Production of a Biphasic Glow Stick through Organic Synthesis, Fluorescence, and Chemiluminescence

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***Abstract*—This experiment demonstrates the production of a biphasic glow stick using organic synthesis to showcase fluorescence and chemiluminescence. Fluorescein, synthesized from phthalic anhydride and resorcinol, produces green fluorescence in a basic solution. TCPO (tetrachlorophenyl oxalate), prepared from 2,4,6-trichlorophenol and oxalyl chloride, exhibits blue chemiluminescence when reacting with hydrogen peroxide. Combining these components in a dark environment results in a striking dual-phase glow, highlighting energy transfer mechanisms in light emission.**

I. Introduction

Fluorescence and chemiluminescence are key phenomena in photochemistry with diverse applications, including biomedical imaging and energy-efficient lighting. In this experiment, we synthesize a glow stick by combining these two processes. Fluorescein, a fluorescent dye, and TCPO, a chemiluminescent compound, are synthesized through organic reactions and then used to produce visible light without requiring an external energy source. This experiment explores the principles underlying chemical light emission, where energy from a chemical reaction excites molecules to emit light.

The biphasic glow stick illustrates fluorescence, where fluorescein absorbs energy and re-emits it as green light, and chemiluminescence, where TCPO reacts with hydrogen peroxide to generate blue light. This process serves as a practical demonstration of energy transfer between chemical components, highlighting the connection between molecular properties and light emission.

II. Theory

Luminescence refers to the emission of light by certain materials upon absorbing energy, without the involvement of heat, distinguishing it from incandescence. This phenomenon, often called "cold light," can occur via several mechanisms. The main types of luminescence are as follows:

1) **Photoluminescence:** Light emitted when a material absorbs photons (light energy) and re-emits them. It includes:
   a) **Fluorescence:** Light is emitted almost instantaneously (within nanoseconds to microseconds) upon excitation and stops as soon as the excitation source is removed.
   b) **Phosphorescence:** Light emission persists even after the excitation source is removed, lasting from microseconds to minutes or longer, and is typically of lower energy.
2) **Chemiluminescence:** Light produced through a chemical reaction. Glow sticks are an example. A subcategory of chemiluminescence is bioluminescence, which occurs in living organisms such as fireflies and certain deep-sea species.
3) **Electroluminescence:** Light emitted in response to an electric field or current passing through a material. Examples include light-emitting diodes (LEDs) and electroluminescent displays.

The differences between chemiluminescence and fluorescence are as follows:

1) **Source of Energy:**
   - **Fluorescence:** Emission occurs when a material absorbs photons (light energy) and re-emits them. The absorbed energy excites electrons to higher energy levels, and light is emitted when they return to the ground state. External light (often UV or visible) serves as the excitation source.
   - **Chemiluminescence:** Emission arises from the energy released during a chemical reaction, without the need for an external light source. The reaction releases energy that excites electrons, resulting in light emission as they return to their ground state.
2) **Duration of Emission:**
   - **Fluorescence:** Light is emitted almost immediately and ceases as soon as the excitation source is removed, typically occurring on a nanosecond to microsecond timescale.
   - **Chemiluminescence:** Light continues to be emitted as long as the chemical reaction is active.
3) **Mechanism:**
   - **Fluorescence:** Excitation and emission involve photon absorption and emission cycles.
   - **Chemiluminescence:** Excitation occurs due to energy released from chemical bond formation or rearrangement during a reaction.

The biphasic glow stick comprises two layers: a fluorescent aqueous upper layer, excited by the energy transfer from an organic chemiluminescent lower layer.

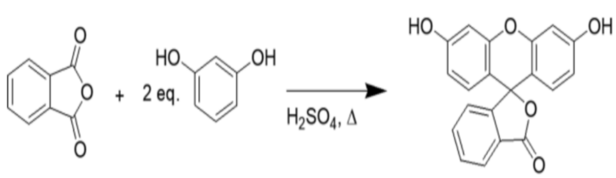

Fig. 1. Synthesis of fluorescein

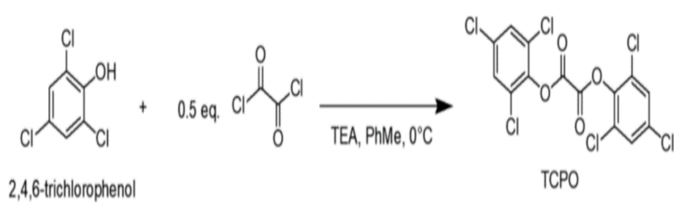

Fig. 2. Synthesis of tetrachlorophenyl oxalate (TCPO)

