



ISTITUTO ISTRUZIONE SUPERIORE “A. AVOGADRO”

(ENTE DOTATO DI PERSONALITA' GIURIDICA E DI AUTONOMIA AMMINISTRATIVA, ORGANIZZATIVA e DIDATTICA - R. D. 24.08.1933 N. 21933 - DP. N. 3917-P/C16 DEL 7.3.2000)

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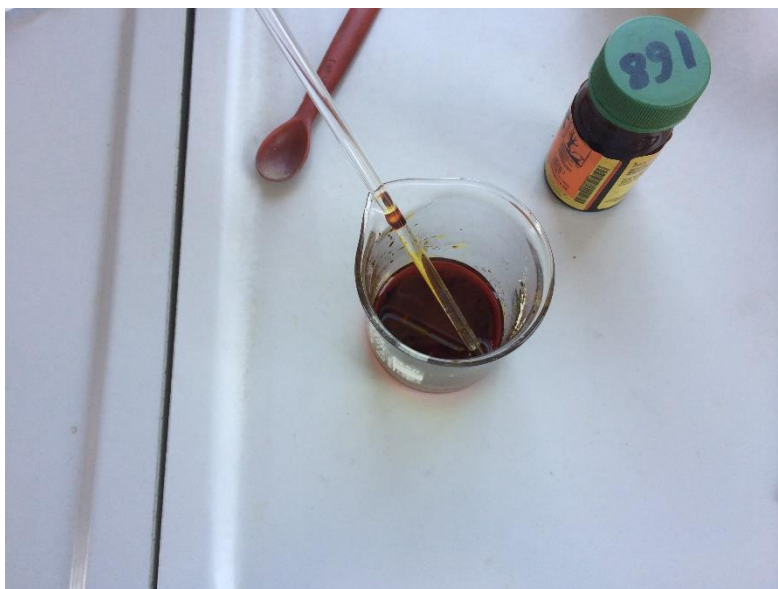
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DSSC – Assembling procedure

1. ELECTROLYTE PREPARATION

The electrolyte consists of an iodine/iodide water free ethylene glycol solution. In order to prepare this electrolyte solve 8,3 g of KI and 1,27 g of I_2 into 100 mL of ethylene glycol.

Note that it is very important to be sure there are no water leavings in the glass used to prepare the solution so it is recommended, before starting the preparation, to wash it with absolute ethanol and let it dry for 10-15 minutes. Store the electrolyte in a cold ($4^{\circ}C$ would be great) , dry and dark environment.



Ptc. 1) The typical color of a iodide/iodine solution.

2. DYE PREPARATION

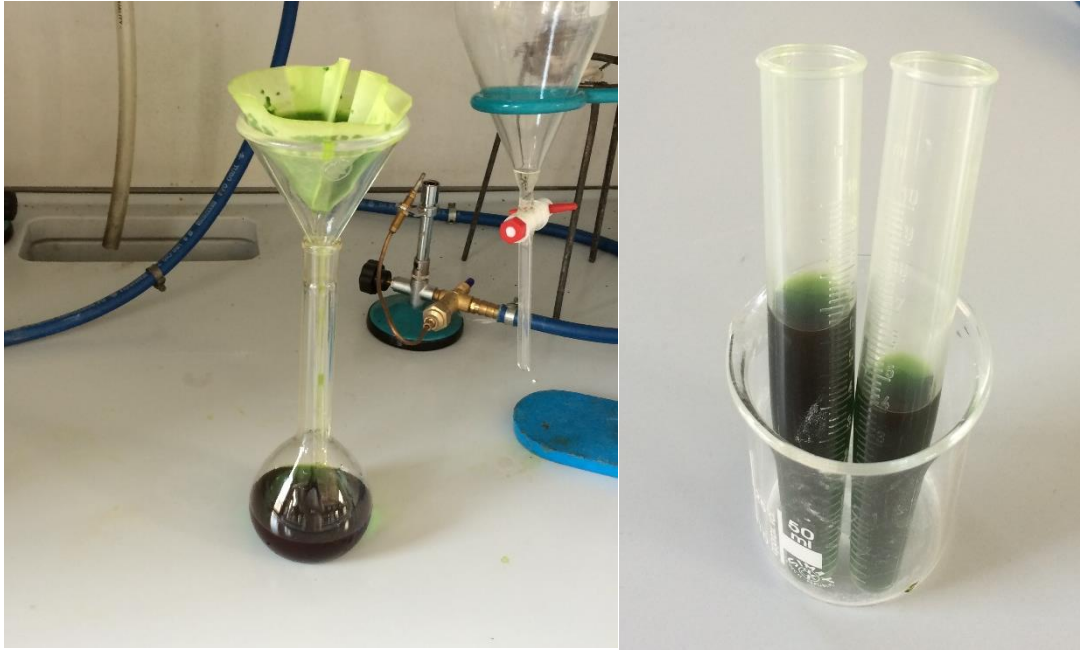
The extraction of chlorophyll from spinach leaves was performed without heating because of its instability; in fact, we opted to extract it with a solvent.

