Internet of Things (IoT) at UCF

UNIVERSITY OF CENTRAL FLORIDA

Design and Fabrication of Environmental Sensors

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UCF

SUMMARY

I approached the R.E.T. summer research project with 3 goals in mind:

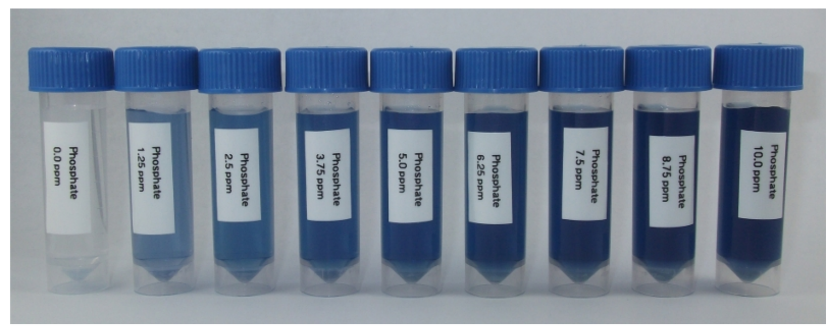
1. The Florida curriculum for Chemistry is extremely dated, covering topics relevant to Chemistry not much beyond the science in the 1950's. I wanted to get exposure to cutting edge technology and to be schooled in its use for my labs.
2. Micro-sensor technology offers the opportunity to measure chemical elements and compounds directly in the field, which is a huge step beyond laborious biochemical testing (i.e. taking samples in the field and then completing assays in the lab).
3. I wanted to design and fabricate sensors and teach my students the same.

THE R.E.T. PROGRAM FULFILLED ALL MY GOALS

COMPARISON OF TECHNIQUES

BIOCHEMISTRY - STANDARD METHODOLOGY FOR ASCORBIC ACID PO_4 ASSAY

Ammonium molybdate and potassium antimonyl tartrate react in acid medium with orthophosphate to form a heteropoly acid, phosphomolybdic acid, that is reduced to intensely colored molybdenum blue by ascorbic acid. This method for measuring phosphate is based on APHA Standard Method 4500-P E



Reagents and Equipment

Assembled colorimeter
Cuvettes
5 mL screw capped tubes
Sulfuric acid, (14% v/v)
Potassium antimonyl tartrate solution
Ammonium molybdate solution (4%)
Ascorbic acid (0.1M)
1mL micropipette
Distilled water.
Colorimeter: Wavelength: 625 nm.
Assorted phosphate concentrations (pre-prepared)

Methodology

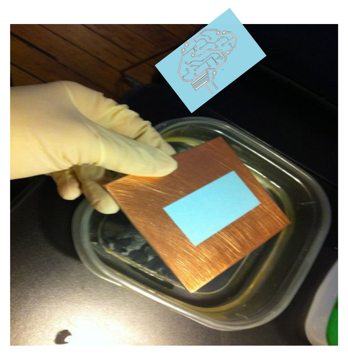
Step 1: In a beaker dissolve 0.88 g of ascorbic acid in 50 mL distilled water. In a separate beaker add the reagents in the following order:
25 mL sulfuric acid solution;
2.5 mL potassium antimony tartrate solution;
7.5 mL ammonium molybdate solution;
15 mL ascorbic acid solution (prepared in step 1);
Mix the ascorbic acid reagent. The reagent is stable for 4 hours.
Step 2: Fill an empty sample bottle to the 5 mL line with water sample. Add 800 µL of test reagent solution, cap and invert several times to mix.
After at least 5-10 minutes, pour sample into a cuvette, place in the colorimeter or spectrophotometer and measure.

Results

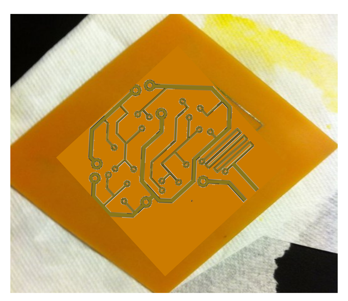
Graph the relationship of colorimetry readings to known concentrations of PO_4 in ppm

NANOTECHNOLOGY - METHODOLOGY FOR FABRICATION OF MEMS PO_4 SENSOR

Step 1: Design the board using a simple graphics program. Note that the design will be flipped when applied to the PCB board.
Step 2: Print out the design onto shiny side of the transfer paper.
Step 3: Sand the copper plate to create a rough surface for the design transfer.
Step 4: Wash the copper with some water and rubbing alcohol and let it dry



Step 5: Cut out the design and place face down on the copper. Leave a margin around the design for handling the coupon.
Step 6: Run the copper plate with the design face down through a hot laminator 5 times or use a hotplate instead of a laminator.