III. Experimental Procedure

A. Apparatus and Materials Used

- **Test tubes:** Small glass tubes used for mixing and reacting chemicals.
- **Distilled water:** Purified water used in the experiment to avoid contaminants that could interfere with reactions.
- **Magnetic bead and stirrer:** A magnetic bead, used in conjunction with a stirrer, facilitates stirring of liquids without physical contact.
- **Beaker:** A cylindrical container used to hold liquids and carry out chemical reactions.
- **Droppers:** Used to transfer small volumes of liquids accurately.
- **Phthalic anhydride:** An organic compound used as a reactant in the synthesis of fluorescein.
- **Resorcinol:** A phenolic compound used in the synthesis of fluorescein.
- **Mortar and pestle:** Tools used for grinding and mixing solid chemicals into a powder.
- **Oil bath:** A device used to maintain a stable temperature for reactions, especially when heating chemicals.
- **Vacuum filter:** Used to separate solid products from liquids by creating a vacuum to pull liquid through filter paper.
- **2,4,6-trichlorophenol:** An organic compound used in the synthesis of TCPO (tetrachlorophenyl oxalate).
- **Septum:** A rubber or silicone stopper used to seal containers for reactions under controlled conditions, such as nitrogen purging.
- **Nitrogen gas:** An inert gas used to create an oxygen-free environment during reactions, preventing oxidation.
- **Dry toluene:** A solvent used in the synthesis of TCPO, prepared free of moisture to ensure effective reactions.
- **Oxalyl chloride:** A reactive compound used in the synthesis of TCPO, facilitating the formation of ester linkages.
- **Ice bath:** A cooling method used to control the temperature of reactions and prevent them from becoming too exothermic.
- **Methanol:** A solvent used for washing and purifying solid products, particularly in the synthesis of TCPO.
- **Sodium hydroxide solution:** A strong base used to dissolve fluorescein, helping it to fluoresce under UV light.
- **Sodium acetate:** A buffer used in the preparation of the chemiluminescent solution.
- **Diphenyl anthracene:** A compound used in the chemiluminescent solution to enhance light emission in the glow stick.
- **Diethyl phthalate:** A solvent used to dissolve diphenyl anthracene and facilitate the formation of the chemiluminescent solution.

B. Methodology

1) **Step 1: Synthesis of fluorescein**
   a) Weigh 1 g of phthalic anhydride (1 equivalent) and 1.4 g of resorcinol (2 equivalents).
   b) Using a mortar and pestle, grind the phthalic anhydride and resorcinol into a fine powder.

   c) Transfer the powdered mixture, along with a magnetic stirrer bead, into a 100 mL beaker.
   d) Place the beaker in an oil bath on a magnetic stirrer, and heat the mixture to a temperature between 150°C and 180°C while stirring until it melts completely.
   e) Once the mixture has melted, carefully add concentrated sulfuric acid to the beaker.
   f) Observe the color change that occurs during the reaction.
   g) After the reaction is complete, remove the beaker from the oil bath.
   h) Add additional concentrated sulfuric acid to dissolve the thick paste in the beaker.
   i) Once the paste is dissolved, slowly add water to dilute the solution.
   j) Perform vacuum filtration to isolate the product.
   k) Collect the final product, fluorescein, as a yellowish powder.

2) **Step 2: Synthesis of TCPO**
   a) Set up a 10 mL round-bottom flask on a stirrer and place a magnetic stirrer bead inside.
   b) Add 500 mg (1 equivalent) of 2,4,6-trichlorophenol to the flask, cover it with a septum, and purge the flask with nitrogen gas.
   c) Dissolve the trichlorophenol in dry toluene using a metallic needle, while maintaining the nitrogen atmosphere.
   d) Add 0.35 mL (1 equivalent) of triethylamine to the flask and stir the mixture for 5 minutes under nitrogen conditions.
   e) After placing the flask in an ice bath, carefully add 0.1 mL (0.5 equivalent) of oxalyl chloride to the solution. The solution will begin to solidify.
   f) Remove the flask from the stirrer and proceed to vacuum filter the solidified solution.
   g) Wash the solid product with methanol to purify it.
   h) Collect the final product, TCPO, which will appear as a white powder.

3) **Step 3: Glow stick assembly**
   a) **Fluorescein Solution:** Dissolve a small amount of fluorescein powder in a sodium hydroxide solution to produce a bright green fluorescent color.
   b) **Chemiluminescent Solution:**
      i) Prepare a solution by mixing sodium acetate and diphenyl anthracene in equal parts (1:1) with diethyl phthalate.
      ii) Add a small amount of TCPO to the chemiluminescent solution, resulting in a blue chemiluminescent glow.
   c) **Glow Stick Assembly:**
      i) In a test tube, combine the prepared fluorescein solution with the chemiluminescent solution.
      ii) Move to a dark area and observe the glow stick effect, where both chemiluminescence and fluorescence are visible together.