Cyanidin and betanin has been extracted with propanone and purified by phase separation with petroleum ether.

Apigenin was extracted by heating flower petals in absolute ethanol.

i. EXTRACTION OF CHLOROPHYLL

- Wash the leaf with some ethanol for 2 minutes to remove water leavings.
- Put 30 g of spinach leaves in a blender and add 0.5 g of sodium hydrogencarbonate NaHCO_3 to neutralize the leaf acids. Turn the blender on for 1 or 2 minutes.
- Transfer the leaves in a becker and add 150 mL of absolute ethanol.
- Stir energetically every 5 minutes and leave the extraction proceeds for at least 45 minutes in a dark and dry environment. Add 2 g of anhydrous sodium sulfate Na_2SO_4 to remove water leavings.
- Perform two filtrations of the extract to remove completely the cell fragments and the leavings of the inorganic salts that can be still present in the chlorophyll-ethanol solutions. OPTIONAL (but it improves the extract quality): centrifuge the extract at 3000 rpm for 5 minute.



Ptc. 2) Filtering chlorophyll ethanol solution (on left) and two centrifuge test tube containing the final chlorophyll extract (on right).

ii. **EXTRACTION OF APIGENIN**

- Wash 5 g of Chrysanthemum petals with absolute ethanol.
- Put the petal into an Erlenmeyer flask with 50 mL of ethanol and heat with a magnetic stirrer for 15 minutes. The temperature must be controlled continuously to avoid the ethanol to boil.
- Perform a filtration of the yellow solution to remove the petals and any other possible leavings.



Ptc. 3) Two steps of the apigenin extraction from yellow Chrysanthemum petals.

iii. **EXTRACTION OF CYANIDIN**

- Put 30 g of blueberries in a mortar and press them with a pestle. It is recommended to wash the blueberries with a 0.1 M acetic acid solution and rinse them with water.
- Add 30 mL of acetone and wait for 15 minutes. Every now and then, you can stir the mixture.
- Filter the extract and transfer it in a separator funnel.
- Add 20 mL of petroleum ether and 20 mL of water and shake energetically for 1 or 2 minutes. Then wait for 15 minutes for the phase separation.
- Remove the water/acetone phase and centrifuge it to remove some possible petroleum ether leavings.



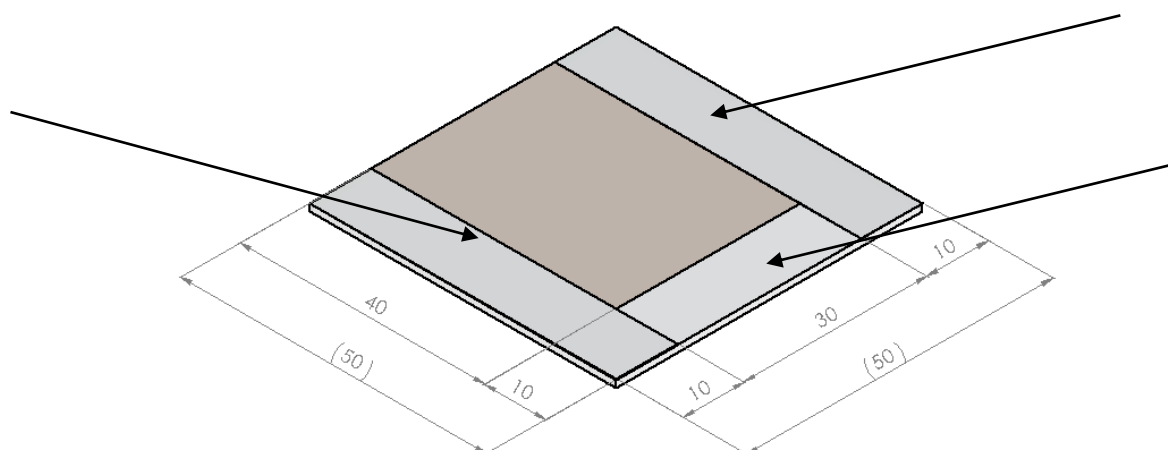
Ptc. 4) Blueberries in a diluted acetic acid solution, blueberries in acetone and phase separation extraction with petroleum ether (from left to right).

