications of conducting polymers for consumer products.

Level: Advanced high school or undergraduate chemistry

Student Learning Objectives:

- The student will be able to predict how the materials and steps of the experiment will help synthesize polyaniline.
- The student will be able to perform the correct procedure for preparing the polyaniline-dipped cloth and affix it to the finger of a pair of gloves.
- The student will be able to observe and record what is happening at each step of the activity.
- The student will be able to summarize the experiment.
- The student will be able to explain some of the beneficial uses of conducting polymers for industrial, commercial, and academic markets.

Teacher Background: This lesson contains content that could and would be covered in an electrochemistry or organic chemistry course. It would be beneficial for the teacher to review educational material on redox reactions and synthesis reactions. A link to a series of informative videos on these topics is provided for convenience:

Inorganic Chemistry Review
http://education-portal.com/academy/lesson/redox-oxidation-reduction-reactions-definitions-and-examples.html#lesson

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Organic Chemistry Review
https://www.khanacademy.org/science/organic-chemistry

Brief History on Discovery of Conducting Polymers

A tremendous amount of research has been carried out in the field of conducting polymers since 1977 when the polymer polyacetylene was discovered to conduct electricity. The 2000 Nobel Prize in Chemistry awarded to Alan Heeger, Alan MacDiarmid, and Hideki Shirakawa recognized the discovery of conducting polymers and over 25 years of progress in this field (https://www.nobelprize.org/prizes/chemistry/2000/summary/) . In recent years, there has been growing interest in research on conducting polymer nanostructures (i.e., nano-rods, -tubes, -wires, and -fibers) since they combine the advantages of organic conductors with low-dimensional systems. A low-dimensional system typically is at the nanoscale 1-100nm and occur where at least one of the three dimensions is intermediate between the characteristics of atoms/molecules and the characteristics of the bulk material. At this level, interesting physicochemical properties occur that may have potentially useful applications.

For example, the conductivity of polyaniline depends on its ability to transport charge carriers along the polymer backbone and between polymer chains. Chemical interactions with polyaniline that alter either of these carrier processes affect its conductivity. Dopants are used to change these properties (see NNCI lesson on dopants https://www.nnci.net/node/5377) A dopant is a trace element (impurity) that is inserted into a substance (in low concentrations) to change the material's electrical properties. Dedoped polyaniline chains are nearly neutral and without charge carriers to transport electrical charges. This is responsible for the low conductivity of dedoped polyaniline. Protonation, or the addition of a proton, of the imine nitrogens by exposure to acid will introduce positive charge carriers to the polymer chain and increase the conductivity of the polyaniline.

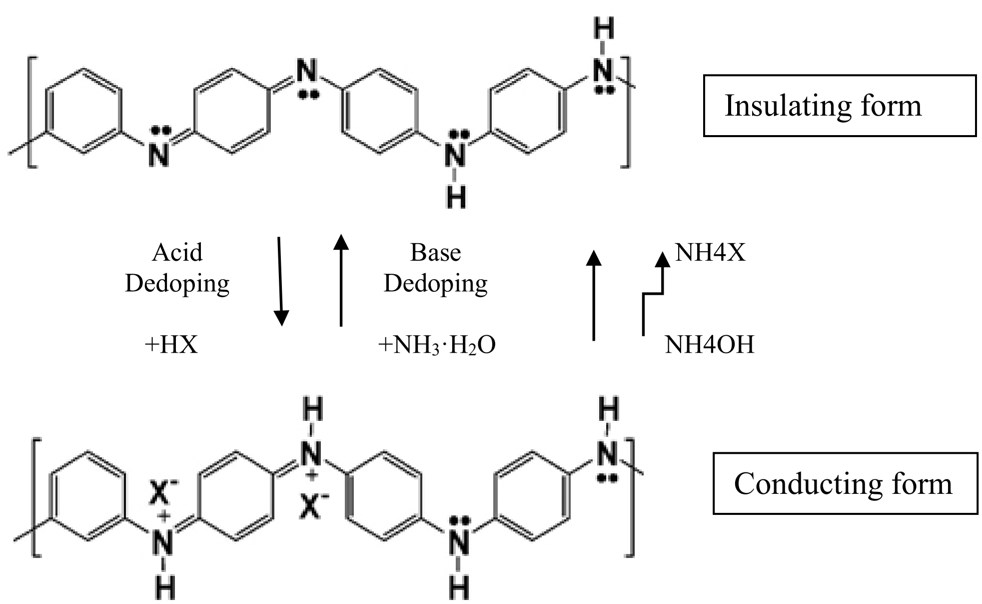

Figure 1. Reversible doping mechanism in polyaniline. HX= protonic acid

The reversibility and the control of the doping/dedoping mechanism in polyaniline is the underlying property that makes polyaniline useful in many applications such as sensors (gas, pH,