Step 7: Place the plate In a cold bath and agitate until the paper floats off,
Step 8: Place the PCB Into the KCl (or H_2SO_4) etching solution and agitate until all the copper has dissolved (30 min.).
Step 9: Rinse in water bath. Let It dry and rinse thoroughly with rubbing alcohol.
Step 10: Holes need to be drilled into the PCB coupon using a Dremel 1/64-inch cobalt drill bit, to receive the cobalt drill tips.

Results

Graph the relationship of potentiometer readings to known concentrations of PO_4 in ppm

LESSON PLAN

RET Site: CoMET Lesson/Unit Plan

TOPIC: COMPARATIVE ANALYSIS

Measurement of Environmental Contaminants: Micro-sensors vs Biochemical Analysis

Courses: Environmental Science
Chemistry

Grade Levels: 10-12

Length of Lesson: 3 labs (9 periods @ 50 minutes each)

- 1 period pre-lab prep
- 1 period lab experimentation
- 1 period lab report

1. Sensor fabrication
2. Calibration & testing
3. Sensor & biochemical results

Materials/Technology Needed

- Single-sided Printed Circuit Board (75 cm x 75 cm)
- Multimeter
- Alligator clips & connectors
- 0.1M potassium chloride (KCl)
- Potassium bi-phosphate samples (KH_2PO_4)
- Cobalt drill bits (1/64th in. or 0.015 mm)
- Hot plate
- Access to laser printer essential

Where this Fits

The lab examines state-of-the-art field testing of various environmentally significant chemicals. The intent of the lab is to compare the use of MEMS technology with standard biochemical testing. Cobalt micro-sensor performance will be compared with the 'Ascorbic Acid Method' of phosphate detection:

Lesson Objectives/Learning Goals

Students will fabricate, test, and utilize a micro-sensor of their own design. The target molecule is the phosphate ion (PO_4^{3-}) but alternates are suggested. They will also complee a standard biochemical test for phosphate, by way of comparison.

Standards/Benchmarks:

- Environmental Science
  - SC.912.1.17.20 - Impact on environmental systems
  - SC.912.1.17.14 - Adequate waste management strategies
- Chemistry
  - SC.912.P.8.8 - Oxidation/reduction reactions; types of reactions
  - SC.912.P.8.10 - Oxidation/reduction in living systems

Evidence of Learning (Assessment Plan)

- Pre-testing for the lab will be conducted by a short bellringer 'refresher' of basic concepts in electro-chemistry and the resolution chemical equations, followed by two self-testing interactive PhET simulations. (See 'Pre-Test' below.)
- The Lab Reports will form a major part of the assessment, reflecting as they do the students' appreciation of the concepts involved. Reports for the two separate labs will be prepared. The final test will be a formative 'Lab Test' in which I provide specific questions on the comparative analysis of the two techniques employed.

Instructional Strategies

- Simulations & modeling
- Fabrication and calibration of a micro-sensor
- Cooperative Learning
- Lab organization & methodology
- Inquiry-based Learning
- Formative assessment

Description of Lesson Activity

Pre-Lab:

1. Data on human contamination of the environment will be presented and discussed. The basics on the use of environmental sensing techniques will be reviewed, including problems and opportunities. The role of phosphates in the contamination of natural water resources will be presented by means of a PowerPoint presentation.

1.1 The PhET 'Conductivity' lab will be introduced for the Lab 1 Fabrication to understand the nature of electrochemistry. Then the students will watch the YouTube video '*How to etch PCB with ferric chloride*' (https://youtu.be/lXKwkcgimZI)

1.2 The PhET 'Reactants, Products and Leftovers' Simulation will be conducted prior to the ascorbic acid experiment to demonstrate the interaction of chemical reactants to produce products and how to predict the successive chemical reactions. Then the students will watch the YouTube video '*How to assay the free inorganic phosphorus*' (https://youtu.be/OUoEBwLjSJM)

Lab Experiments:

2. Two labs will be conducted, by way of comparison, one using a cobalt micro-sensor to obtain virtually immediate PO_4 readings from potentiometer readings and the other based on the time-honored ascorbic acid assay of PO_4 concentrations.