iv. **EXTRACTION OF BETANIN**

- Put 30 g of beetroots in a blender.
- Add 30 mL of acetone and wait for 15 minutes. Every now and then, you can stir the mixture.
- Filter the extract and transfer it in a separator funnel.
- Add 20 mL of petroleum ether and 20 mL of water and shake energetically for 1 or 2 minutes. Then wait for 15 minutes for the phase separation.
- Remove the water/acetone phase and centrifuge it to remove some possible petroleum ether leavings.

3. TiO₂ NANOSTRUCTURED LAYER PREPARATION

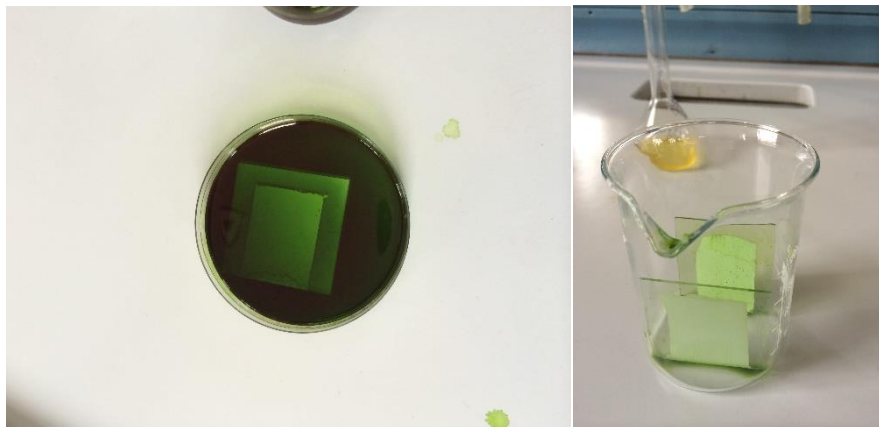
- Put the ITO glass on the table, being sure the conductive side is facing up.
- Place three strips of Scotch on the glass as shown below.



- Prepare 5 mL of a solution of nitric acid 0.002 M (or hydrochloridric) or acetic acid 0.2 M and 5 mL of ethanol and add to it 6 g of TiO₂ powder. Add 4 drops of a sodium laurilsulfate 0.001 M solution. Add some more acid if the solution is too firm. Wait for 15 minutes before going to the next step.
- Drip few drops of TiO₂ on the glass surface and immediately squeegee the solution down with the paste spreader. Let dry for a few minutes and then remove carefully the Scotch tape. Then remove the Scotch tape and wipe off any remaining with isopropyl alcohol and a paper towel.
- Let the electrode dry in a stove for 20 minutes at 250°C in air stream; once the drying is completed let slowly cool to room temperature. The TiO₂ darkening is a signal which means the sinterization has significantly occurred.
- Place the electrode into the dye solution for 15 minutes being sure that the TiO₂ layer is facing down.
- Rinse the electrode, first with distilled water, and then rinse again with ethanol and wait 10 minutes for the electrode drying.