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etc). Due to their low cost, room temperature sensitivity, ease of deposition onto a wide variety of substrates, and the chemistry of structural modifications, polyaniline and other conducting polymers promise new materials and devices with many applications. Current areas of application include drug delivery, display devises, photovoltaic cells, polymer LEDs and micro/nano electronics. Finally, an advantage of polymeric materials, such as polyaniline, is that they can be synthesized and processed on a large scale at relatively low cost.

There are two methods for synthesizing polyaniline as discussed below:

Method 1. Chemical Synthesis of Polyaniline Using the Interfacial Polymerization Method
Interfacial polymerization is performed in an aqueous/organic biphasic system with aniline dissolved in an organic solvent along with the oxidant, ammonium peroxydisulfate, dissolved in an aqueous acid solution. Nucleation centers can be seen appearing to drop between the two layers. To see the resultant layers visit: https://www.youtube.com/watch?v=HF0a5AOIskw and https://www.youtube.com/watch?v=9ZpPUS23sT8. This method produces uniform polyaniline nanofibers with diameters below 100 nm. The process of polymerization can be readily scaled to make kilogram quantities.

Method 2. Chemical Synthesis of Polyaniline Using the Rapid Mixing Method
Polyaniline is a precipitated product from an aqueous solution containing typical reagents such as an oxidation agent ammonium peroxydisulphate (aka persulfate), and dopant acids like hydrochloric, sulphuric, nitric, perchloric, and monomer aniline. This direct route method represents the classical approach to polyaniline synthesis. This route causes aniline, the monomer, to be converted directly to a conjugated polymer by a condensation process. This is the method utilized in this activity.

Videos: There are some very good YouTube videos that would be helpful for students to view before doing the activity:

- Brown University's Science Center - Conductive Polymers https://www.youtube.com/watch?v=UjMbwS0LOkU
- Northwestern University's Department of Materials Science and Engineering Polyaniline Touch Gloves https://www.youtube.com/watch?v=YStXwgSmz_E and Polyaniline Cloth for Capacitive Touch Screens - https://www.youtube.com/watch?v=XxkCMcEfW1Y
- A Plastic that Conducts Electricity? https://www.youtube.com/watch?v=DlLZVxNK3Jg

Vocabulary:

Low dimensional system: a low dimensional system is a system where the motion in one or more directions is severely restricted. Here, the moving particles effectively behave as is they are in two dimensions (ex.: 2D electron gas), one dimension (nanowire), or even zero dimensions (a quantum dot). Definition from https://www.cityu.edu.hk/phy/low-dimensional-systems

Protonation: transfer of a proton to (a molecule, group, or atom), so that a coordinate bond to the proton is formed. The addition of a proton to an atom, molecule, or ion, forming the conjugate acid.

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Deprotonation: also refered to as dehydronation, is the removal (transfer) of a proton (or hydron, or hydrogen cation), (H^+) from a Brønsted–Lowry acid in an acid-base reaction. The species formed is the conjugate base of that acid. Definition from https://en.m.wikipedia.org/wiki/Deprotonation .

Dopant: a substance used to produce a desired electrical characteristic in a semiconductor. A **dopant**, also called a **doping agent**, is a trace of impurity element that is introduced into a chemical material to alter its original electrical of optical properties. The amount of dopant necessary to cause changes is typically very low. See https://en.wikipedia.org/wiki/Dopant for more information.