2.1 Lab 2a: The fabrication lab will create of an etched micro-sensor as per the methodology shown on the left panel. Then the sensor will be calibrated prior to performing test readings of various sample phosphate concentrations with a highly sensitive potentiometer.

2.2 Lab 2b: The Ascorbic Acid Method will be employed to biochemically measure PO_4 concentrations, demonstrating an alternate method for . A colorimeter or spectrophotometer can be employed to precisely measure light transmission through the cuvettes

Lab Reports:

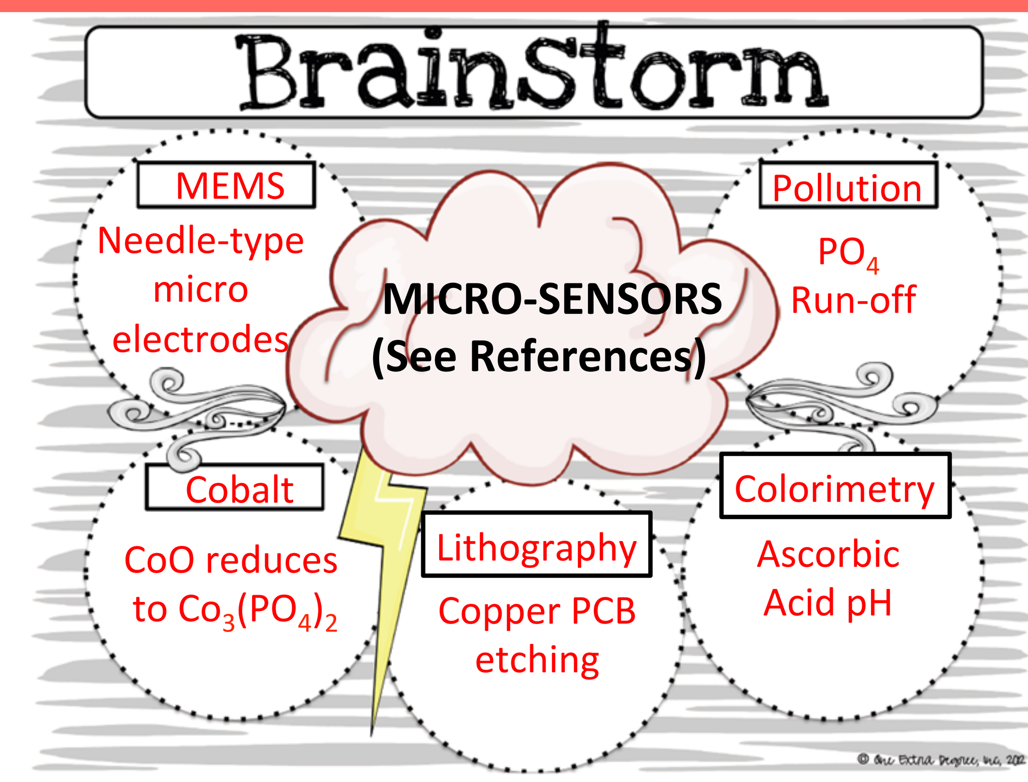
3.1 A full Lab Report will be written on the measurement of phosphate concentrations employing a cobalt micro-sensor. The protocol for writing lab reports will have been practiced many times at this stage of the course on previous labs.

3.22 A full Lab Report will be written on the measurement of phosphate concentrations employing the ascorbic acid method.