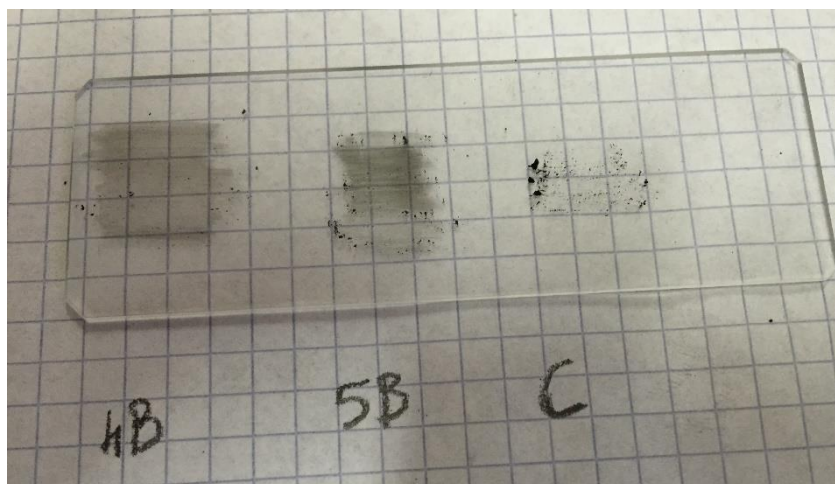
Ptc. 6) The preparation of the TiO_2 surface on conductive glass.



Ptc. 7) Dye absorption on TiO_2 layer.

4. COUNTER ELECTRODE PREPARATION

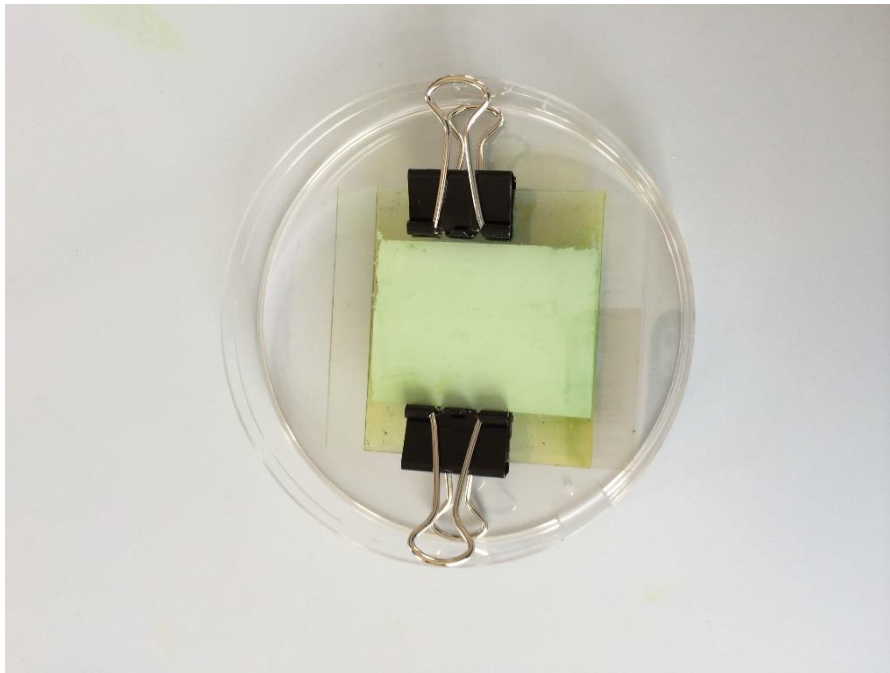
- Clean the glass very gently with ethanol being careful to preserve the conductive layer and let it dry for some minutes.
- With a very soft pencil (5B/6B or even 7B) deposit a thin layer of graphite on the conductive side of the glass. Pay attention to cover uniformly the surface.



Ptc. 8) Below you can see the deposition of graphite on a microscope slide performed with two pencils (4B and 5B) and a charcoal.

5. FINAL ASSEMBLY

- Assemble the device, as shown in picture, by using two binder clips. Note that the graphite layer must be in contact with the dye sensitized one.
- Use a glass capillary tube to fill the space between the two electrode with the I^-/I_3^- . Please be sure to remove all the excess of electrolyte to avoid possible short-circuits.
- Connect two copper wire to the electrode (check you are connecting the wire to the conductive side) by using a conductive epoxy resin.



Ptc. 9) Assembled working device (sensitized with chlorophyll).