Materials for Teacher Preparation of Polyaniline using Rapid Mixing Method

- 0.2 M aniline hydrochloride (purum; 2.59 g, 20 mmol) CAS# 142-04-1
- 0.25 M Ammonium peroxydisulfate (ammonium persulfate; purum; 5.71 g, 25 mmol) CAS# 7727-54-0
- 100 mL N-Methly-2-pyrrolidone (NMP) CAS# 872-0-4 or formic acid
- (3) 100 mL portions of 0.2 M HCl
- (3) 100 mL portion of acetone
- 100 mL of distilled water
- (2) volumetric flask (200 mL)
- 4 filter papers
- funnel
- wash bottle
- beaker (250 mL) (to catch wash chemicals)
- beaker (500 mL)
- vacuum chamber (not required; may leave cloths overnight to dry)
- micropipette (100 – 1000 μL)
- 20 mL glass vial
- lab apron
- chemical gloves
- chemical goggles

PROCEDURE: Must be done in a fume hood

Preparation of Polyaniline (PANI) – **Instruction can do this procedure 2 – 3 days prior to the lab if students are not doing the synthesis.**

1. Dissolve 2.59 g of aniline hydrochloride in 50 mL of distilled water in a volumetric flask; stir well until dissolved. Keep at room temperature (~18 – 24 ˚C).
2. Dissolve 5.71 g ammonium peroxydisulfate in 50 mL of distilled water in a volumetric flask and stir well until dissolved. Keep at room temperature for 1 hour (~18 – 24 ˚C).
3. Mix both solutions in a 500 mL beaker and stir briefly. Allow to rest for 24 hours to polymerize. The polyaniline will precipitate out of solution.

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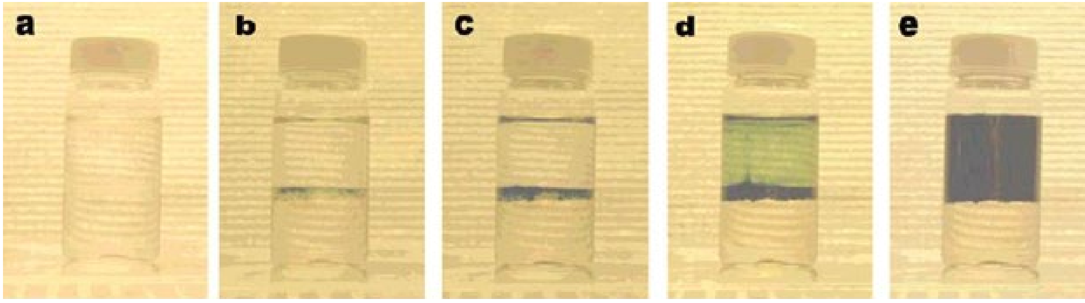

Figure 2: Snapshots showing interfacial polymerization of aniline in a water/chloroform system. From a to e, the reaction times are 0, 1.5, 2.5, 4, and 10 minutes respectively. The top layer is an aqueous solution of 1.0 M perchloric acid and ammonium, the bottom layer is aniline dissolved in the organic solvent chloroform.

4. The next day, collect the polyaniline precipitate on a filter and wash with 100-mL portion of 0.2 M HCl. Discard wash solution per lab procedures.
5. Then wash the polyaniline precipitate in 100-mL portion of acetone. Discard wash solution per lab procedures.
6. Repeat steps 4 & 5 two more times.
7. Dissolve the polyaniline in 10 mL of N-Methly-2-pyrrolidone (NMP) or formic acid to de-dope the polyaniline. (Should be enough for 2 – 3 groups per class; enough to saturate the cotton fibers)
8. The solution is now ready to be used with the fabric in the student procedure.

Advance Preparation: The teacher will make the polyaniline polymer solution prior to the lab using a fume hood using above method. If having students prepare the polyaniline, ensure sufficient hood space for lab groups. The preparation time will take approximately two days, and will need to provide ~ 100 mL solution depending on class size.

Safety Information:
Working with the chemicals for the polymerization method requires the use of standard safety lab equipment including goggles, gloves, and fume hood. MSDS sheets for each chemical should be kept in a binder located in the laboratory or classroom.

Suggested Pre Activities:
1. Introduce students to nanotechnology. You may assign them to read Nano 101 on the National Nanotechnology Initiatives website (https://www.nano.gov/nanotech-101). There are also numerous You Tube videos that introduce the topic.
2. Have students view the recommended videos on conducting polymers and polyaniline.
3. Review redox reactions and synthesis reactions.
4. Have students read through the background information on the student sheet. They should answer all questions about the concepts before beginning the activity.

Cleanup: Special care must be taken to dispose of all chemicals from the lab. Follow school procedures. **Do not dispose in the sink.** All lab equipment and areas should be cleaned according to school protocols.

