Degradation of an organic pollutant in water using LED light and photocatalyst

BS 192, Experiment: 4

We will use light to degrade organic dye molecules, which are commonly used in dye industries. They are often present in industrial wastewater. We will use regular LED torches and a photocatalyst to remove/degrade them. We will study the kinetics of the reaction. We will also understand the usefulness of a photocatalyst.

Materials:

Methylene blue (organic pollutant) and Iron Oxide Nanoparticles (Fe_3O_4) (photocatalyst)

Basic principle:

The photocatalyst absorbs light and produces holes and electrons which help in degrading the dyes. The nanoparticle may also adsorb some dyes due to electrostatic interactions.

$$\boldsymbol{Fe_3O_4 + h\gamma \rightarrow Fe_3O_4(e^-(CB) + h^+(VB))}$$
$$\boldsymbol{MB + h^+(VB) \rightarrow Oxidation\ of\ MB}$$
$$\boldsymbol{H_2O + h^+(VB) \rightarrow OH^\cdot + H^+}$$
$$\boldsymbol{MB + OH^\cdot \rightarrow CO_2 + H_2O}$$

Procedure:

1. Prepare three vials with the following details:

- Vial 1: 5 ml methylene blue (stock solution) + 5 mg of Fe_3O_4 nanoparticles
  Wrap vial 1 with Aluminum foil and keep aside
- Vial 2: 5 ml methylene blue (stock solution) + 5 mg of Fe_3O_4 nanoparticles+ + 1 magnetic bead
- Vial 3: 5 ml methylene blue (control experiment)

Put vial 2 and 3 under the LED light on top of the magnetic stirring bar

N.B. Start the timer when you add Fe_3O_4 nanoparticles to the solution.

Do not mix up the vials – Recommendation: one person in each group can handle one solution specifically.

2. You need to record UV-Vis absorption spectra of the solution in every 10 minutes for 1h, see below:

In every 10 minutes

(i) Put **vial 1** near the magnetic post/bar to remove the Fe_3O_4 nanoparticles and then take 2 ml of the sample and measure absorbance spectrum. Pour this 2 ml back in the vial (after measurement) and keep it in dark.

(ii) Take **vial 2** out and put near the magnetic post/bar to remove the Fe_3O_4 nanoparticles and then take 2 ml of the sample and measure absorbance spectrum. Pour this 2 ml back in the vial (after measurement) and put the vial back under LED at the earliest.

(iii) Take 2 ml from **vial 3** and measure absorbance spectrum. Pour this 2 ml back in the vial (after measurement) and put the vial back under LED at the earliest.

3. Determination of concentration from absorbance:

According to Beer-Lambert's law:

$$Absorbance = \varepsilon . c . l$$

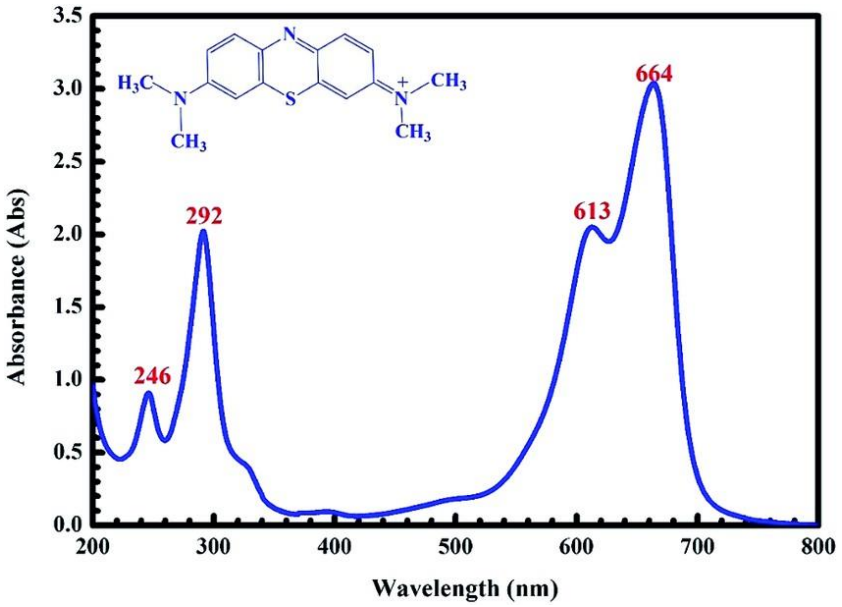
Where ∈= molar extinction coefficient, a property of the molecule being studied. C is the concentration, and l is the path length. For a given molecule and a given experimental set up, ε and l are constant. Molar extinction coefficient (ε) is a measure of how strongly a chemical species or substance absorbs light at a particular wavelength Thus absorbance will be proportional to concentration.

3.1. Calibration with known concentrations:

You will be given 2 ml MB solution with a concentration of 5 ppm. Record the UV-Vis spectra of the following solutions systematically (one by one):

(i) TT 1: 2 ml MB
(ii) TT 2: 1 ml MB + 1 ml water
(iii) TT3: 1 ml MB + 2 ml water
(iv) TT4: 1 ml MB + 3 ml water
(v) TT5: 1 ml MB + 4 mL water

- Take ~ 2 ml from each of the test tube and measure their absorption spectra.
- Plot absorbance maximum (peak ~ 664 nm, typical spectrum of MB is given below) with different MB concentrations.
- From the slope of this graph, calculate ε. Note that l = 1 cm.



The lab report should include the following graphs and calculations:

1. Plot of concentration *vs* absorbance maximum (Calibration plot) for MB solutions with different concentration (Refer to point 3) and calculation of molar extinction coefficient (ε) of MB.

   All the recorded UV-Vis spectra measured for this calibration plot should be included in the report.

2. Calculate concentrations of MB in vial 1, 2, and 3 at different time (refer to point 2 in procedure) using the determined ε of MB from the calibration plot.

   All absorption spectra measured for vial 1, 2, and 3 should be included in the report. One plot for each vial, showing the decrease of absorbance with respect to time.

3. Plot $\ln(C_0/C)$ *vs* t for vials 1, 2, and 3 and should be reported in a single figure.

   Calculate the slopes from each graph.

   C_o is the concentration of MB at t = 0 time.

   Comment on the kinetics of the reactions.

4. Calculate the amount of MB degraded after 30 minutes of light exposure in presence of the catalyst (vial 2) and compare this value with the situation where MB was exposed to light in absence of the catalyst (vial 3).