Experiment # 5: UV-Vis Spectrophotometer

Objectives:

- Understand the functionality of the UV-Vis spectrophotometer and how does it work
- Be able to construct a calibration curve for a certain solution
- Be able to apply Beer-Lambert equation to estimate unknown concentration of given solution (i.e., dyes)

Apparatus:

- GENESYS 10S UV-VIS Thermo Science Spectrophotometer (Figure 1)

Figure 1: UV-Vis Spectrophotometer

- 1 cm width Quartz/Glass or plastic cuvette

Figure 2: Cuvette

Theory:

To calculate the concentration C:

Lambert-Beer Law:

$$A = \varepsilon \, x \, l \, x \, C \qquad (1)$$

Where:

A: The absorptivity of the solution.

ε: The molar absorptivity, L /mol.cm.

l: The cuvette path length, cm.

C: The concentration of the solution, mol/L.

Procedure:

1) Turn on the UV-Vis Spectrophotometer and leave it for 10 min to warm-up.
2) Remove any previous left-over cuvettes
3) Calibrate the device by using an appropriate reference (blank) (water in case of dye solutions)
4) Scan your targeted solution (Methylene Blue) between 200-800 nm to find the maximum absorbing wavelength (λ_{max})
5) Repeat step 4 for other dye solutions (green, yellow, red dyes)
6) Prepare methylene blue solutions with different concentrations (0 to 35µM) and observe their absorbance at λ_{max}
7) Find the absorbance of unknown solution's concentrations, apply Beer-Lambert equation and find their concentrations.

Experiment # 6: Synthesis of Carbon Nanodots (CNDs)

Objectives:

- Synthesize CNDs from different precursors such as ethanolamine and instant coffee.
- Study the effect of oxidant agent on CNDs formation rate and properties
- Examine the resulted CNDs by spectrophotometer and UV Lamp.

Apparatus:

- GENESYS 10S UV-VIS Thermo Science Spectrophotometer (Figure 1)

Figure 1: UV-Vis Spectrophotometer

- 365 nm UV Lamp

Figure 2: UV Lamp

Theory:

CNDs can be synthesized through two routes; top-down and bottom up by different methods.

Procedure:

Part I: Synthesis of CNDs from ethanolamine

1) Add 3 mL of ethanolamine into two 200 mL beakers.
2) Add 4.5 mL of hydrogen peroxide (30 wt% H_2O_2) to one of contained ethanolamine beakers.
3) Heat both beakers on a hot plate till a black residue formed at the bottom.
4) Cool both beakers down to room temperature.
5) Add 200 mL deionized water into both beakers to get CNDs concentrated solution.
6) If any undispersed particles are observed, centrifuge both solutions (or sample from them) at 3000 rpm for 15 minutes and filter the supernatant through 0.22 µm microfilter.
7) Dilute 0.5 mL of concentrated solutions and observe under UV lamp and UV-Vis spectrophotometer.

Part II: Synthesis of CNDs from instant coffee

1) Dissolve 0.5g of instant coffee in 50 ml water.
2) Sonicate for 10 min once alone and another with 200µL H_2O_2.
3) Centrifuge and filtrate the sample
4) Dilute 0.5ml of concentrated carbon nano dots solution and observe under UV light and UV-Vis spectrophotometer.

Results:

- The effect of H_2O_2 on CNDs formation
- The fluorescence of samples under UV lamp
- The UV-Vis spectra of all synthesized CNDs

Discussion:

- Discuss the mechanism of CNDs synthesis
- Discuss the difference of CNDs formation in the absence and presence of H_2O_2
- Discuss the behavior of UV-Vis spectra obtained for synthesized CNDs and the fluorescence observed under UV lamp.

Experiment # 7: Photodegradation of Methylene Blue Dye by Carbon Nanodots

Objectives:

- Understand the degradation of process of dyes by the use of photocatalysts such as CNDs
- Monitor the concentration drop of methylene blue with time upon exposure to visible light by the use of UV-Vis spectrophotometer.
- Estimate the degradation rate and efficiency of methylene blue.

Apparatus:

- GENESYS 10S UV-VIS Thermo Science Spectrophotometer (Figure 1)

Figure 1: UV-Vis Spectrophotometer

- 50 W Tungsten lamp and quartz cuvette

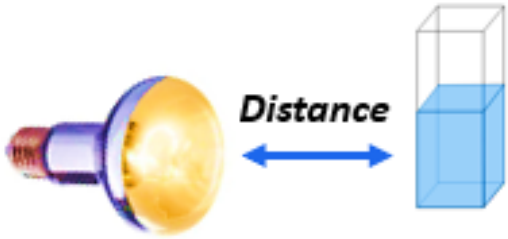

Figure: Visible light source and cuvette

Theory:

The degradation efficiency was calculated according the following equation:

$$\boldsymbol{Degradation\ Effeciency}\ (\%\boldsymbol{D}) = \frac{\boldsymbol{A_0 - A}}{\boldsymbol{A_0}} \times \mathbf{100}\% \qquad (\mathbf{1})$$

Where A_0 the absorbance of M.B at time zero, A absorbance at time = t.

Another important degradation parameter that should be estimated is the degradation rate. Herein, Langmuir–Hinshelwood dynamic model will be used, where the degradation kinetics of M.B could be simplified according to the pseudo first-order kinetic as shown in equation (2)

$$\mathbf{ln}\left(\frac{\mathbf{Co}}{\mathbf{C}}\right) = \mathbf{k} \times \mathbf{t} \qquad (\mathbf{2})$$

Where C_0 and C should be the equilibrium concentration of M.B and the concentration of M.B after irradiation time t, respectively. C_0 and C are equivalent to the absorbance of the dyes at time zero (A_0) and the absorbance of the dyes after degradation time t (A), respectively and k represents the dye degradation rate constant.

Therefore, a fitted plot of $ln(A_0/A)$ versus time leads to a straight line with a slope representing the degradation rate

Procedure:

1) Prepare Methylene blue solution with initial concentration of 40 µM and transfer 3 ml to quartz cuvette.
2) Spike the above solution with 500 µL of CNDs (0.2 mg/mL).
3) Leave the above mixture under dark for 10 minutes to achieve equilibrium.
4) Test the absorbance of the above solution and record A_0 at $t = 0$.
5) Irradiate the sample by visible light (50 W tungsten lamp) and record absorbance every 10 to 20 minutes for a total period of 120 minutes.

Results:

- The relation between absorbance and irradiation time
- Calculate the efficiency and degradation rate of methylene blue

Discussion:

- Discuss the mechanism of photodegradation.
- Discuss your observations