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Student worksheet
(with answers in red)

Polyaniline Synthesized Conducting Polymer for Applicable Use in Touch Gloves

Introduction:
This lesson will introduce you to polyaniline, a conducting polymer. You will explore how nanofibers significantly improve the processing of polyaniline and its performance as a conventional application involving polymer interactions with its environment (i.e. inorganic and organic uses). The lesson shows just one example how polyaniline has great technological potential due to its physicochemical characteristics (i.e. lightweight, conductivity, mechanical flexibility), and for its inexpensive methods for synthesis. For this lesson, the instructor or you will synthesize polyaniline, which will be used with cotton cloth to create "gloves" that are conductive for use with a smartphone during the wintertime.

Touch screen capable gloves can be expensive to buy and usually do not work very well in relation to their cost. Researchers have been exploring the use of conducting polymer fibers, such as polyaniline, to provide an alternative and inexpensive way to create touch screen gloves. In this lesson, you will use synthesized doped polyaniline solution to make inexpensive gloves that will have the ability to interact with the capacitive touch screen.

Essential Question:
Will a conducting polymer, such as polyaniline, provide the ability for a non-conducting pair of winter gloves to interact with the capacitive touch screen of a smartphone?

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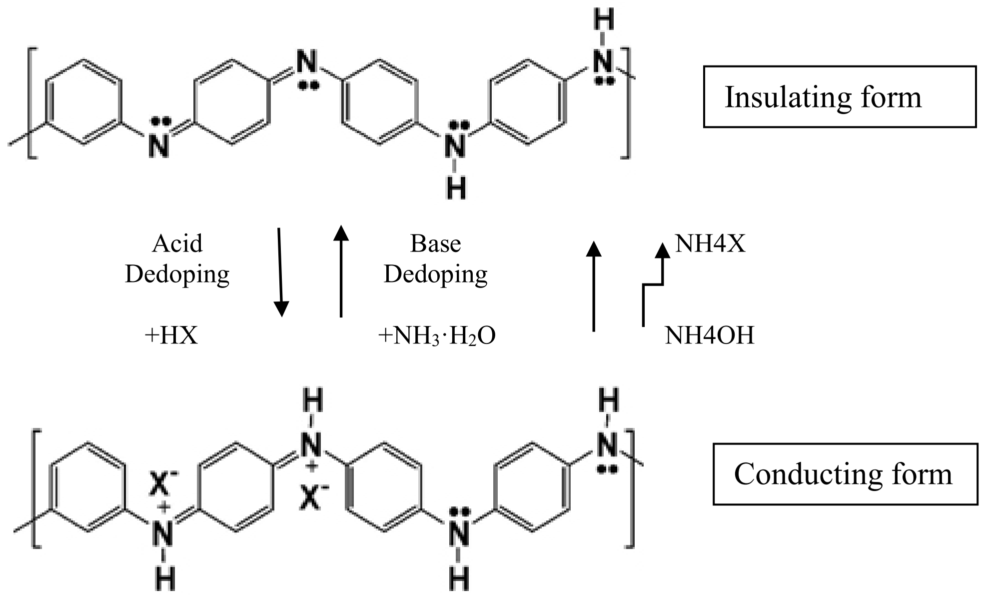
nitrogens by exposure to acid will introduce positive charge carriers to the polymer chain and increase the conductivity of the polyaniline.



Reversible doping mechanism in polyaniline. HX= protonic acid

The reversibility of the doping mechanism in polyaniline is the underlying property that makes polyaniline useful in many applications as sensors (gas, pH, etc). Due to their low cost, room temperature sensitivity, ease of deposition onto a wide variety of substrates, and the chemistry of structural modifications, polyaniline and other conducting polymers promise new materials and devices with many applications. Current areas of application include drug delivery, display devises, photovoltaic cells, polymer LEDs and micro/nano electronics. Finally, an advantage of polymeric materials, such as polyaniline, is that they can be synthesized and processed on a large scale at relatively low cost.

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Method 2. Chemical Synthesis of Polyaniline Using the Rapid Mixing Method

Polyaniline is a precipitated product from an aqueous solution containing typical reagents such as an oxidation agent ammonium peroxydisulphate (aka persulfate), dopant acids like hydrochloric, sulphuric, nitric, perchloric, and monomer aniline. This direct route represents the classical approach to polyaniline synthesis. This route causes aniline, the monomer, to be converted directly to a conjugated polymer by a condensation process. This lesson uses the Rapid Mixing Method to create the polyaniline.