IV. Results

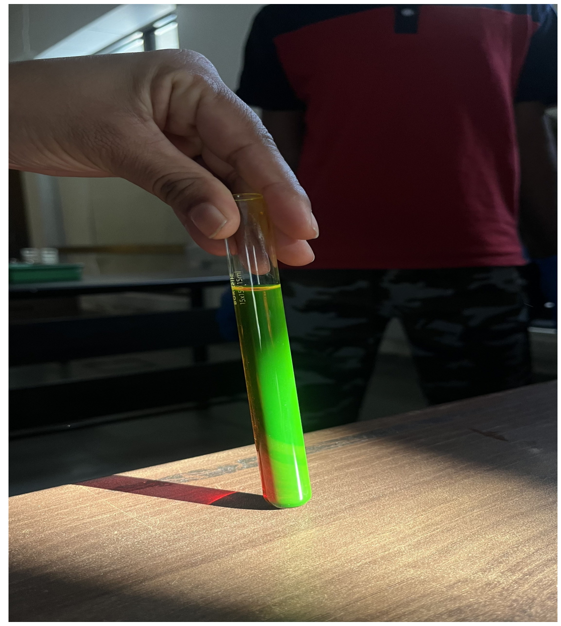

Fig. 3. Fluorescein Solution

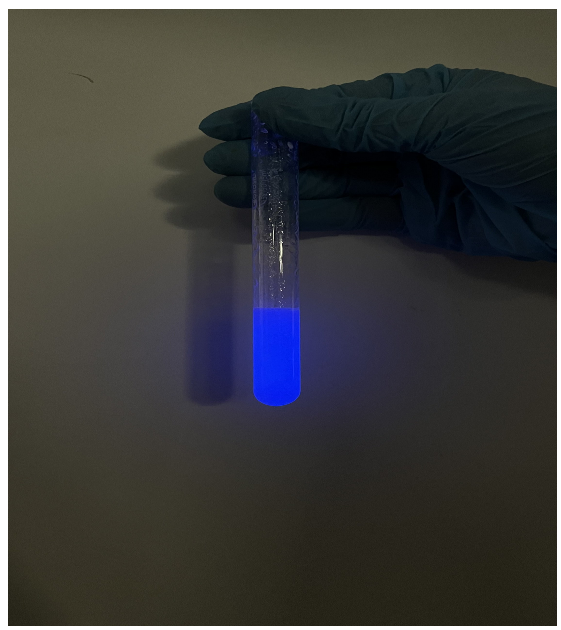

Fig. 4. Chemiluminescent Solution

1) The fluorescein solution exhibited sustained luminescence, maintaining stable emission over an extended period.
2) The TCPO solution used for chemiluminescence showed a gradual decline in light intensity, as the reactants were consumed and the intermediates reduced over time.

3) The degradation of luminescence in the chemiluminescent solution followed an exponential decay rather than a linear pattern.
4) The biphasic glow stick displayed two distinct layers due to the differing densities of the two solutions, while simultaneously exhibiting the characteristics of both.

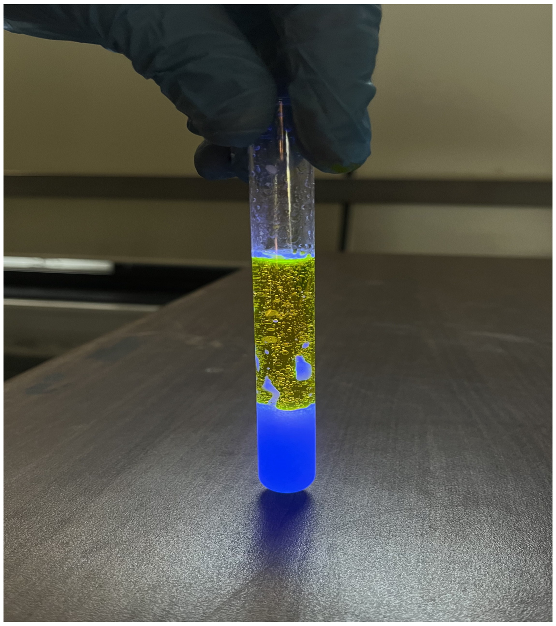

Fig. 5. Biphasic Glow Stick

V. Conclusion

In this experiment, we successfully created a biphasic glow stick that displayed both fluorescence and chemiluminescence. Through the preparation of fluorescein and tetrachlorophenyl oxalate (TCPO) and their careful combination, we observed luminescent behavior. The chemiluminescence exhibited a non-linear decay, while the fluorescence persisted over time.

The glow stick proves to be a versatile light-emitting device, offering a unique combination of continuous fluorescence and temporary chemiluminescence. The findings from this experiment confirm the potential of the biphasic glow stick for practical applications, such as in safety lighting and rescue operations.

VI. Precautions & Challenges

- Always wear gloves, safety goggles, and other necessary protective equipment.
- Handle all chemicals with caution as they can be highly corrosive.
- Avoid inhaling any fumes emitted during the experiment.
- Perform any procedures that release fumes or pose hazards in a well-ventilated fume hood.

VII. Acknowledgment

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