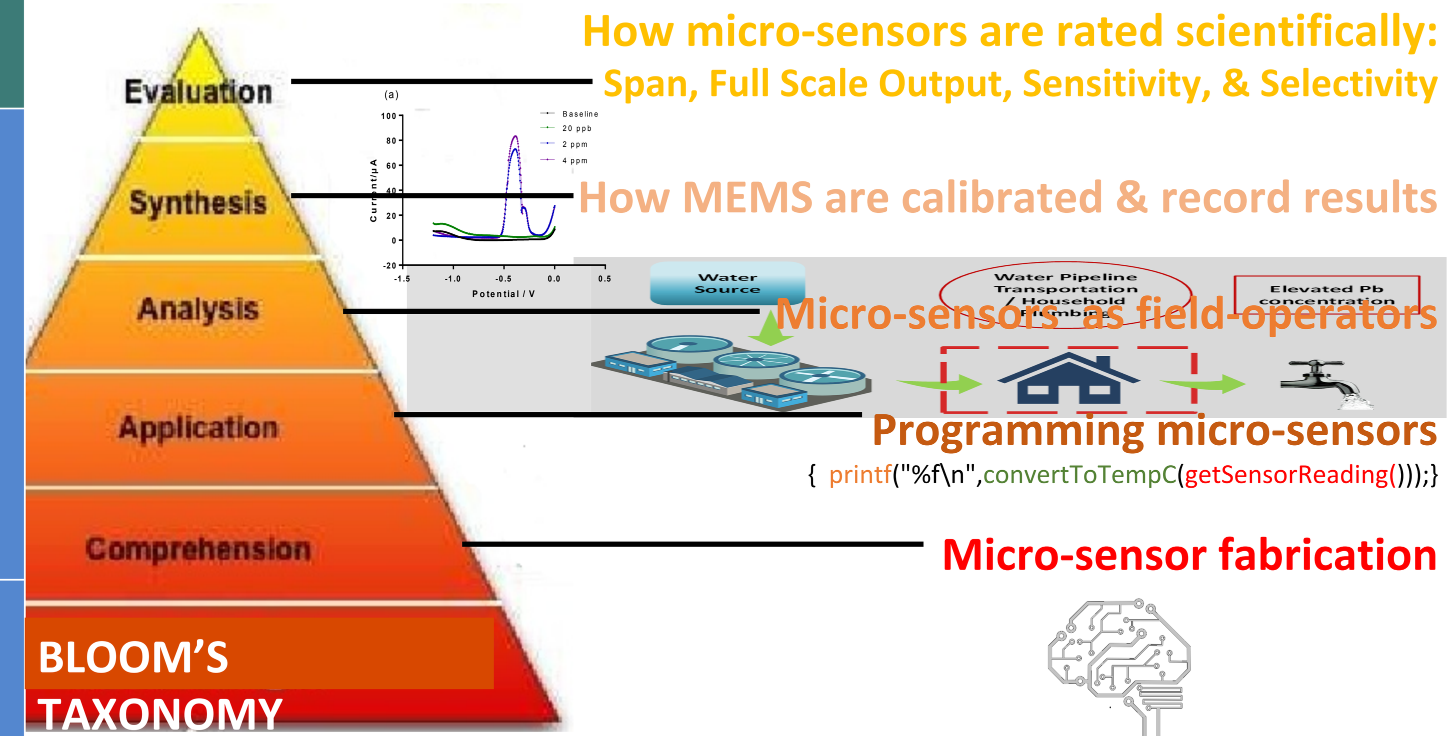
There will be a post-lab 'formative 'Lab Test' in which he comparative analysis of the labs will be completed based on questions I prepare to direct them towards a full discussion and dissection of what their lab results mean. They will also review the accuracy and precision of each technique.

List of Materials/Resources Used: See attached labs: *Phosphorus Determination using the Colorimetric Ascorbic Acid Technique* AND *Cobalt-coated needle-type microsensors*

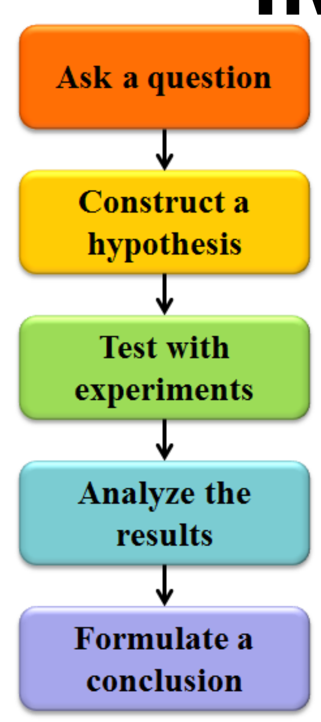
RESEARCH ACTIVITIES



LESSON LEARNED & ASSUMPTIONS



IMPLEMENTATION STRATEGY



How are pollutants measured?

Ions need to be isolated, identified and recorded. Construct the biochemistry involved.

Measure the isolated ion be measured indirectly: pH, electric potential, precipitates, temperature, etc.

Identify and characterize the regression pattern

Graph the curve and extract the formula.

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1. **A cobalt-coated needle-type microelectrode array sensor for *in situ* monitoring of phosphate;** Jin-Hwan Lee, Woo Hyoung Lee, Paul L Bishop and Ian Papautsky, Journal of Mechatronics and Microengineering, Volume 19, Number 2
2. **Scaling Issues in Chemical and Biological Sensors;** Marc J. Madou and Roger Cubicciotti, Proceedings of the IEEE, Vol. 91, No. 6, June 2003