Advanced Preparation (2-3 days prior to student activities below)

Chemical Synthesis of Polyaniline (Rapid Mixing Method) - Your instructor may prepare the polyaniline in advance; if done by instructor, skip to Day 1 activities. If synthesis is to be done by students, follow procedure below and as well as all safety precautions for your lab.

Materials: To make 100 mL of solution

- 0.2 M aniline hydrochloride (purum; 2.59 g, 20 mmol) CAS# 142-04-1
- 0.25 M Ammonium peroxydisulfate (ammonium persulfate; purum; 5.71 g, 25 mmol) CAS# 7727-54-0
- 100 mL N-Methly-2-pyrrolidone (NMP) CAS# 872-0-4 or formic acid
- (3) 100 mL portions of 0.2 M HCl
- (3) 100 mL portion of acetone
- 100 mL of distilled water
- (2) volumetric flask (200 mL)
- 4 filter papers
- funnel
- wash bottle
- beaker (250 mL) (to catch wash chemicals)
- beaker (500 mL)
- vacuum chamber (not required; may leave cloths overnight to dry)
- micropipette (100 – 1000 μL)
- 20 mL glass vial
- lab apron
- chemical gloves
- chemical goggles

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Procedure:

1. Dissolve 2.59 g of aniline hydrochloride in 50 mL of distilled water in a volumetric flask; stir well until dissolved. Keep at room temperature (~18 – 24 ˚C).
2. Dissolve 5.71 g ammonium peroxydisulfate in 50 mL of distilled water in a volumetric flask and stir will until dissolved. Keep at room temperature for 1 hour (~18 – 24 ˚C).
3. Mix both solutions in a 500 mL beaker and stir briefly. Leave at rest for 24 hours to polymerize. The polyaniline will precipitate out of solution.
4. The next day, collect the polyaniline precipitate on a filter and wash with 100-mL portion of 0.2 M HCl. Discard wash solution per lab procedures.
5. Then wash the polyaniline precipitate in 100-mL portion of acetone. Discard wash solution per lab procedures.
6. Repeat steps 4 & 5 two more times.
7. Dissolve the polyaniline in 10 mL of N-Methly-2-pyrrolidone (NMP) or formic acid to de-dope the polyaniline. (Should be enough for 2 – 3 groups per class; enough to saturate the cotton fibers)

Day 1 Student Activity:

Materials per group:

- 1 set of gloves
- 4 pieces of cotton T-shirt
- 1 20 mL glass vial
- 1 glass petri dish
- 1 multimeter
- 1 scissors
- 1 tape dispenser
- 1 lab apron
- 1 chemical gloves
- 1 chemical goggles
- Polyaniline solution (shared among groups)

Procedure:

1. Using tweezers, soak your cotton cloth pieces in the polyaniline solution until saturated.
2. Using the tweezers, place the pieces into the glass vials and ready the container for the high vacuum drying or overnight drying in a fume hood.
3. The cotton cloth pieces will be vacuumed for approximately 8 hours as NMP has a very high boiling point.

Day 2: Lab Procedures
(Make sure all cotton samples are completely dried)

Step 1: Polyaniline was synthesized by using the Rapid Mixing Method. **(May have been done by the teacher)**

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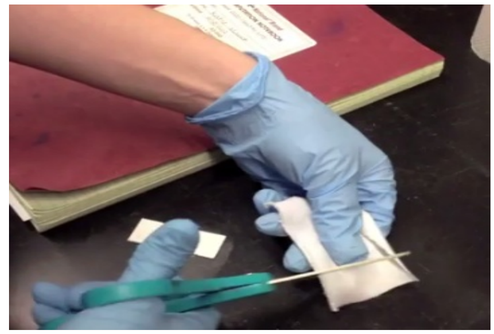
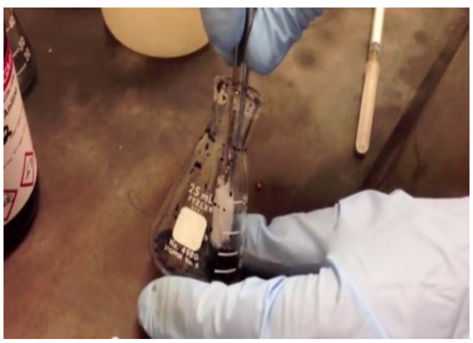
Step 2: Next, the polyaniline was dissolved in N-Methly-2-pyrrolidone (NMP) or formic acid which de-doped the polyaniline. **(May have been done by the teacher)**

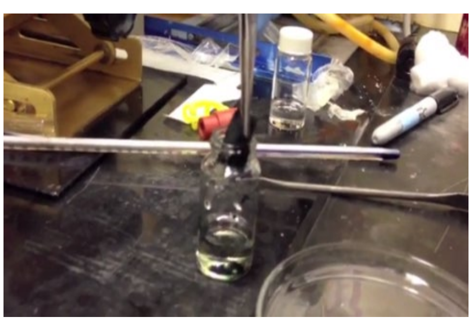


Step 3: A cotton T-shirt or similar fabric was cut up in small squares as test materials. **(May have been done by the teacher)**



Step 4: Using tweezers, dip the small pieces of fabric into the NMP solution with the dissolved polyaniline fibers. The polyaniline solution fully impregnates the fibers of the fabric upon immersion.

Step 5: Place the dipped pieces of fabric into vials and dry under high vacuum as NMP has a very high boiling point. It is best to vacuum for 8 hours to ensure the fabric is completely dry. Alternatively, dry overnight in a fume hood.



Step 6: Dip two of the dried pieces of cloth into hydrochloric acid to re-dope the polyaniline polymer. Gently dry one of the re-doped pieces of the fabric, which will cause the fabric to turn a greenish color indicating the polyaniline polymer has been re-doped. Keep another piece wet. Once doped fabric piece is dry, place the four pieces of fabric on a petri dish. One of each: untreated, treated de-doped, treated doped (wet), treated doped (dry). Note the placement of the samples so you know which one you are testing.

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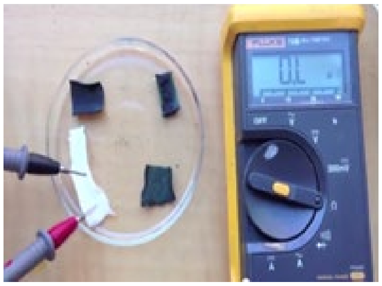
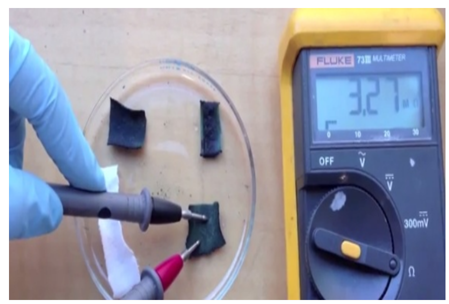


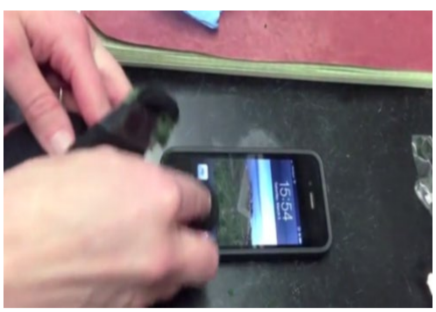
Step 7a: Measure the conductivity of the 4 cotton fabric samples. Begin with the untreated cotton fiber fabric square sample as the control. Record the resistance in your data table.

Step 7b: Test the de-doped PANI-treated fabric sample. Record its resistance.

Step 7c: Test the freshly doped (wet) PANI-treated fabric. Record result.



Step 7d: Test the doped (dry) PANI-treated fabric. Record result.



Step 8: Test the material by using a set of winter gloves that were not treated with the doped PANI-treated fabric.

Step 9: Attach the dry doped fabric to the glove. Use tape to adhere the fabric to the glove. Test the winter gloves using the doped PANI-treated fabric to shows it can easily open the screen and allows the ability to interact with the capacitive touch screen. This is a crude method for showing how conducting polymers could provide an inexpensive way to create gloves that are conductive for smartphone winter time use.

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Cleanup: Special care must be taken to dispose of all chemicals from the lab. Do not dispose in the sink. All lab equipment and area can be cleaned according to normal protocol.

Name:

Pre-lab questions

Prediction

1. What do you think the resistance will be for the untreated cotton sample? Why?
   It would be zero as cotton is not conductive.

2. Do you think the resistance will increase once the cotton sample has been treated with the doped polyaniline solution? Why? Yes it will increase as the doing introduces an impurity that changes the materials electrical properties.

Experiment

1. As you do the activity, fill out this table with questions that you have and observations that you make. Be as detailed as possible about what you observe at each step of the experiment.

Observations	Questions

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2. Record the conductance for each cotton fabric.

Cotton Fabric	Conductivity (Ω)
Untreated	0
Treated De-doped	~0
Treated Doped (Wet)	1.6KΩ
Treated Doped (Dry)	3.2MΩ

POST-LAB ASSESSMENT QUESTIONS:

1. Why did you place the dried polyaniline-dipped cotton cloth into the hydrochloric acid?
Answer: Dipping the treated polyaniline-dipped cotton cloth into hydrochloric acid, or any strong acid, causes the molecule to be protonated, or doped, and will reduce the resistance.

2. What do you think would happen to the resistance if the treated cotton fabric was exposed to a base instead of a strong acid?
Answer: By adding polyaniline to a base, the molecule is dedoped (deprotonated), which increases resistance.

3. What is the resistance of the untreated cotton fabric? What is the resistance of the doped-polyaniline dipped cotton fabric?
Answer: The resistance of the untreated cotton fabric is 0 kΩ. The resistance of the doped polyaniline-dipped cotton fabric should be around 3.2 MΩ.

4. What do you think would happen to the resistance if the treated cotton fabric was exposed to a base instead of a strong acid?
Answer: By adding polyaniline to a base, the molecule is dedoped (deprotonated), which increases resistance.

5. What is the chemical formula of vinegar, hydrochloric acid and ammonia? Are they acids or bases? Which is the strong acid? Why?
Answer: Vinegar (CH3COOH) and hydrochloric acid (HCl) are acids. Hydrochloric acid is a strong acid because all of the H+ ions will dissociate in solution, whereas vinegar is a weak acid because not all of the H+ ions dissociate in solution. Ammonia (NH3) is a base.

6. What are some uses for polyaniline as a conducting polymer?

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Answer: Polyaniline have potential for applications due to their light weight, conductivity, mechanical flexibility and low cost. Polyaniline is especially attractive because it is relatively inexpensive, has three distinct oxidation states with different colors and has an acid/base doping response. This latter property makes polyaniline attractive for acid/base chemical vapor sensors, supercapacitors and biosensors. The different colors, charges and conformations of the multiple oxidation states also make the material promising for application as actuators, supercapacitors and electrochromics. They are suitable for manufacture of electrically conducting yarns, antistatic coatings, electromagnetic shielding, and flexible electrodes.

7. How would you change the methodology to improve the conductance of the polyaniline treated fabric? Be creative in developing a different design solution.
   Answers will vary but could include changing the dopant, the fabric, the size of the fabric, concentration of the polyaniline.

8. How is this activity connected to nanotechnology?
   Answers will vary but should include that the results polyaniline fibers are nanofibers; the chemical reaction to create the polyaniline occurs at the molecular/atomic level (nanoscale); the electrical properties are at the nanoscale.

Additional information on conductive polymers:
Literature: Numerous articles have been published on conductive polymers and polyaniline in particular. The resource section of this activity provides several of these for further reading on the chemistry of these unique polymers, their synthesis, and possible applications.

Resources:

9. J.Huang and R.B. Kramer. A general chemical route to polyaniline nanofibers.. J. Am. Chem. Soc. 126, 851-855 (2004).
10. Masoud Mozafari and Narendra Pal Singh Chauhan (Eds). *Fundamentals and Emerging Applications of Polyaniline, 2019.* Elsevier, The Netherlands.
11. T. J. Skotheim, R. L. Elsenbaumer, J. R. Reynolds. *Handbook of Conducting Polymers*, 2nd ed., p. 1097, Marcel Dekker, New York (1998).
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Next Generation Science Standards

- **HS-PS1.A** Structure and Properties of Matter
    - The structure and interactions of matter at the bulk scale are determined by electrical forces within and between atoms.
- **HS-PS2-6** Communicate scientific and technical information about why the molecular-level structure is important in the functioning of designed materials.
- **HS-PS1.B** Chemical Reactions
- **HS-ETS1.C** Optimizing the Design Solution

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